Production and Evaluation of Waste and Short-fibre composites

By

Craig Anthony Smith

[redacted]

A thesis submitted to the University of Birmingham for the degree of MASTER OF RESEARCH IN THE SCIENCE AND ENGINEERING OF MATERIALS

School of Metallurgy and Materials

University of Birmingham

April 2012

Craig Smith - [redacted]

Abstract

This research project investigated techniques to enable the fabrication of fibre reinforced composite components from waste generated in the manufacture of glass fibre fabrics. These waste fabrics and fibres were used to manufacture filament wound tubes using a new technique termed “clean filament winding”. The end-use application for the filament wound tubes was to replace cardboard inner tubes that are currently used by the sponsoring industrial partner to over-wrap the woven fabrics.

Pre-manufactured glass fibres were also used in this study to investigate the production of aligned short-fibre prepregs via a vibration-based alignment technique. The aligned short-glass fibre preforms were autoclaved to produce composite panels.

The short-fibre composites were evaluated through the degree of alignment of the short-fibres in the preform, tensile and flexural strengths, fibre volume and void fractions. The filament wound tubes were characterised by: fibre volume and void fractions, hoop-tensile strength, lateral compression strength and inter-laminar shear strength (ILSS).

The composite tubes produced using the clean filament winding process proved comparable to those produced using conventional wet-filament winding. Lateral compression strength of the filament wound tubes was significantly superior compared to the cardboard tubes. The degree of short-fibre alignment using the vibration-based alignment technique was over 80%.

Contents Page

1. Introduction

A composite is a material of two or more components which, when combined are stronger than the individual constituents. The reinforcement can consist of short fibres, continuous fibres or particulates where the fibres are held in position via a matrix. The use of fibre reinforced polymer composite consisting of glass, carbon, aramid or more recently natural fibres, reinforcing a thermosetting or thermoplastic polymer are a more popular choice with industry.

Fibre reinforced polymers are used extensively in the marine, automotive and sporting sectors, due to their high strength to weight ratios, corrosion resistance and the ability to obtain tailor-made properties and shapes (Conroy *et al*., 2005). It is because of these highly desirable properties that the production rate of carbon fibre in particular, is roughly 3000 tonnes a year in the USA and Europe. The production rates of these composites are only likely to increase, with virgin carbon fibre production projected at around 100,000 tonnes by 2018 (McConnell, 2010).

In addition, approximately 15 million end-of-life vehicles are currently being scrapped each year in Europe (Kilde and Larsen, 2000) where 20% of an average 120 kg end-of-life vehicle is composite waste (Mckechnie and Wegman, 2008). These scrappage figures are projected to rise rapidly with approximately 16.5 million vehicles currently being forecast for 2015 which presents a serious waste management issue (Kilde and Larsen, 2000).

Therefore due to the economic and environmental issues associated with the disposal of waste in landfills, a series of European Union directives have been put in place to encourage the composites industry to deal with composite waste. These EU directives include the Waste Framework Directive (2008/98/EC), Integrated Pollution Prevention and Control (IPCC) Directive (2008/1/EC), Landfill Directive (2003/33/EC) and the End-of-life Vehicle Directive (2000/53/EC).

Due to these directives and potential costs to the producer, a waste hierarchy, as depicted by Conroy *et al*., 2005, can be drawn up. This discusses the most and least desirable options when looking at the life cycle of products. The diagram presented in

Figure 1 considers the possible after-lives of products and therefore should be considered during the design of a new product.

Figure 1. The waste hierarchy as depicted by Conroy et al., 2005.

The most desirable option of the hierarchy is reduction which, based on the projections on production rates and the extensive use of composites, could be a very difficult option. Therefore methods of reuse must initially be considered for the composite industry. Thermoplastics can be potentially re-shaped into new forms, which is not possible for thermosetting composites. Any other 'reuse' would have to come from waste production. A reuse method for this waste material must therefore be found but problems with this re-processing lie in achieving consistency. As the material is waste, this can lead to variance in product quality and type which can make it difficult to achieve reliable re-processing methods.

However these reuse options for waste and thermoplastic matrix materials still do not apply to thermosetting materials. This means the current option for a number of composite producers/users is to landfill their composite products, which is the least desirable option in the waste hierarchy shown above. This is generally due to the lack of options for mainly thermosetting composites where a grinding method of recycling can be expensive or of no real use to the producer.

However due to the elevated cost and restrictions on landfilling, much interest has been aimed at finding recycling methods to recover the fibre proportion of fibre reinforced composites. The numerous methods that have been investigated can be divided into four categories: thermal, chemical, mechanical and re-use. Nevertheless

the fibres attained from these methods generally produce fibres of a discontinuous form and in order to produce the strongest composite possible, methods of orientating these short fibres have to be studied.

This project aims to investigate the re-use of waste and recycled glass fibres. The two main focuses of this project will be to investigate methods to fabricate high quality composite components from: (i) continuous waste glass fabric material and (ii) short E-glass fibres. The continuous fibres will be processed using a method termed clean filament winding, with the discontinuous fibres managed using a custom-built short fibre deposition system.

The waste glass fibre fabric is generated from the weaving of a continuous glass fibre fabric but two forms of waste are created; direct-loom waste and waste slittings. The direct-loom waste is generated from a weaving process incorporating the E-glass fibres. An image of the production and extraction of direct-loom waste is shown in Figure 2.

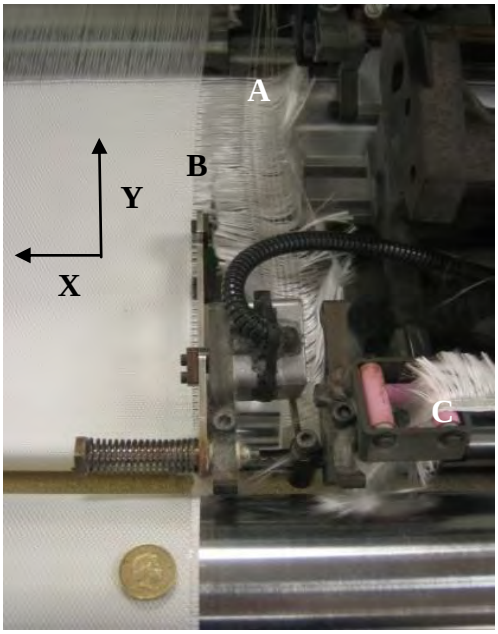


Figure 2. The production and extraction of the direct-loom waste from the weaving of a continuous E-glass fibre fabric.

With reference to Figure 2, the glass fibres are woven in the x and y-directions to create the fabric. The excess fibres can be seen in A, where they are supported by cotton strands so that the fibres are secured in position during the trimming operation. The excess fibre (A) is cut at point B in order to create a straight edge for the fabric.

Once cut the excess fibres are pulled round to point C where they are then extracted through suction to capture any loose fibres, safely and securely.

The waste slittings are generated once the direct-loom waste has been cut away and the fabric has been treated with a resin sealant. Once this sealant has cured, the waste slittings are then trimmed away from the fabric prior to the fabric being wound around a cardboard inner tube. This process can be shown in more detail in Figure 3.

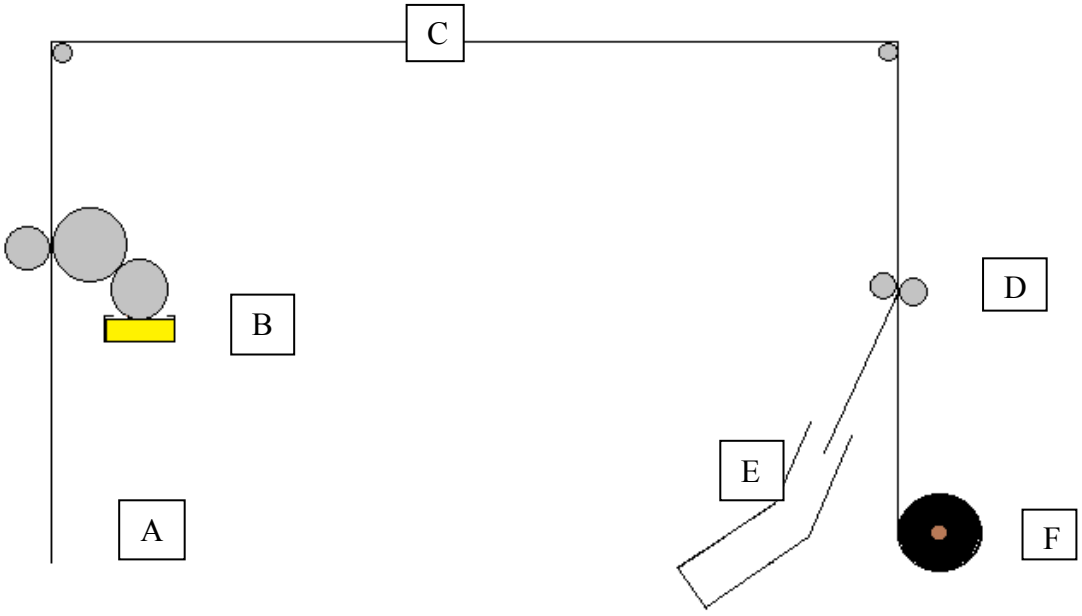


Figure 3. A schematic illustration demonstrating how the waste slittings are generated from the finishing process of a glass fibre fabric.

With reference to Figure 3, once the fabric (A) has passed through the aforementioned direct-loom waste stage, it then passes on to point B where the resin sealant is applied by rollers. These rollers ensure that a sufficient amount of resin is applied to the fabric. Once this resin is applied, it is allowed to "dry" at point C. The dried fabric continues to point D where the edge of the fabric is cut to ensure that the fabric is of a specific width, therefore generating the waste slittings. These waste slittings are then extracted via suction at point E. The glass fibre fabric then is wound around a cardboard inner tube (F) to complete the process.

The discontinuous E-glass fibres used in this study will be pre-chopped 4.5 mm E-glass fibres and represent discontinuous fibres created during recycling methods.

2. Aims

Due to the aforementioned issues associated with waste management, this study includes the development of two production methods to fabricate recycled composites. In particular, the aims and objectives of this study were:

i. To complete a detailed literature review of any previously published composite recycling methods.

ii. To develop manufacturing methods, that can produce composite components from waste fibre material.

iii. To develop quantitative and qualitative evaluation techniques to assess and compare any recycled composites fabricated during this study.

iv. To assess the feasibility of fabricating recycled composite components.

3. Literature Review
3.1 EU Directives

Due to mounting public interest in environmental issues and waste management overall, recycling and landfilling concerns are a priority. Numerous economic and environmental issues associated with landfilling are forcing radical changes to all aspects of day-to-day life. The composite world is no different and a series of European Union directives have been issued to encourage the industry to find methods for dealing with their composite waste. These EU directives include the Waste Framework Directive (2008/98/EC), Integrated Pollution Prevention and Control (IPCC) Directive (2008/1/EC), Landfill Directive (2003/33/EC) and the End-of-life Vehicle Directive (2000/53/EC).

The waste framework directive (2008/98/EC) aims to reduce landfill levels and hence greenhouse gases from this reduction. This is achieved by the reduction of waste produced during manufacture as well as an increase in the recycling of waste. Specifically, within the UK, a waste management licensing regulation (WMLR) must be given before any waste can be dealt with (2008/98/EC). The WMLR entitles the holder to landfill, dispose of waste using plant equipment (incineration, shredding or sorting) or treatment, dispose and store material on land. A permit is acquired by producing a detailed report on the nature of the waste. This includes how this waste must be stored and treated, ensuring that this will have no detrimental effect on the environment. The UK environmental agency has also adopted a ‘polluter pays’ attitude in the directive.

In addition to this waste framework directive, a landfill directive and the Integrated Pollution Prevention and Control (IPCC) Directive (96/61/EC) must be complied with. This directive, similar to the WMLR, states that all landfills must have an IPCC permit. This permit implements further rules for producers to follow, for example they must: (i) use the best available techniques in preventing pollution; (ii) prevent all large-scale pollution; (iii) prevent, recycle or dispose of all waste in the most environmentally friendly manner; (iv) use energy efficiently: and (v) restore sites to their original state once operations are completed. In addition to this all landfills must be registered for a specific type of waste i.e. hazardous or non –hazardous and ensuring no cross contamination can occur.

Finally the End of Life Vehicles directive (2000/53/EC) has received much attention

due to the implications for composites. The ELV directive states that all vehicle manufacturers must provide free-of-charge authorised treatment facilities (ATF). These ATFs must reclaim their vehicles in their 'end-of-life' forms and recover as much material for recycling and/or reuse as possible. The directive (2000/53/EC) states that by 2015 greater than 95% of all ELVs must be recoverable. Currently it is estimated that only 75% of all vehicles (percentage by weight) are recyclable, with this achieved through the metal content of these vehicles (Kanari *et al*., 2003). The remaining 25% consists of composite materials, glass, textiles and rubber components. The vast majority of this waste is discarded into landfills and with an estimated 9 million tonnes of waste a year produced by end-of-life vehicles (McKechnie and Wegman, 2008), these levels will soon become unmanageable. Consequently the composite industry is investigating various reusing or recycling methods in order to comply with these directives and make them directly comparable to their competitors. In addition to these directives, a landfill tax has also been placed on disposal sites across the UK. This tax, enforced in 2008, aims to increase the landfill tax by £8/tonne each year up to 2014/2015 where it will reach a standard rate of £80/tonne (Chancellor's Budget, 2010).

The aforementioned large waste volumes produced by end-of-life vehicles (McKechnie and Wegman, 2008) as well as various other forms of waste have forced the European Union to act in order to limit waste disposal. Nevertheless, with demand and production rates only increasing, especially for composites, alternative solutions must be found to minimise waste. The following section will therefore outline methods to recycle composites and possible end-uses.

3.2 Composite Recycling Methods

Due to the aforementioned issues with the EU legislations (2000/53/EC, 2008/98/EC, 2008/1/EC, 2003/33/EC) there is a real push from the composites industry to find solutions to these problems, with reusing or recycling of these composites appearing to be the most attractive of those in the waste hierarchy by Conroy *et al*., 2005. The subsequent section will detail various methods that have been employed to recycle composites and will also review their effectiveness.

As stated previously the recycling of composites as a whole can be grouped into four categories (i) thermal; (ii) mechanical; (iii) chemical; and (iv) re-use. Figure 4

presents an overview of composite recycling as a whole and was developed from recycling review diagrams by Pickering *et al*., 2000 and Otheguy *et al*., 2009.

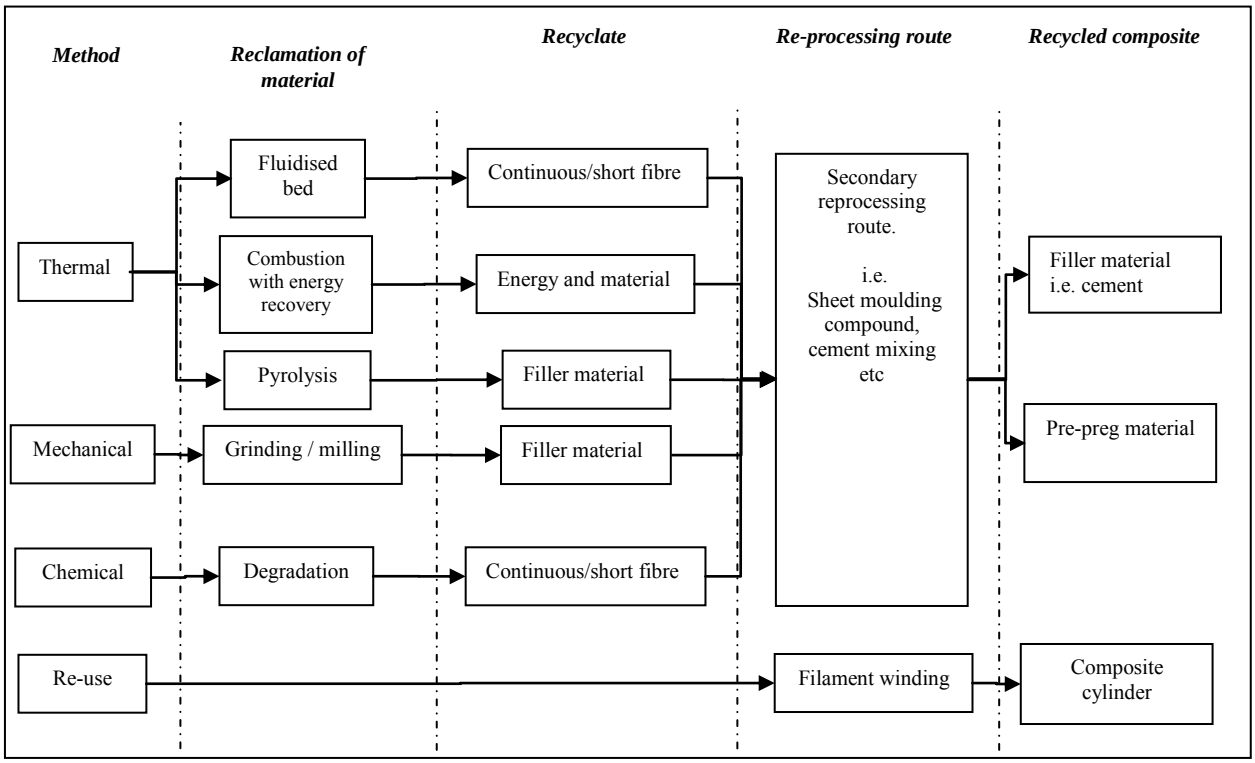


Figure 4. An overview of composite recycling techniques developed from Pickering *et al*., 2000 and Otheguy *et al*., 2009.

3.2.1 Thermal degradation

The thermal aspect of recycling comes in the form of combustion with energy recovery, pyrolysis or a fluidised method which involves burning off the polymer matrix at high temperatures, ~450°C, to leave just the fibres (Pickering *et al*., 2000; Kennerley *et al*., 1998; Cunliffe & Williams, 2003; Yip *et al*., 2002). Pickering and Kennerley along with colleagues at the University of Nottingham used a fluidised method of glass fibre recovery which is presented in Figure 5. This involves placing the composite onto a hot bed of sand in order to degrade the polymer matrix. The fibres can then be blown out by air and collected, with these fibres shown to have a 50% reduction in strength (Pickering et al., 2000) compared to virgin material.

Figure 5. The fluidised bed method of fibre recovery used by Pickering *et al*., 2000 and Kennerley *et al*., 1998, at the University of Nottingham

Yip *et al*., 2002 were able to use the same process to recycle carbon fibre. Their paper went on to analyse the fibre's strength retention but also the surface topology and chemistry using scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS) respectively. The recycling process did reduce the oxygen containing functional groups on the surface on the fibre following XPS analysis with the fibre retaining 75% of its original strength.

Cunliffe and Williams, 2003, used a pyrolysis process to recycle a polyester/styrene copolymer reinforced with glass fibre. The recovered fibre from the pyrolysis process was then placed into a second muffled furnace at 450°C. This fibre was then used as a replacement (25 weight percentage) for virgin fibre in a dough moulded compound. Experimental data showed a 73%, 90% and 81% retention of Charpy impact strength, flexural strength and flexural modulus respectively. In particular, the strength reduction was attributed to the degradation of the fibre during the recycling process. Despite the indication of fibre damage, a good surface finish was evident within the composite containing the recovered fibre. Here the authors suggested that these fibres could have some use in terms of lower strength applications or as a filler material where surface finish is an important factor.

A novel method proposed by Lester *et al*., 2004, was also able to show an efficient process of thermoset composite recycling via microwave heating. Four 3g sheets of carbon fibre composite were placed into a bed of sand with the multimode microwave cavity powered for 8s at 3 kW. Glass wool was used to prevent solids leaving the microwave cavity. A steady stream of nitrogen gas (5 l/min) was used to create an inert atmosphere and therefore prevent combustion of the fibres during heating.

The microwave heated fibres were shown to have strengths close to those of virgin fibres. However, the more noticeable result is that the tensile strength of these fibres was 7% higher than those of the fibres recovered from the fluidised bed method, although their tensile modulus was shown to be 14% lower. The improvements shown in the fibre were due to the less turbulent atmosphere in the process resulting in less damage, which suggests the sand is not the cause of the problem with the fluidised bed method.

3.2.2 Chemical Degradation

The chemical degradation methods showed a much higher strength retention in the fibres than the grinding or thermal methods. Yuyan *et al*., 2006, were able to effectively degrade the polymer matrix of glass/epoxy composites with the use of nitric acid. The samples were placed into 8M of acid solution for 5 hours at 90°C. Yuyan *et al*., 2006, were able to show strength losses of 3.5% in a single fibre test compared to virgin fibres. This shows a much higher strength compared to Pickering *et al*., (2000) with the fluidised bed process whilst having a lower processing temperature. The strength losses were still shown to be just as minimal when incorporated into a composite and placed under an inter-laminar shear strength test, clearly showing good interfacial bonding with these recovered fibres.

Jiang *et al*., 2008, used supercritical n-propanol to degrade the polymer matrix of a carbon fibre composite in a semi-continuous flow reactor. After 40 minutes, the matrix had been fully decomposed and was washed with the n-propanol. These recovered clean fibres were then characterised via SEM, a single fibre test, XPS and a micro-droplet test. The tensile and modulus of the recovered and virgin fibres were very similar however the XPS analysis confirmed that the surface chemistry was affected, agreeing with the work conducted with Yip *et al*.,2002. Nevertheless, the strength of the recovered fibres is much higher with Jiang's chemical method of degradation. This again raises the question of whether the slightly elevated temperatures in Yip *et al*.'s work (310°C compared to 450°C) diminished the strength of the fibre.

Pinero-Hernanz *et al*., 2008 were also able to show very good strength retention of the recovered fibres, up to 99% making this comparable to the findings of Jiang *et al*., 2008. However, Pinero-Hernanz *et al*., 2008, incorporated the use of supercritical

alcohols (methanol, ethanol, 1-propanol) and acetone to degrade the polymer matrix, where a slightly higher temperature is required to make the liquids supercritical, between 400 and 500°C. Although this process seems to be effective, the lower temperatures used by Jiang *et al*. would be more cost effective in the long run.

A second paper by Pinero-Hernanz *et al*., 2008 was concerned with the use of a more environmentally friendly system using supercritical water. The degradation took place in a batch-type reactor with temperatures ranging from 523 to 673 K (250-400 °C) and pressures from 4.0 to 27.0 MPa and 79% of resin was removed with the initial set-up as depicted in Figure 6A. However, with the addition of a catalyst (potassium hydroxide, 95 wt%) was able to remove the bulk of the resin which is evident in Figure 6B. Single fibre tensile tests on the supercritical water recovered fibres showed strength retention of 90-98% of the strength of virgin fibres compared to 96.5% strength retention shown by Yuyan *et al*., 2006 using alcohols.

Figure 6: (A) A scanning electron microscope image of a carbon fibre recovered from a chemical recycling process using supercritical water without a catalyst. (B) A scanning electron image of the carbon fibre recovered from a chemical recycling process using supercritical water and a catalyst (Pinero-Hernanz *et al*., 2008).

It may also be possible that the degradation media could be recycled, if distilled, enhancing the green credentials of the process. However, due to the benefits of this recycling method, high consumer interest is anticipated due to the current expense of virgin carbon fibre (~£30000/tonne, Boeing Environmental Technotes, 2003). Comparing these values to the potentially lower costs of recovering carbon fibre that are estimated by Adherent technologies inc. to a cost of £3473/tonne (Boeing

Environmental Technotes, 2003), clearly shows the economic viability of using these recovered fibres.

3.2.3 Mechanical Degradation

Recycling using mechanical techniques consists of grinding or milling the component to pellets that can be re-processed or using the recyclate as a filler material in wood, concrete or more notably thermoplastic composites. Palmer *et al*., 2009, used a hammer mill to break up glass fibre reinforced automotive front fenders. The best material obtained from this process would then be reincorporated into a virgin fibre composite. These fibres were separated via a zigzag air classifier which separates materials of different shapes or densities. The coarser grades would be disposed of with the finer grades reprocessed to obtain an even more refined fibre grade. The re-incorporation of finer grades of fibre by weight (10%) was shown to have no detrimental effects on the composites performance. However Bernasconi *et al*., 2007, were able to show that adding more recycled material to the composite has a more detrimental effect on performance.

Recycling thermoplastics is generally achieved by grinding the unwanted composite into pellets which are then injection moulded (Otheguy *et al*., 2009; Chu & Sullivan, 1996). This is only achievable when the pellets can be re-melted to form a new component. However, it has been shown that the more the pellets are re-processed the strength loss becomes greater (Sarasou & Pouyet, 1997). Although this appears to be a process of downgrading, the initial reprocessed material can show comparable mechanical and physical properties to virgin material of the same form (Steenkamer & Sullivan, 1998).

3.3 Applications for Recycled Fibres

For the majority of the recycling processes, it seemed evident that the strength values reported would not be sufficient for deployment into any load bearing applications. These fibres therefore generally end up as fillers in thermoplastic composites (Perrin *et al*., 2007, Kouparitsas *et al*., 2007). However, the recovery methods used in these

studies were based on grinding or shredding methods and therefore produced a lower grade of fibre.

Alternative uses for these types of recovered fibres are as a filler material in wood to create GRP/plastic lumber which can be used for light bridge foundations and jetties (Conroy *et al*., 2005). Other applications include GRP reinforced wood particleboard to replace wood in flooring (Conroy *et al*., 2005), particularly due to a good finish being possible (Cunliffe and Williams, 2003). Some fibres have also been used as a filler in bitumen (Bartl *et al*., 2005).

A feasible application for these recovered fibres includes incorporating them into concrete. Fibres implemented under the correct conditions can increase the compressive strength of concrete by up to 45% (Asokan *et al*., 2010) whilst reducing the density of the concrete composites by 12% (Asokan *et al*., 2009). Other feasible applications include '…thermo-resistant insulation materials' (Larsen, 2009), where glass fibres have been recovered from a pyrolysis method. The fibres can also be used for fibre-reinforcement in fillers, glue, thermoplastic components, asphalt, concrete and possibly raw material for new glass fibres. The energy generated from the process could be used to power district heating and to generate electricity (Larsen, 2009).

A final method of using recycled or waste fibres is re-use. In this project reuse recycling was defined as the direct use of used/waste material in a manufacturing process or secondary application. This method does not involve reclaiming the fibre unlike the aforementioned recycling methods but endeavours to process the waste forms cheaply and simply.

3.4 Summary of Recycling Methods

Due to the EU directives, and the demand for thermo-setting composites in load-bearing applications the focus of research has been aimed at this sector. It is therefore inevitable that various methods will arise and it is essential to assess these in terms of their suitability and efficiency.

For the recycling method to be successful, there also needs to be a viable application at the end of the process i.e. flooring or work surfaces. In addition, if the process is only recovering the fibres for a filler material it seems to be a short term solution and not necessarily cost effective. However studies (Cunliffe & Williams, 2003) have shown that by-products from a recycling process can be used as a fuel. This study was

able to show that the oil by-products of a pyrolysis process showed a similar calorific value to that of fuel oil with the recovered fibres still showing some strength retention. The other thermal methods also show some positive results although the elevated temperatures and lower strength retention of the fibres is a concern. The process seems to be causing some damage the fibres, however if the by-products can also become useable it could prove to be a feasible method.

The grinding method of recycling seems to be a short-term answer for the vast amount of waste being created but this is a method that should be phased out through the development of the other methods of recovery due to the severe damage to the fibres.

The chemical method seems to be the way forward for thermosetting resins as this method achieves a better strength retention in the fibres. (Pinero-Hernanz *et al*., 2008a, 2008b; Yuyan *et al*., 2004; Jiang *et al*., 2008). These methods generally use lower temperature methods and could also recycle the degradation media resulting in a more financially viable option of degradation. If these processes can be optimised through obtaining strong fibres; creating low emission processes and recycling of the degradation media, there is no reason why recycled composites cannot have a place in the engineering sector.

3.5 Short-Fibre Orientation

Short-fibres are generally used for low weight bearing applications due to their reduced strength compared to continuous fibre composites (Kardos, 1985). This is due to stress concentrations created at the ends of the short-fibres (Maclaughlin & Barker, 1972). However Fu *et al*., 1996 stated that short-fibre composites offer numerous advantages in their use, such as low production costs, greater feasibility for mass production, reduced production times and can yield equivalent mechanical properties to continuous fibre composites. These short-fibre composites also retain some of the drape-ability of a non-reinforced polymer and therefore allow the production of more complex shapes than a continuous fibre composite (Kardos, 1985). Nevertheless, in order to obtain the optimal properties of short-fibre composites they must be aligned (Flemming, Kress & Flemming, 1996) particularly in the loading direction and vast array of research has been aimed to achieve this goal.

Due to the aggressive nature of some of the aforementioned recycling methods it is difficult to recover continuous fibres and therefore much of the fibre obtained is in a

discontinuous form. The subsequent section will therefore go on to explain various short-fibre orientation methods applicable to the recycled fibres.

3.5.1 Methods for Aligning Short-Fibre

3.5.1.1 Hydrodynamic and Rapid Prototyping

A hydrodynamic method of fibre alignment was employed by Guell and Graham, 1996. This used a 0.16 cm diameter plastic rod to align short-fibres which were immersed into a resin slurry. The plastic rod was passed manually through the un-cured resin slurry and served to 'push' the fibres into alignment. The preform was cured and the mechanical properties (ultimate tensile strength, modulus and ultimate tensile strain) of aligned and randomly oriented short-fibre composite plates were evaluated in samples ranging from 0.4 to 17% fibre volume fraction. The results showed that the aligned short-fibre plates were 90 % stiffer and 100 % stronger than the randomly oriented short-fibre composite plates. The fairly simple method of fibre alignment seems feasible in terms of the strength values achieved however the fibre volume fraction investigated would be too low for an industrial application.

Another fairly simple method of fibre alignment was shown by Peng *et al*., 1999, where the authors assessed the orientation of free-formed short-fibre composites and the effect their orientation had on the strength of the composite produced. Here the authors proposed a method to produce aligned short-fibre composite using a rapid prototyping method (Figure 7). A resin slurry was dispensed through a syringe and the extrudate was use to "write" onto a hot substrate to initiate cross-linking of the resin system. Layer-upon-layer deposition of the slurry was used to build up a 3-D composite component.

Figure 7. A schematic illustration of the 'writing' method used by Peng et al., 1999.

With reference to Figure 7, the epoxy resin system was ‘written’ in conjunction with commercially available milled glass fibres with a nominal length of 0.8 mm and a radius of 5 μm. The authors claimed that it was possible to produce a short-fibre composite component with a 90% degree of orientation with the fibre volume fraction of 18%.

The paper presents a rapid prototyping method but presents no evidence for the testing of the samples produced. The paper also quotes very highly aligned fibres (0°) in some graphs but does not give an indication of the percentages of these and gives a predicted value of fibre volume fraction. It later stated however that ‘the experimental system has fibres with a misalignment of up to 20°’ appearing to not match up with the earlier figures.

3.5.1.2 Extrusion

Hine *et al.*, 1997 discussed a number of methods on how to orientate short fibres including vibration and electric fields. However, their paper investigated the alignment of glass fibres during melt extrusion in polypropylene with the samples created having fibre volume fractions of 8 and 13% respectively. ‘The average fibre length was measured to be 425 μm in the starting pellets, falling to 330 μm after extrusion: with an average fibre diameter of 13.4 μm…’ which gave the fibres a final

aspect ratio of 25. The product was then extruded through two different dies (conical and slit) as well as with a breaker plate in place to see its effect on the final orientation. This was then measured by a 'controlled image analysis system developed at Leeds University'.

The test pieces were then tested mechanically and the highest aligned composites were found to have 85% of their theoretical maximum and high crack resistance with the breaker plate in place close to the mould entrance.

Extrusion methods have also been implemented to achieve short-fibre orientation (Sariglou *et al*., 2004; Zhang *et al*., 1997 and Sanomura & Kawamura, 2003). Sanomura & Kawamura, 2003, aimed to produce well aligned short-glass fibre composite via ram extrusion. Short-fibre polypropylene samples with a short-fibre weight percentage of 10% were produced via ram extrusion.

The Young's modulus of the samples was shown to increase with an increase in the extrusion ratio. This was due to the high fibre orientation increasing the strength of the matrix. The mean fibre length was found to decrease linearly with an increasing extrusion ratio due to fibre breakage. This also showed a small decrease in the Young's modulus due to the fibre breakage.

Although the paper by Sanomura & Kawamura, 2003, showed evidence of being able to create a good degree of orientation, the fibre content in the samples is very scarce and would not have any real application in an engineering environment. Nevertheless higher volume fractions could affect the use of this process due to the fibre breakage noted within the paper and any composites produced would have to be thoroughly analysed.

3.5.1.3 Electrostatics

Other methods of fibre alignment include a process called electrostatics, where the fibres are charged and aligned due to the presence of an electric field. Itoh *et al.*, 1994, investigated the use of electrostatics for the orientation of alumina short-fibre in a liquid. The motion of these fibres was then examined under an optical microscope. These fibres averaging 200 μm in length and 3 μm in diameter were analysed in a fibre-suspension with a small volume of surfactant to help separate the fibres. The experiment set up is shown in Figure 8 and demonstrates how the fibre motion can be recorded in the DC field.

Figure 8. A schematic illustration of the experimental set-up used to observe fibre alignment using electrostatics (Itoh *et al*., 1994).

Unexpectedly, a linear relationship was not found between the angle of the fibre and the field direction (log (tan θ)) which can be seen in Figure 9. This was reportedly due to convection in the liquid affecting the fibres rotation. It was also stated that the irregularity of some of the fibres could have been a contributing factor. The authors also concluded that the orientation of the fibre was dependent on the medium conductivity and the strength of the electric field.

Figure 9. Micrographs showing the orientation of the fibres within two different DC fields after the application of power (a) EO = 0.75 kV/cm, (b) EO = 0.25 kV/cm (Itoh et al., 1994).

Kim *et al.*, 2006, developed another device to form fibre webs with fibre laid onto a rotating disc, where the fibres were orientated circumferentially and/or vertically.

Figure 10 shows a schematic illustration of the fibre orientation and deposition process developed by Kim *et al.,* 2006.

Figure 10. Schematic illustration of the fibre orientation and lay-down process Kim *et al*., 2006.

This device consists of a rotating disc (ground electrode) which collects the fibres from the dispenser and a rod which lays the fibres down. The electrostatic field between the upper and lower electrodes charge and align the fibres perpendicular to the disc. The surface of the fibre collecting area was coated with a layer of adhesive to adhere the fibres to the surface once contact was made. The lay-down rod lays the fibres down in a circular direction. The desired fibre orientation was obtained by controlling the rotating speed of the disc. A series of experiments were conducted to study the effect of the electrostatic field, disc rotation speed and the diameter of the lay-down rod on the distribution and fibre orientations. It was reported that approximate 50+ % of fibres oriented to within 5°.

3.5.1.4 Magnetic and Fibre Spraying

Yamashita *et al*., 1989, demonstrated the feasibility of fibre orientation in a liquid matrix using magnetic fields. The graphite fibres coated with ferromagnetic nickel used in this experiment were of lengths 500, 100 and 3000 μm with diameters of 7.2

and 7.5 μm. The fibres were distributed uniformly and then stirred until the orientation distribution was random. The mixture was placed between a pair of magnetic coils and a magnetic field was applied for 30 minutes to induce fibre rotation in the direction of the magnetic field. The resin was cured for 24 hours at room temperature. The composite produced was then cut into samples to be analysed. The degree of fibre orientation was quantified by the ratio of susceptibilities parallel and normal to the magnetic field, but an overall pattern could also be seen with the naked eye due to the transparent resin. The main point of interest in the study was to identify the critical volume fraction of orientation, which would be the highest volume fraction at which the fibres could be orientated.

Figure 11. Indicates the critical volume fractions (V_f(%)) at which the fibre susceptibility ratio(r) becomes higher and therefore easier to orientate. The figure also shows how the addition of ultrasonic vibration helps with orientation (Yamashita *et al.*, 1989).

With reference to Figure 11, if can be seen that at a fibre volume fraction of 5% orientation became difficult and it was only able to be increased up to ~10% with ultrasonic vibration. These small volume fractions would clearly not be applicable to industry due to their small reinforcement and apparent slow processing speeds to align the fibres. However this method has shown that orientation with magnetism is possible depending on the types of fibres but other methods may be needed.

A paper by Harper *et al*., 2009, outlined the development of a directed carbon fibre preforming (DCFP) method. This method incorporated the use of a fibre spraying/chopping method to align and deposit discontinuous fibres. Initially

continuous fibre reinforcements are fed into the sprayer/chopper where they are cut to a predetermined size (3.175 – 115 mm). Once cut, the fibres are then sprayed onto a perforated tool to form the preform. This substrate's perforation allows airflow through it to hold the fibres in place with a second 'perforated tool' then matched up with the first to compress the products. Hot air is then passed through these perforations to fuse the binder (which is sprayed with the fibres) to create a solid product. This product could then be removed and taken to a mould were the introduction of liquid resin can be achieved to create the composite. The perforated tool was kept at a constant distance of 5 mm with a deposition rate of 0.15 kg/min. The apparatus used in the paper can be found in Figure 12.

Figure 12. A schematic illustration of the discontinuous fibre spraying/chopping method adopted by Harper *et al*., 2009.

Photography was used to measure the degree of fibre orientation once the fibres were deposited onto the perforated tool. Image analysis results showed that a 94 % degree of orientation was possible for 115 mm chopped fibre lengths. However, with the use of a shorter fibre length of 28 mm, only a 38 % degree of orientation was attained. It therefore seems that this method is only effective for the production of 'relatively long' short-fibres (~100 mm). Furthermore, Harper concluded that there were three main mechanisms that caused fibre misalignment, these include; (i) fibre rotation during deposition; (ii) interaction between the fibres being deposited and previously deposited fibres; and (iii) high air velocity present in the chopper/sprayer. The paper also discusses the benefit of aligning the fibres where all of the aligned samples outperformed the randomly aligned structures in a tensile test.

The paper was able to show high degrees of alignment in the 'longer' discontinuous fibres. However, the degrees of alignment were stated to decrease as the amount of layers increased due to the methods outlined by the author. The quality of the composite is also difficult to decipher with the test method analysis, so more work is required.

3.6 Properties of Short-Fibre Composites

White & Abdin, 1985, investigated the properties of aligned short carbon fibre composite in both flexure and torsion. The specimens for these tests were in the form of plates, beams and rods being made from different procedures but all with the same aim i.e. to control alignment and volume fraction.

Prepreg sheets of aligned carbon fibre with varying lengths of fibre ranging from 0.25 to 3 mm were made via a centrifugal process. The other forms of specimens were created via a moulding process outlined in Abdin's paper 'Dynamic properties of some carbon fibre reinforced plastics'. The experimental results for the shear modulus and shear loss modulus of the aligned short-carbon fibre rods showed that shear modulus increased and that shear loss modulus decreased with increasing fibre length. It was also found that as the fibre length decreased at a set fibre volume fraction the loss factor was increased, however the modulus remained relatively high in comparison to carbon reinforced composites reinforced with continuous fibres at the same volume fraction (Figure 13).

Figure 13. The effect on the material loss factor with fibre volume fraction shown across increasing fibre lengths.

Fu *et al*., 2000 were able to show that an experimental relationship between failure strain, fibre volume fraction (V_f), fibre length and the fibre radius exists. These parameters all help to determine the tensile strength of the composite. Increasing fibre volume fraction has shown to increase strength up to a limit; however this increase can also be shown to encourage fibre misalignment (Yamashita et al., 1989). Increasing fibre length has been shown to aid orientation (Harper et al., 2008) and increase strength. However the fibres must be short as possible so the preform can be draped in the manufacturing process but long enough to yield the best possible mechanical properties (Flemming, Kress & Flemming, 1996).

A hydrodynamic method was used in Papathanasiou *et al*.'s small-scale pilot facility to align short-carbon fibres in an epoxy matrix. The orientation was achieved when a "cylindrical element placed in a flowing suspension giving rise to a localised elongational flow in its wake, where the fibres tend to become aligned irrespective of the orientation at which they approach the mesh" (Papathanasiou *et al*., 1995). Computations revealed that randomly located but aligned fibres significantly improve the effective composite stiffness, compared with randomly placed and randomly

oriented fibres. For the composite produced using the hydrodynamic technique, the mechanical properties showed that it was 90 % more effective at stiffening the composite when the fibres were aligned than in the randomly aligned sample. There was good agreement between computations and experimental data.

Mouhmid *et al*., 2006, were also able to show that the use of short-glass fibre, in a non-reinforced polymer, was effective in increasing the tensile strength and elastic modulus of the material. Polyamide 66 pellets and chopped E-glass bundles were compounded using a single screw extruder with varying weight contents of glass fibre (0, 15, 30 and 50% (wt)).

Figure 14. The stress-strain curves for composites of varying glass fibre content (wt%).

Mouhmid *et al*., 2006, were able to show that the increase in reinforcing fibre even if discontinuous can be beneficial to a polymer material. As also shown by Fu *et al*., 2000 the effect is shown to increase with increased fibre volume fraction although Fu *et al*., 2000 showed this was only beneficial up to a point. The paper does mention orientation as a key factor in the use of short-fibre composites but gives no mention of measuring this factor and therefore cannot quantify it. The strain rate studied seemed to have a minimal effect on performance however temperature was shown to lead to more ductility and less stiffness.

In relation to the aforementioned section on the recycling of composites it is necessary to look at the properties achievable from the use of these types of fibres.

Generally the fibre produced from these types of degradation does result in a discontinuous fibre and as previous research has shown (Papathanasiou *et al.* 1995, Flemming, Kress & Flemming, 1996, Fu *et al*., 2000) these fibres are more effective in a composite when orientated in the loading direction. The literature seems to be limited in that short fibre composites are scarcely used in load bearing applications. However as shown by Taib, 1998, the key parameters in a short-fibre composite are (i) fibre dispersion, (ii) fibre matrix adhesion, (iii) fibre aspect ratio, (iv) fibre orientation, and (v) fibre volume fraction, therefore if all these parameters can be maximised the use of short-fibre composites can be expected to rise, especially due the series of EU directives.

4. Experimental

4.1 Materials

Three types of waste materials were investigated during this study: (i) waste slittings (ii) direct-loom waste and (iii) 4.5 mm chopped E-glass fibres. During this study, continuous E-glass fibres were used as reference materials.

4.1.1 *Direct-loom Waste*

The direct-loom waste (DLW) material was acquired during the weaving of a continuous E-glass fibre fabric. During the weaving process, the DLW is fabricated from a trimming process which creates a straight-edge for the woven glass-fibre fabric (as well as cutting it to specific widths). The direct-loom waste material consisted of glass-fibres coated with a starch/oil binder to aid the weaving process, held in place by either 5 or 7 cotton support threads.

A photograph of the "production" of the direct-loom waste is shown in Figure 1A. Figure 1B shows a magnified view of the direct-loom waste.

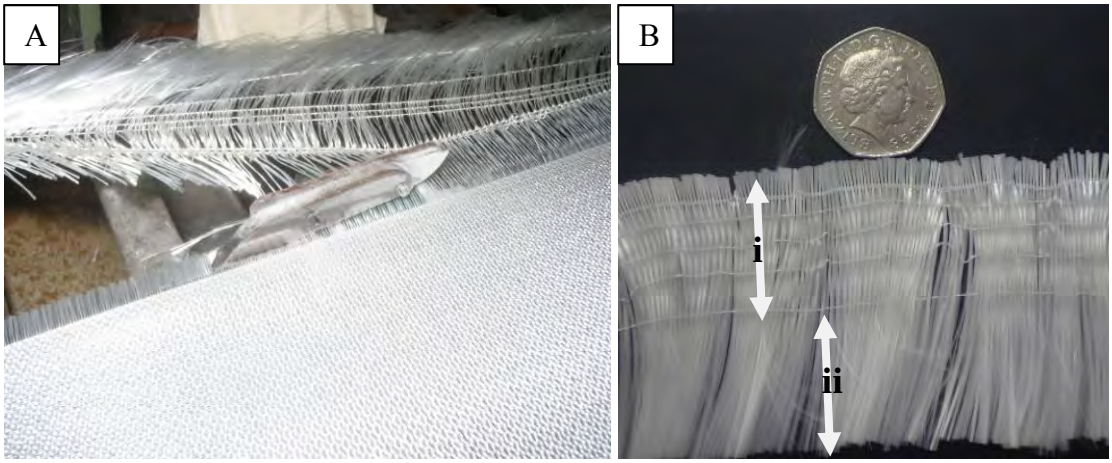


Figure 15: (A) A photograph of the direct-loom waste being produced; and (B) A magnified view of the as-received direct-loom waste. The arrow denoted (i) in Figure 1B indicates the distance between the top of the fabric to the final supportive cotton strand, which is approximately 35 mm. The arrow marked as (ii) in Figure 1B indicates the distance from this final supportive strand to the end of the loose fibres, which is approximately 45 mm in length.

4.1.2 *Waste Slittings*

The waste slittings are acquired after the "finishing" phase of the aforementioned weaving process which produced the DLW. In the current case, the fabric is silane treated, heat-cleaned and coated with a resin sealant to help consolidate or stiffen the fabric. The waste slittings are trimmed from the edge of the fabric to produce a fabric of the required width. The fabrics are trimmed just before it is wound on to a cardboard tube. An image of the waste slittings is shown in Figure 16.

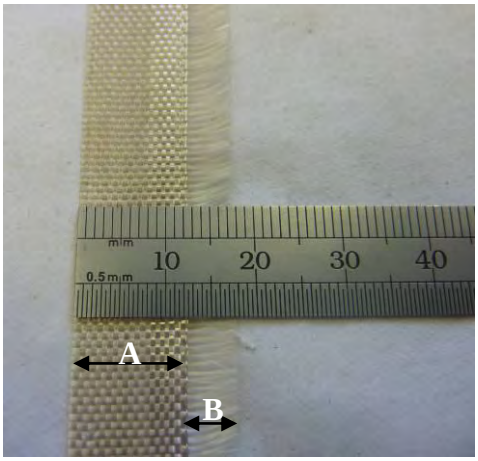


Figure 16. A Photograph of the waste slittings where, the gold coloured woven section of the waste slittings, indicated by arrow A, is approximately 12.5 mm wide. The 'frilly' edge section of the fabric, shown by arrow B, measures approximately 5 mm in width.

4.1.3 *Pre-chopped E-glass Fibres*

Pre-chopped 4.5 mm E-glass fibres, with an epoxy compatible sizing (ChopVantage HP3786), were also selected for use in a feasibility study to manufacture aligned short-fibres. These pre-chopped fibres were heavily bound to hold the individual filaments together; creating small individual bundles as depicted in Figure 17.

Figure 17. The 4.5mm pre-chopped E-glass fibres used during this study.

4.1.4 Resin Systems

An epoxy resin system (LY3505/XB3403), supplied by Huntsman Advanced Materials, was used during all filament winding trials. Conversely, all the short-fibre trials were undertaken using a (HexPly® 913) epoxy matrix film supplied by Hexcel.

4.1.5 Reference Materials

Continuous E-glass fibres (1084 K19 EC15, 1200 Tex) were used as the reference material during this study. When used as a comparison with the pre-chopped waste fibres, the continuous E-glass fibres were impregnated with the HexPly® 913 resin matrix film. The continuous E-glass fibres and continuous waste fibres were impregnated with an LY3505/XB3403 epoxy resin system when processed as filament wound tubes.

4.2 Composite Production

4.2.1 Filament Winding

A schematic illustration of conventional filament winding is shown in Figure 18.

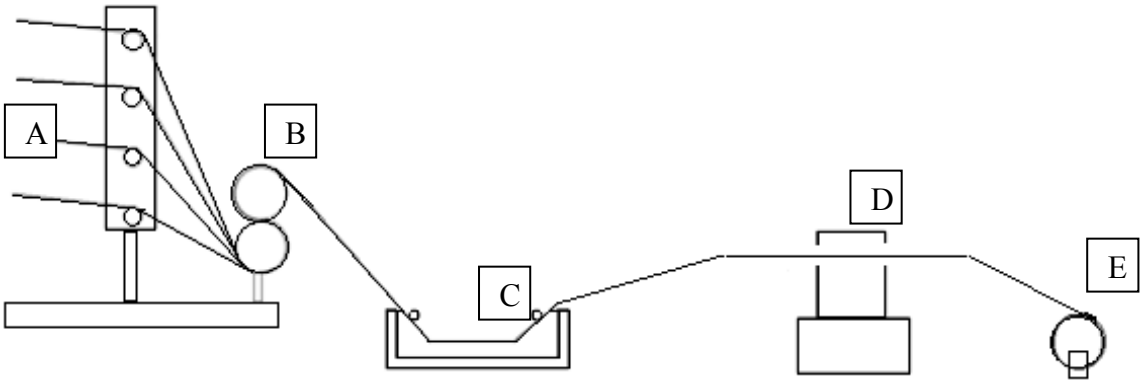


Figure 18. A schematic illustration of conventional filament winding. The key components of the process include (A) E-glass fibre bundles, (B) Tensioning system, (C) Resin bath, (D) Traversing carriage and (E) Mandrel.

With reference to Figure 18, fibres (A) are pulled from creels/bobbins through a tensioning system (B). These fibres then pass through a large resin bath (C), which is approximately 5 L in capacity, where the fibres are impregnated. These impregnated

fibres then pass through a traversing carriage (D) where they are then wound around a mandrel (E) to create the final product. Whilst this process is able to effectively produce composite components, there are many problematic issues associated with this method. These include:

(i) the resin and hardener are weighed out in the correct stoichiometric ratio and mixed manually, which can lead to inadequate mixing and/or incorrect proportions of the components;

(ii) the health aspect for the individual workers with regards to the inhalation of solvents from this open process;

(iii) the volume of the resin bath is also an issue as the resin can cross-link in the bath. This is likely to hinder the production process as well as producing large waste volumes of resin; and

(iv) the high volume of solvent needed to clean all resin contaminated pieces of equipment including the resin bath itself is impractical.

Due to the issues mentioned above, a modified version of this process termed 'clean filament winding' was developed. A schematic illustration of the clean filament winding process is shown Figure 19.

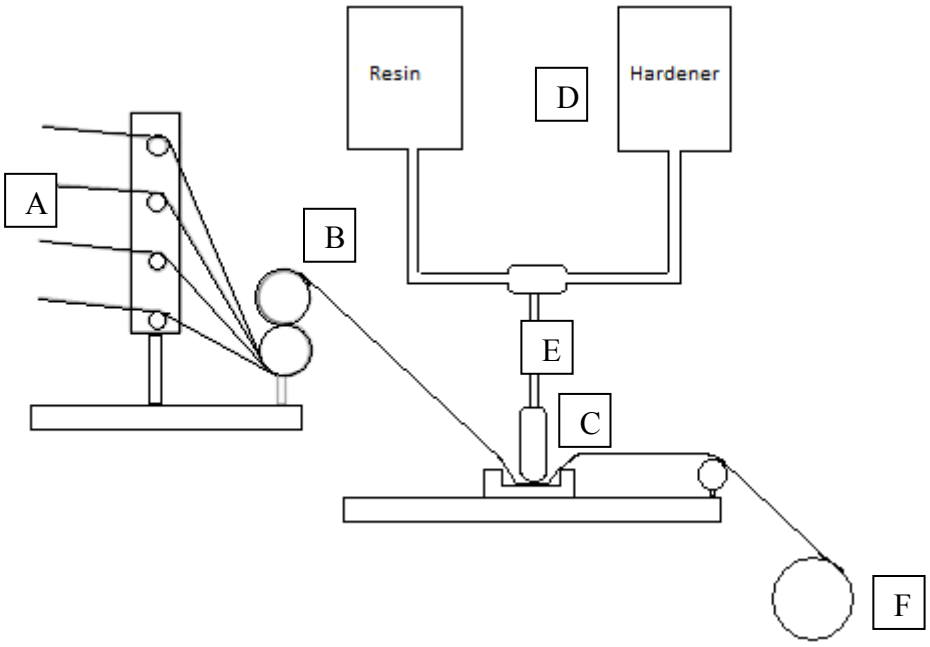


Figure 19. A schematic illustration of clean-filament winding. The highlighted components include: (A) E-glass fibre bundles, (B) Tensioning system, (C) Impregnation unit, (D) automated pumping system, (E) Static mixer and (F) Mandrel.

With reference to Figure 19, E-glass fibres (A) are pulled from creels and through a tensioning system (B). However, instead of passing through a resin bath they now pass through a custom-built impregnation unit (C) mounted upon the traverse arm of the filament winding machine. This impregnation unit is supplied with resin and hardener in the correct stoichiometric ratio by automated precision gear pumps (D), which was incorporated to improve the accuracy and negates the need for any manual mixing and pouring. The resin and hardener are in two different containers and are not mixed until they impinge at the static mixer (E) for more economical use of materials. The unit is also reduced in size (approximately 50 ml) which therefore reduces the amount of standing resin and the opportunity for resin to cross-link. The smaller unit also reduces the amount of solvent needed to clean any resin-coated equipment. Since the unit also reduces the emissions released into the air, there is a reduced exposure for the workforce and generally a more environmentally cleaner system. Due to the aforementioned environmentally friendly improvements, this method was used for the processing of the continuous waste fibres investigated in this study.

4.2.1.1 *Production of Filament Wound Tubes*

In this study, four types of tubes were produced, these included tubes produced from: (i) waste slittings; (ii) direct-loom waste; (iii) E-glass fibre, manufactured in the laboratory and (iv) E-glass fibre, manufactured on-site. The tubes manufactured on-site are created via the retrofitting of the clean filament winding system on to a generic filament winding system. These tubes in this study were manufactured using the parameters denoted in Table 1.

Table 1. The two key parameters that were used to manufacture the filament tubes in this study via a clean filament winding technique. The winding speed refers to the fibre haul-off rate. The 'pitch' refers to the traverse distance in the horizontal direction, to lay fibre tows or ribbons, in a sequential fashion.

Material	*Winding speed (m/min)*	*Pitch (mm)*
Glass Fibre (CFW GF)	10	4
Glass fibre On-site (Fast speed)	21	4
Glass fibre On-site (Normal speed)	7	4
Waste Slittings	2.5	7
Direct-loom Waste	1	~7

Following initial processing of the tubes, the uncured samples were transferred to a mandrel rotation system to initiate curing at room temperature and produce a smooth surface finish. A photograph of the mandrel rotation mechanism is shown in Figure 20.

Figure 20. Photograph of the mandrel rotation system.

Following the aforementioned initial curing process; the filament wound tube was placed into an air-circulating oven for 6 hours at 70 °C. Following curing, the

filament wound tubes were extracted from the mandrel using a hydraulic ram, as shown in Figure 21.

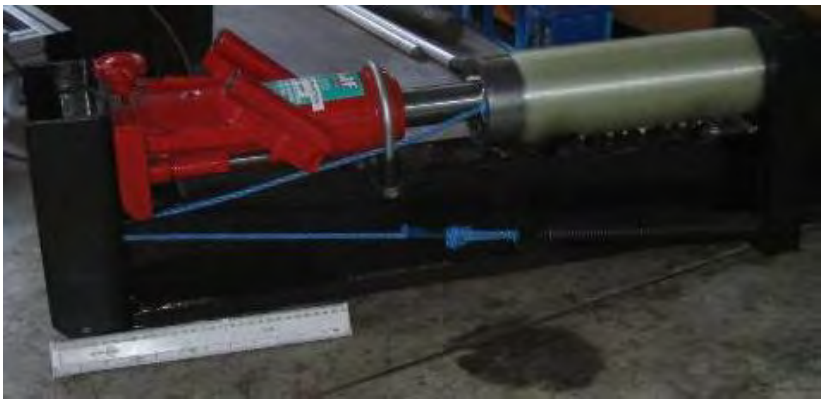


Figure 21. An image of the hydraulic ram used to extract the filament wound tubes from their mandrels; a 30 cm rule indicates the scale.

4.2.2 *Short-fibre vibration-based delivery system*

To provide an alternative processing method for discontinuous fibres, an acoustic vibration-based delivery system was developed. This method involved vibrating the 4.5 mm fibres along the length of a steel foil flute with a loud speaker system. A photograph of the original speaker and steel flute is shown in Figure 22.

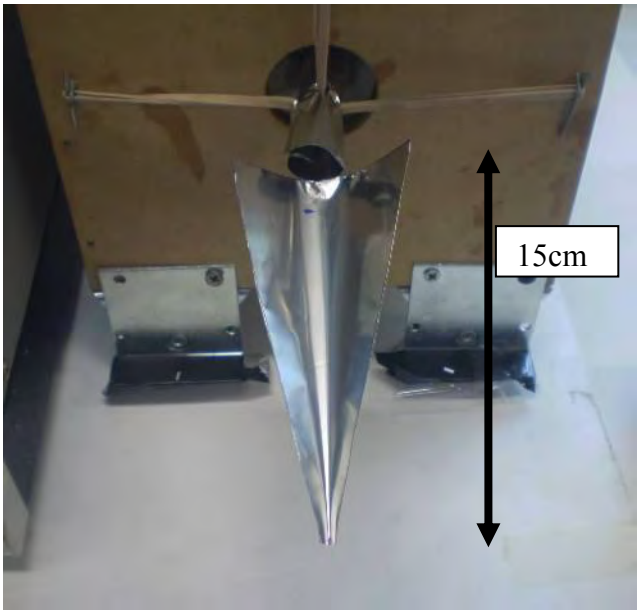


Figure 22. Photograph of the short-fibre orientation method including a speaker enclosure and a steel foil orientating flute.

The viability of the vibration-based short-fibre delivery and orientation system was demonstrated by previous researchers (Wait et al., 2010). However, no system had been developed for production of aligned short-fibre composites. In the current study, the speaker enclosure was initially mounted on a Hounsfield tensometer to enable the lateral movement of the speaker. The initial set-up for the short-fibre orientation method, referred to as 'Protoype I', is shown in Figure 23.

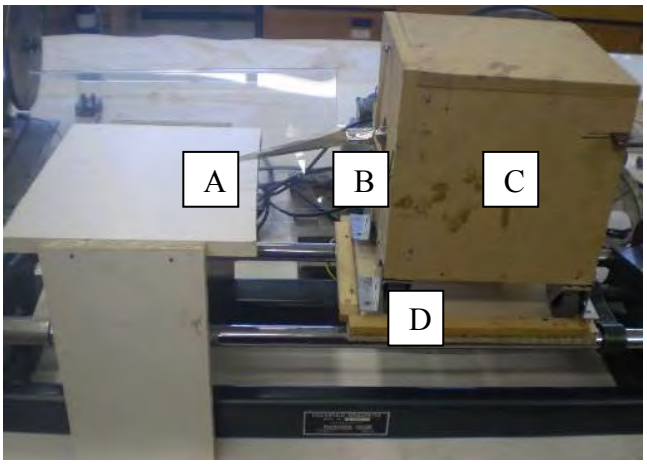


Figure 23. Prototype-I, the initial speaker enclosure mounted on a Hounsfield tensometer, where (A) is the deposition platform, (B) is a steel foil orientating flute, (C) is the speaker enclosure and (D) is the Hounsfield tensometer.

With reference to Figure 23, 4.5 mm chopped E-glass fibres were manually deposited onto the steel foil aligning flute (B). The flute is vibrated by the speaker (C), to enable orientation and transportation of the short-glass fibres along its length. The fibres are the deposited on to the platform in (A). Multiple channels of fibres were achieved via the lateral movement of the speaker using the Hounsfield tensometer in (D).

Although this method allowed movement of the system, the arrangement was very basic and was unable to create a high quality component due to a number of issues. The issues associated with this method include: (i) the reliance on operator competency; (ii) lack of consistency; and (iii) time consuming manner to produce one plate.

Therefore, a second prototype was developed to try to eradicate the previous problems and to create a more dependable orientation method. A schematic illustration of Prototype-II can be found in Figure 24.

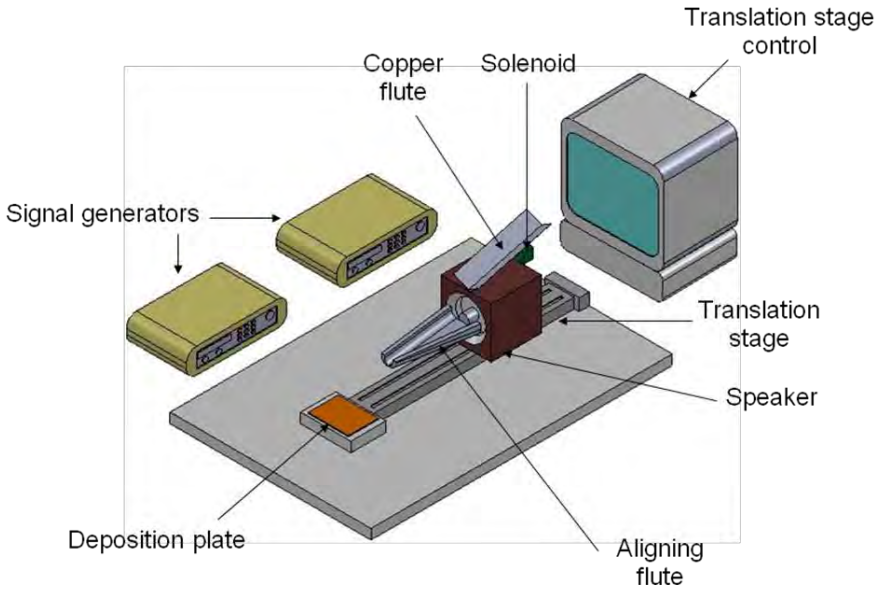


Figure 24. A schematic illustration of Prototype-II, the two-stage short-fibre vibration-based delivery system.

In Prototype-II, a specified mass of short-fibres were deposited onto the preliminary aligning copper flute. This flute (which was vibrated by a solenoid) transported the short-fibres along its length and onto a custom-made aligning flute. Once deposited on the aligning flute, the fibres were then orientated in the flow direction along the length of the flute. This aligning flute was excited by a vibration, supplied from an audio speaker system; these vibrating forces, in combination with the custom-made flutes profile, caused the alignment of the short-fibres. Once aligned, the fibres were then deposited onto a resin substrate which was mounted on a linear translation stage. The linear translation stage was programmed to allow for the deposition of sequential channels of aligned short-fibres onto a platform.

An in-depth description of the main constituents of Prototype-II is presented below:
(a) Preliminary alignment copper flute: In order to control the delivery rate of the short-fibres a preliminary aligning flute was employed. This was used to solve the issues of inconsistent fibre deposition during production with Prototype-1. This flute was vibrated by a solenoid (Series 44A) which was powered by a signal generator.

(b) Custom-made alignment flute: To produce highly aligned short-fibre composites a custom-made aligning flute was incorporated into Prototype-II. This flute, as shown

in Figure 25, was used to align short-fibres and deposit them in a controlled and concise manner onto a resin substrate. The aligning flute (constructed from stainless steel foil) had a converging profile i.e. the entry point was wider than the exit point. As a result, the fibres were directed along a thin section of the flute; which aligned the fibres in the flow direction. This flute was also used in conjunction with a speaker system. The speaker system supplied a vibrating excitation force to the flute and assisted in the transportation of the fibres along the length of the flute. The combination of the vibrating forces and the flute profile allowed for the transportation and delivery of aligned short-fibres onto a resin substrate.

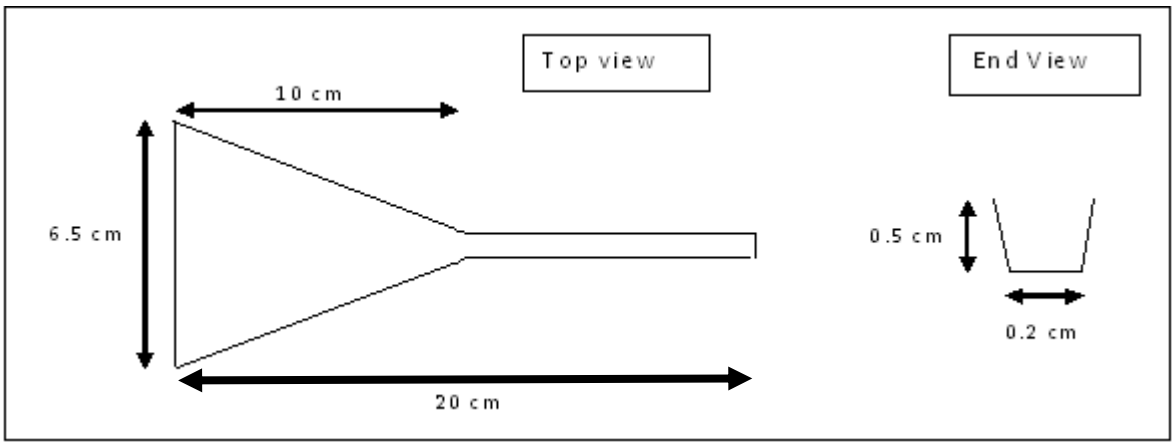


Figure 25.Schematic illustration of the profile and dimensions of the custom-made aligning flute profile.

(c) Audio speaker system: As previously mentioned, a speaker system was used to transport and align the short-fibres along the length of the aligning flute. The audio speaker system (Kenwood KFC 1051s) was powered by a signal generator and was housed inside a custom-made protective casing; which protected the speaker whilst allowing it to be strategically and safely positioned during the production of highly aligned short-fibre composites.

(d) Linear translation stage: A linear translation stage was incorporated to allow for the deposition of multiple 'channels' of short-fibres. This unit (supplied by Rexroth™) allowed for repetitive and automated movement of the aligning

components. The linear actuator had a translation speed of 20 mm/sec over 50 cm range.

Prototype-II helped to solve the issues that were associated with Prototype-I. However, due to the movement of the speaker system on the linear translation stage, the composites produced were limited in size, at roughly 30cm x 10 cm. Therefore, the system had to be developed further to create a larger composite of 30 cm x 30 cm.

Prototype-III was developed incorporating the same components as discussed earlier but these were rearranged in order to maximise production size. This was achieved by mounting the deposition platform on the linear translation stage and providing a raised platform on which the speaker system would be placed. A photograph of Prototype-III is shown in Figure 26.

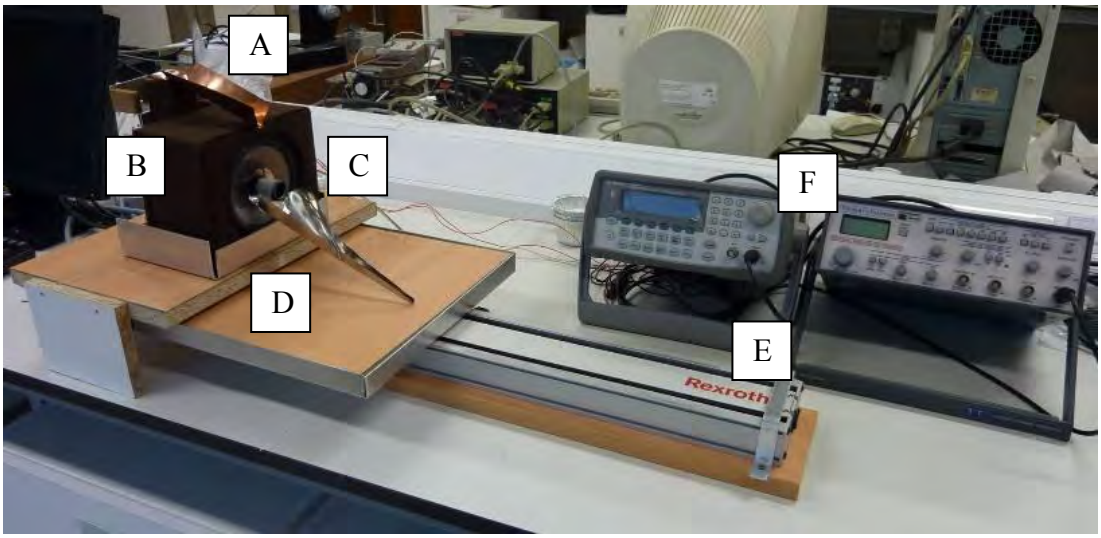


Figure 26. Prototype III allowing for production of 30cm x 30cm short fibre composites. The highlighted components are: (A) the primary alignment copper flute; (B) the speaker enclosure; (C) the custom-made aligning flute; (D) the deposition platform; (E) the linear translation stage; and (F) Two signal generators used to power the speaker and the primary alignment copper flute.

The new arrangement allowed for production of the much larger 30cm x 30 cm short-fibre composite preform. However, this still relied on manual movement of the speaker system in the horizontal direction, which again made it susceptible to human error. Therefore the final prototype incorporated both the linear translation stage and the Hounsfield tensometer to enable horizontal and vertical movement of the system

to further improve reliability and stability. A schematic illustration on Prototype-IV can be found in Figure 27.

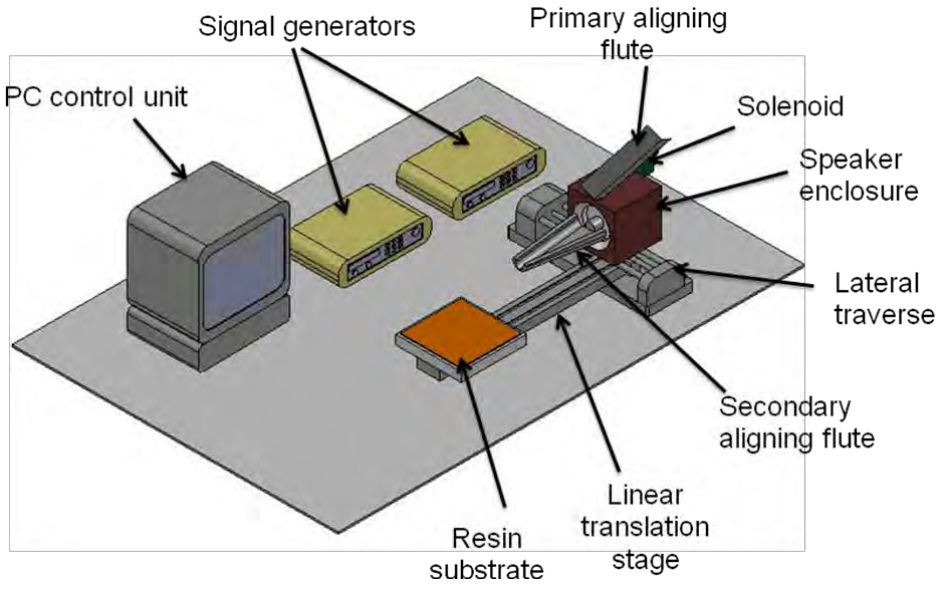


Figure 27. A schematic illustration of Prototype-IV

***4.2.2.1** Short-fibre Composite Production*

Prototype-IV was used to deposit highly aligned 4.5mm short-fibres onto a 30 x 30 cm sheet of Hexply™ 913 resin film. This process consisted of: (i) stimulating the solenoid at 2.05 Hz; (ii) vibrating the speaker at a frequency of 50 Hz with a voltage of 1 Volt peak-to-peak (VPP); (iii) moving the translation stage at 20 mm/sec and (iv) moving the Hounsfield 1.5 turns once a run was completed. This method was repeated eight times to form eight layers of aligned short-fibre prepregs. Additional layers of the Hexply™ 913 resin film were transferred to the short-fibre prepregs, as required, to ensure complete coverage of the individual short-fibres. The individual preforms were laminated, stacked uni-directionally and consolidated using conventional autoclaving procedures. In addition to this, continuous E-glass fibre (1200 Tex) composites were made, incorporating the same Hexply™ 913 resin film and autoclaving procedures.

The laminated short-fibre prepregs and continuous E-glass prepregs were then processed in an autoclave (IBBC Quicklock Thermoclave) using conventional

vacuum bagging procedures. The processing schedule consisted of curing the samples at 125 ^{o}C with a pressure of 0.69 MPa for 60 minutes. The temperature was ramped at 2 ^{o}C per minute up to the final temperature 125 ^{o}C.

4.3. Evaluation of Composites

4.3.1 Mechanical Properties

4.3.1.1 Clean-filament Wound Tubes

Following manufacture, the tubes were assessed for their mechanical properties to allow direct comparison of the composites. Five samples from six tubes of each type of filament wound tube were used per test method. Two conventionally produced industrial standard E-glass fibre filament wound tubes were also tested using the methods outlined below.

4.3.1.1.1 *Inter-laminar shear strength test*

The inter-laminar shear strength testing was carried out in compliance with ASTM standard D2344/D2344M. The recommended length of the test specimen was determined using a span to depth ratio of 5:1. Samples were cut from the tubes and placed on a 3-point bend test fixture which can be seen in Figure 28. The fixture was mounted on an Instron™ 5566 machine. The cross-head speed was set at 1 mm/minute with data acquisition collected every 500 ms via a data acquisition system that was connected to a personal computer.

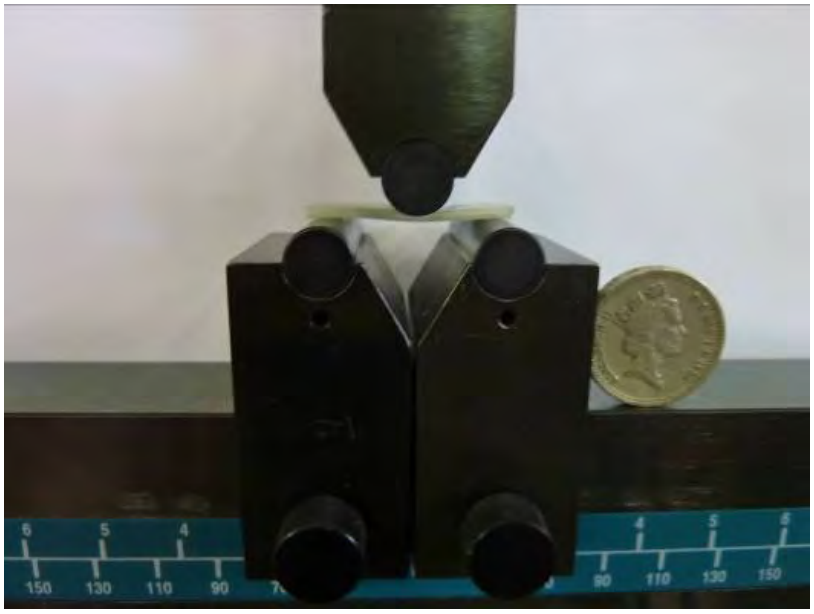


Figure 28. A photograph of the experimental set-up for the inter-laminar shear strength test. This test was completed in accordance with ASTM standard D 2344/D 2344M.

4.3.1.1.2 Hoop tensile strength

Rings of 20 mm width were cut for hoop tensile testing of the filament wound tubes. Notches were introduced on the samples to determine the failure point of the composite. The notches were introduced symmetrically with a 3.2mm radius. All samples were tested to failure with the failure point of the sample noted. A photograph of the experimental set-up can be found in Figure 29.

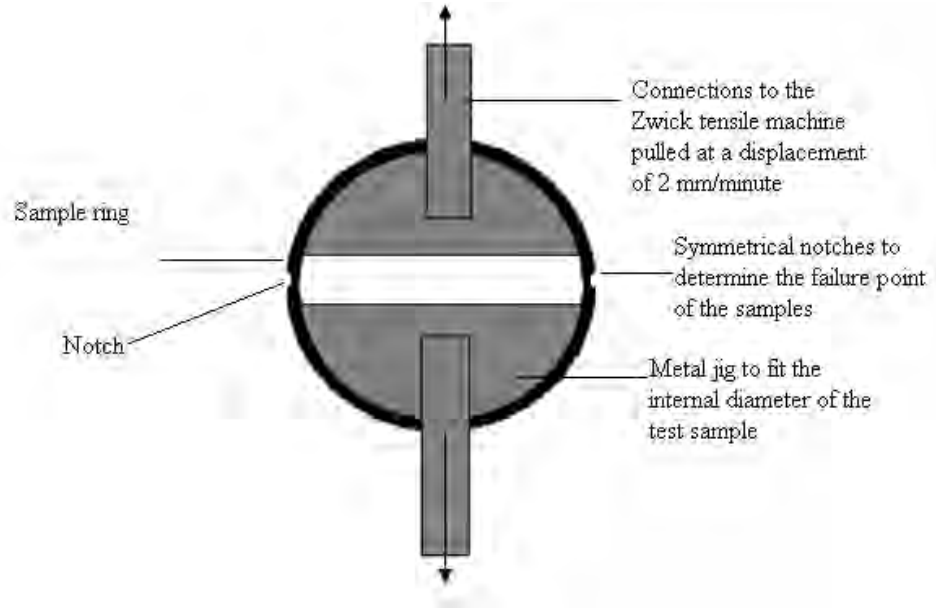


Figure 29. A photograph of the experimental set-up for the hoop tensile strength test. The test was completed in accordance with ASTM standard D2290 at a displacement of 2 mm/min using a Zwick 1484 testing machine

4.3.1.1.3 Lateral Compression Strength

The lateral compression test was performed in conjunction with a paper reported in the literature by Gupta & Abbas (2000). Five samples were cut from six sets of each of the four tube variations. The thickness and width of the test specimens were obtained using a pair of digital callipers. In addition, cardboards tubes were tested by this method, this was to enable a direct comparison of the composite tubes produced in-house with those currently used commercially (to wind on the woven fabrics) by an industrial partner. A schematic illustration of the set-up for this test method can be found in Figure 30.

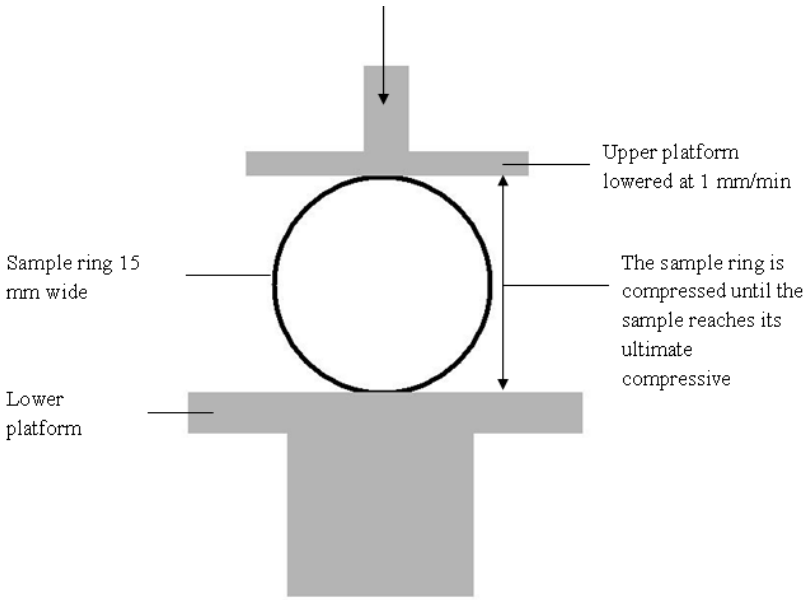


Figure 30. A photograph of the lateral compression testing conducted in this study adopted by Gupta & Abbas, 2000. The test was conducted on an Instron™ 5566 machine at a crosshead speed set at 2 mm/minute.

4.3.1.2 Short-fibre Composites

Following production, the composites were assessed, to determine: (i) mechanical properties; (ii) degree of fibre alignment; and (iii) physical properties.
To assess the mechanical properties of the two composite plates (continuous and discontinuous) they were subjected the following mechanical tests.

4.3.1.2.1 Tensile testing

Five 20mm thick samples were cut from the ~1.5mm thick composite plates, polished and end tabbed using conventional procedures to provide a gauge length of 250 mm all in accordance with ASTM D3039. The samples were then mounted with TML FLA-6-11 strain gauges and tested using a Zwick Z100 fatigue testing machine.

4.3.1.2.2 Flexural Strength

Flexural strength of the composite plates was determined via a four point bend test rather than a three point bend test to increase reliability. A schematic illustration of the set-up for the four point bend test can be found in Figure 31.

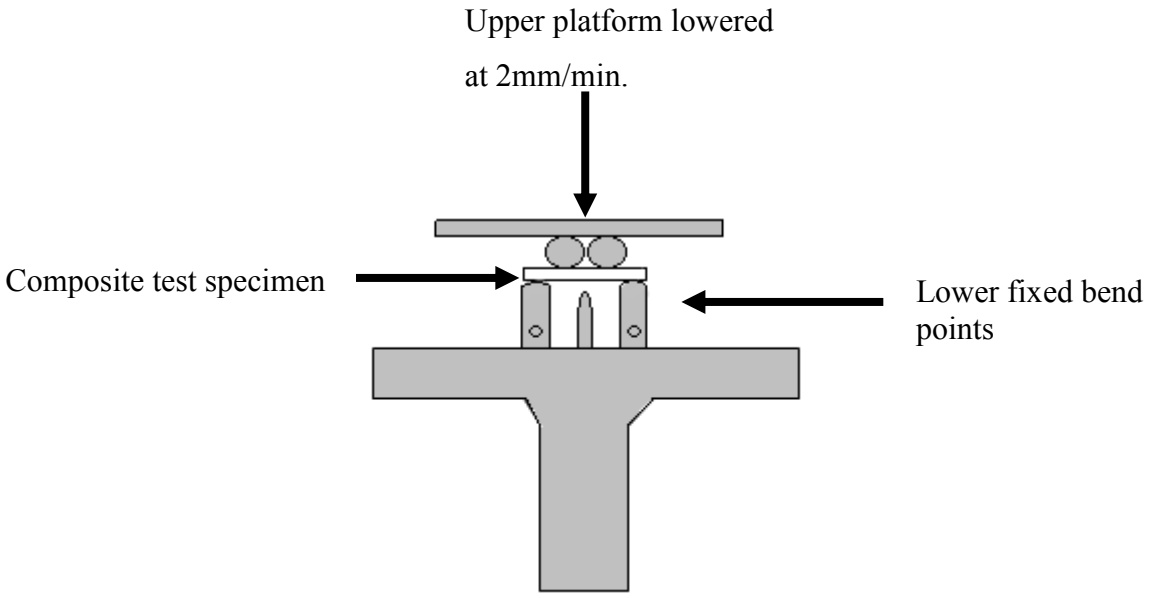


Figure 31. The four-point bend test fixture used to test the short fibre and continuous E-glass fibre composite plates. This was completed in accordance with ASTM D6272 which involved cutting the samples in relation to the thickness of the plate, where the length of the sample was required to equal 32 times its depth and the width was required to equal 10 times the depth. The dimensions of the samples were therefore 45mm in length and 14mm wide.

The five short-fibre samples were tested until failure. However, due to the greater flexibility of the continuous fibre samples, they were taken up to 100 MPa. This enabled the modulus of the samples to be calculated in accordance with D6272, without the samples falling out of the test fixture.

4.3.1.3 Short-fibre Degree of Alignment

The term 'degree of alignment' is defined as the percentage of short-fibres that were within $\pm$ 10^{o} of the desired fibre orientation direction. Images were taken of sections of the short-fibre composite using a digital camera. The orientation of the fibres was measured manually using a protractor from the images taken. The individual fibre bundles were highlighted via a fine line drawn along their lengths in order to compare them to the desired orientation direction (Figure 18).

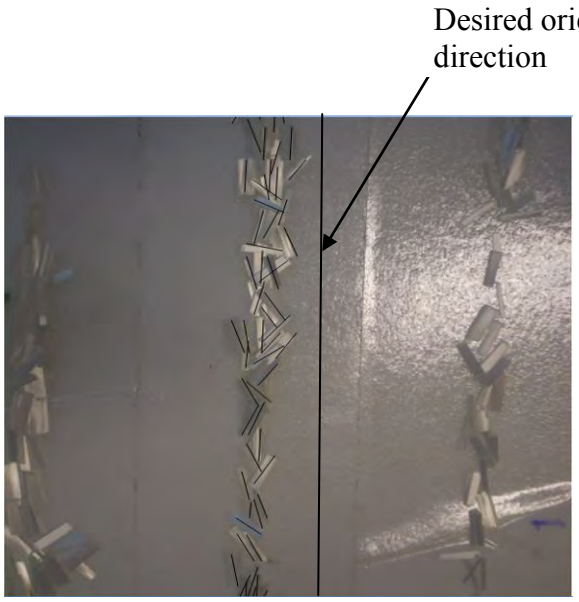


Figure 32. A photograph of the initial fibre deposition with the fibres highlighted to assist in the calculation of the orientation. These highlighted fibres were then measured using a protractor to calculate their degree of orientation.

The sum of these measured angles of orientation was then noted for each category (i.e. <10°, 10-30°, >30°). It was then possible to gain an overall percentage of the aligned fibres in the targeted <10° category.

4.3.1.4 Physical Composite Analysis

4.3.1.4.1 Fibre Volume Fraction

The fibre content in the composites was measured from five samples of each composite variation. The samples measured 2 cm x 2 cm in size, where initially the

weight of the samples was determined using an analytical balance (Ohaus™ Analytical Plus).

Once the weight of the samples was measured, they were transferred to a muffle furnace to pyrolyse the resin. This test was completed in accordance with ASTM D2584 which required the samples to be heated at 565 °C for six hours. The samples were removed from the furnace and allowed to cool before being reweighed. The fibre volume fraction of the samples was calculated using Equation 1:

$$Lw\,\% = \left[\frac{(W_1 - W_2)}{W_1}\right] \times 100\ \% \qquad [1]$$

where, W_1 = Sample weight (before burn-off), W_2 = Weight of residue (after burn - off) and Lw% = the loss in the weight of the sample.

4.3.1.4.2 Void content

The void content was determined using the same samples used to calculate the fibre volume fraction. The void content however was determined by using Equation 2 and 3 whereby the measured weight was compared to a theoretical weight based on the materials used;

$$T = \frac{100}{\left(\frac{R}{D} + \frac{r}{d}\right)} \qquad [2]$$

where, R = weight of the resin in the composite, (%), D = density of the resin, r = weight of the reinforcement in the composite, (%), and d = density of the reinforcement.

$$V\,(\%) = 100\ \frac{T_d - M_d}{T_d} \qquad [3]$$

Equation 3 was used to determine the void content present in the composite samples where, T_d = theoretical density (calculated using Equation 2) and M_d = Measured density of the composite.

4.4 Image analysis

Samples from each composite were mounted in Epofix™ resin and hardener in the ratio of 5:1. Once cured, the samples were initially hand polished with 800 grit paper to create a level surface. These samples were then progressively polished with 1200, 2400 and 4000 grit silicon carbide abrasive paper discs until the fibres appeared clear and smooth. The final stage involved a 3µ polishing solution and an M-Dac™ polishing cloth. These samples were then cleaned and dried before being inspected using a Leica™ optical microscope. The images were taken at x5, x10 and x20 magnifications to assess the composite materials structure.

4.5 Scanning Electron Microscopy

Following mechanical testing, selected regions of fracture surfaces of the composite samples were mounted using clips and adhesive carbon to an aluminium stub. The base of these samples were painted with a conducting silver dag and then gold coated using a Polaron™ SC7640 sputter coater. The images were acquired using a JEOL™ 6060 scanning electron microscope at 20 kV.

5. *Results and Discussion*

5.1 Materials

5.1.1 *Direct-loom waste*

Figures 1A and B show typical images of sections of the as-received direct-loom waste.

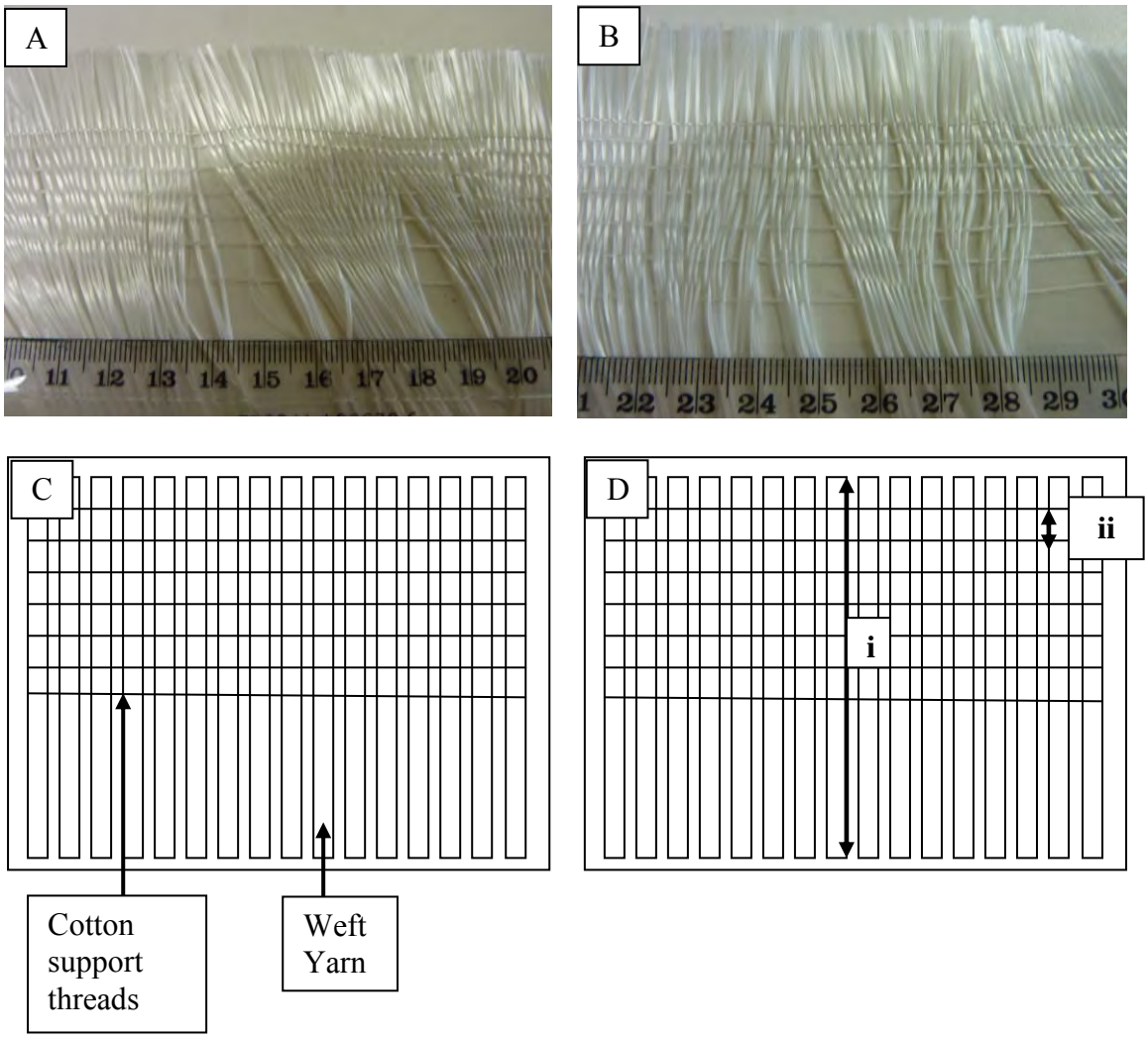


Figure 33 (A-D). (A) and (B) shows photographs of typical sections of the as-received direct-loom waste. (C) Schematic illustration of the ideal structure of the direct-loom waste. (D) Schematic illustration of the direct-loom waste with appropriate dimensions, where 'i' indicates an average fibre width of approximately 80 mm and 'ii' relates to the average width of approximately 5 mm between the seven supporting cotton threads.

With reference to Figure 33A and B, it is clearly apparent that the relative spacing of the weft fibres is highly variable. This was a common feature of the direct-loom waste material. This inherent inconsistency in the as-received state of the direct-loom waste means that the quality of the filament wound tubes is also likely to be variable.

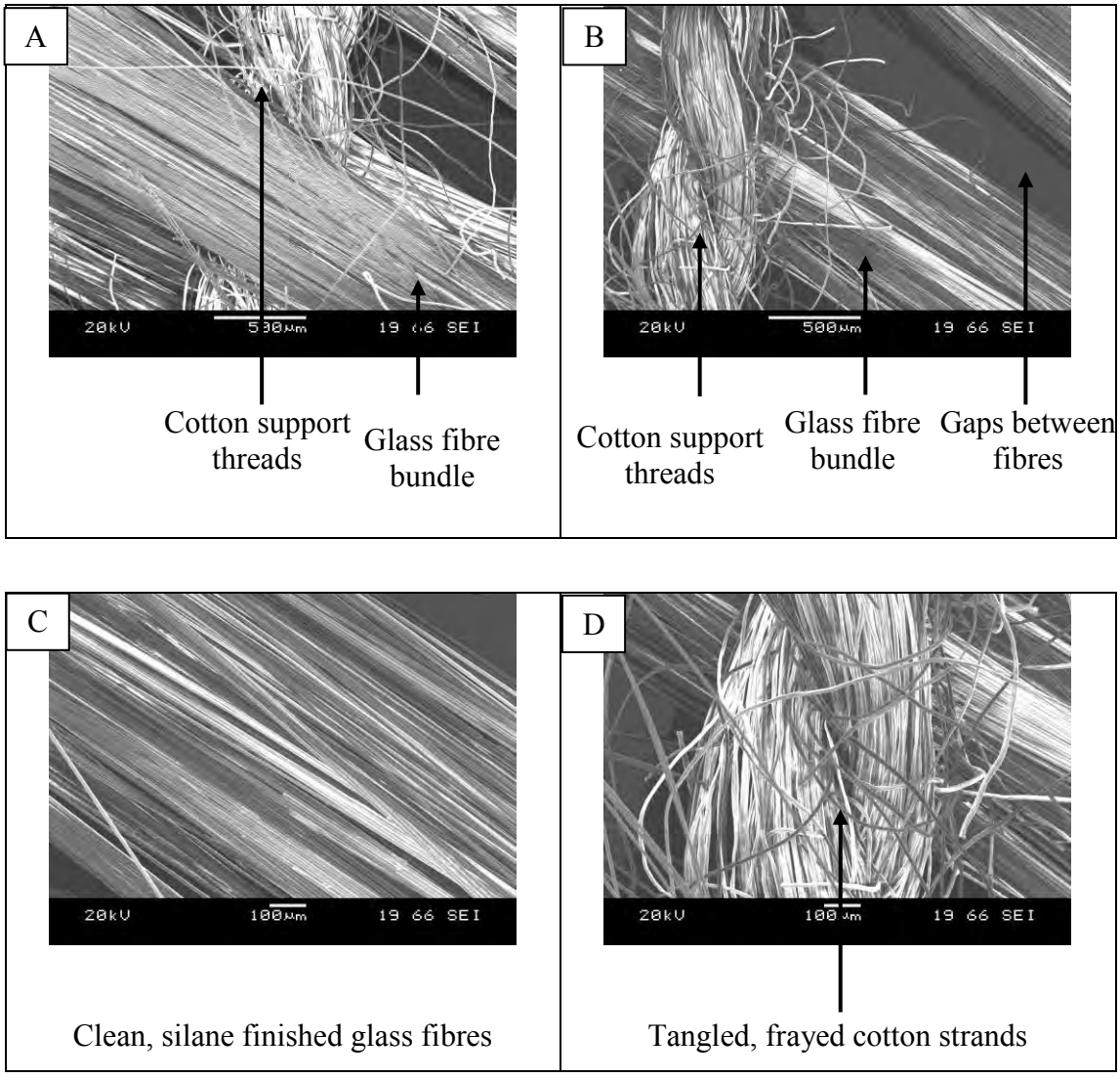


Figure 34 (A-D). (A and B) Magnified images of the direct- loom waste obtained via scanning electron microscopy. (C and D) Magnified views of the weft glass fibre yarns and cotton support threads present in the direct-loom waste.

From reviewing Figure 34 it can be appreciated that the frilly/fluffy nature of the direct-loom waste will affect the overall quality of the filament wound tubes. The cotton 'support' structures within the direct-loom waste can be seen to hold the glass-

fibres in place very loosely. The cotton strands are also frayed and tangled. This was the primary reason as to why the glass fibres were unable to retain their spatial orientation as the material passed under the resin injection unit.

The glass fibres themselves however appear clear and debris free suggesting that these fibres could be impregnated easily. Although the "quality" of the direct-loom waste material was variable, the clean filament winding process is a low-tension manufacturing technique. Hence it was possible to manufacture filament wound tubes. The delicate nature of the direct-loom waste means that it cannot be used with conventional wet-filament winding techniques, where the tension is significantly higher.

The properties of the filament wound tubes manufactured in this study are discussed in Section 4.3.

5.1.2 *Waste Slittings*

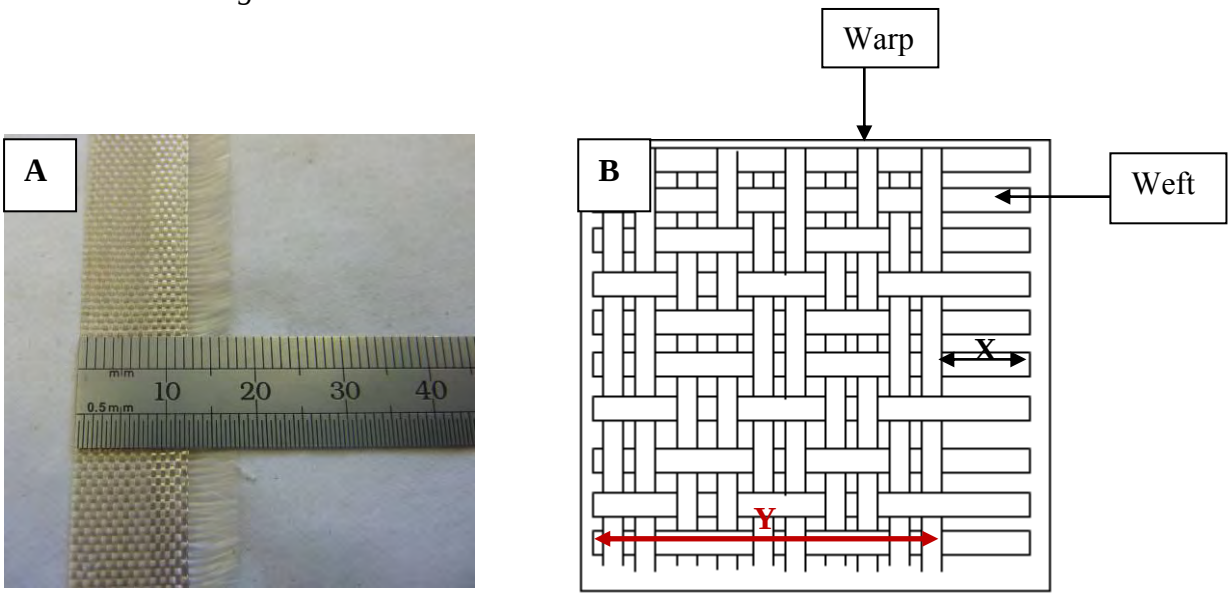


Figure 35 A & B. (A) Shows a photograph of typical sections of the as-received waste slittings. (B) Schematic illustration of the structure with appropriate dimensions, where 'X' is the unsecured section of the weft yarn measuring approximately 5 mm in width and 'Y' denotes the approximate width of the warp fabric at 13 mm.

Magnified images of the waste slittings obtained via scanning electron microscopy are presented in Figure 36 (A & B). Images with a further magnification are presented in Figure 36 (C & D).

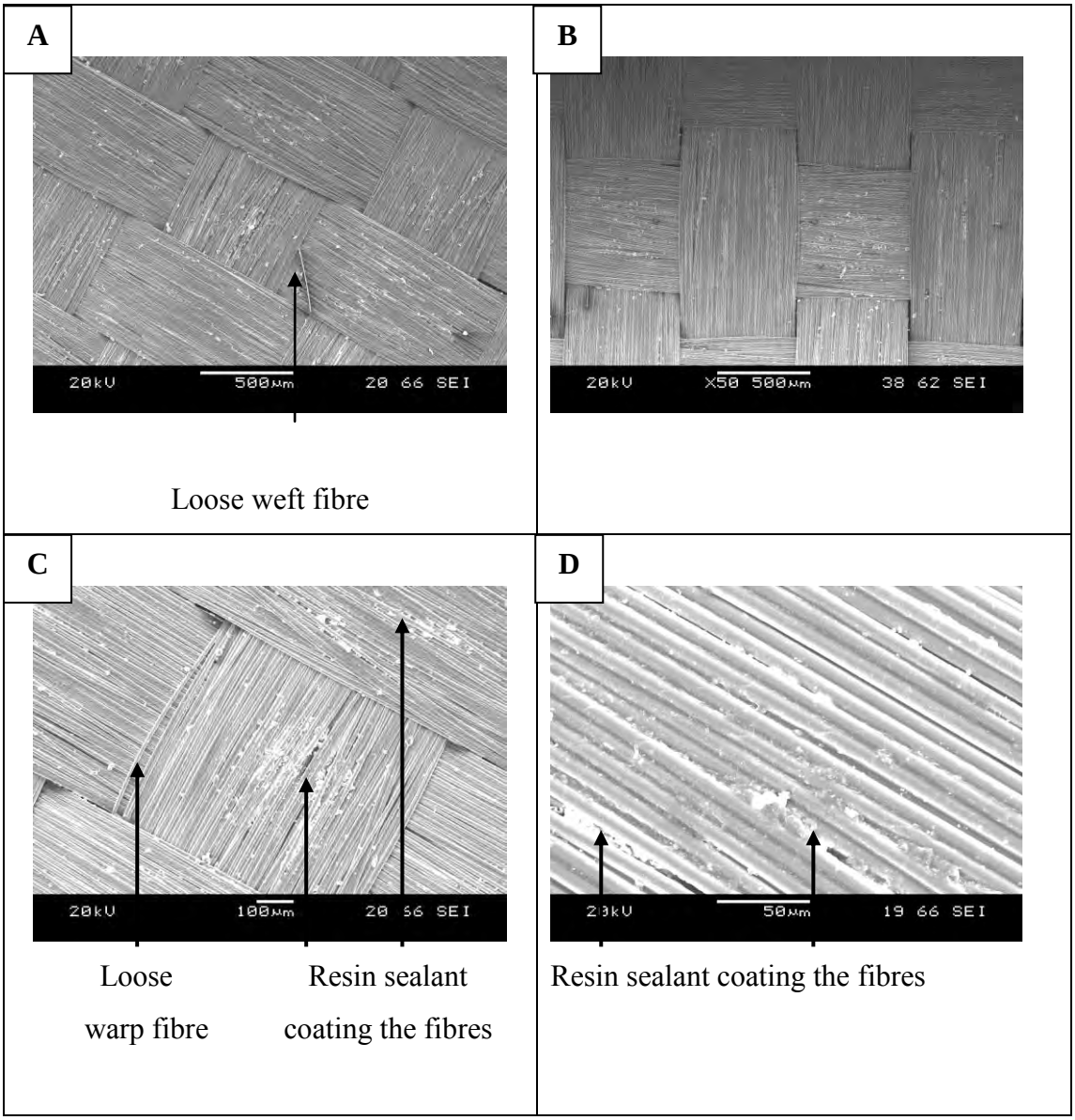


Figure 36(A-D) (A and B) An overall view of the waste slittings fabric showing its tight uniform structure. (C and D). SEM micrographs of the waste slittings with magnification of the fibres within the structure.

From reviewing Figure 36, it is clearly visible that the structure and compactness of the waste slittings made it considerably easier to process than the direct-loom waste. This easier 'process-ability' was due to: (i) its narrower and more consistent width, allowing it to pass freely through the resin injector; (ii) its greater stability, due to the resin sealant being present on the surface (Figure 36 C); and (iii) greater strength of the fabric permitting it to pass under the resin impregnation unit without fraying or

disrupting the spatial orientation of the fibres. The “frilly” edge section of the waste slittings allowed some of this fibre to be impregnated more effectively. Nevertheless, this loose edge meant there was an increased risk of the warp fibres becoming dislodged, which is highlighted in Figure 36C.

5.1.3 *Pre-chopped E-Glass fibres*

The pre-chopped E-glass fibres were used in the short-fibre vibration-based delivery system. The individual, heavily bound entities allowed for extensive tests of various flutes and fibre delivery systems. The durability of the fibres minimised the prospect of fibre breaking or splintering to block various fibre delivery systems that were investigated. The heavy binding on the fibres resulted in a durable short-fibre and therefore enabled quick turnaround of calibration trials. However, although this binding affected the ability to impregnate these fibres, it made the fibres easier to process in comparison to a chopped E-glass fibre.

5.2 *Production of Filament Wound Tubes*

As previously discussed, the processing of the direct-loom waste was found to be more difficult than the waste slittings. This was due to the dimensions and ‘fluffy’ nature of the waste product. As a result, a slower winding speed of 2.5 m/minute was used in order to process the delicate direct-loom waste; this was also the reason why a rough pitch of 7 mm was indicated earlier.

The waste slittings were produced at the faster speed of 5 m/minute due to the easier processing procedures and stability of the material. The 7 mm pitch was adopted to allow for sufficient overlap of the material and to ensure the frilly, un-woven section of the fabric was not left exposed.

The glass fibre “reference” tubes were produced at a winding speed of 10 m/minute. A 4 mm pitch was used due to the narrow dimensions of the fibre. The tubes were then placed onto a rotating mechanism to ensure a smooth surface finish. Photographs of filament wound tubes manufactured using the direct-loom waste, waste slittings and glass fibre are presented in Figures 37 (A to C) respectively.

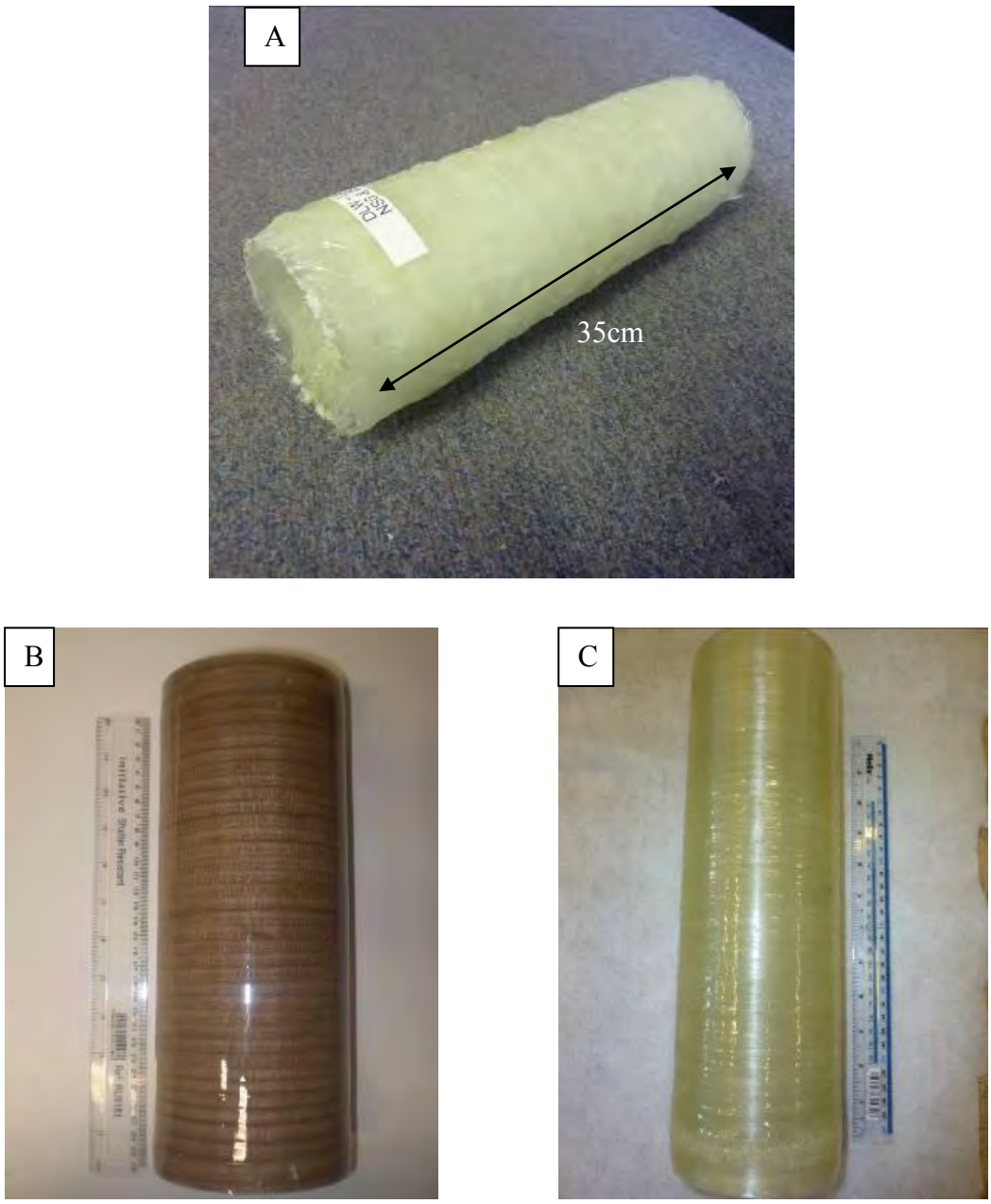


Figure 37: Filament wound composite tubes made from: (A) Direct-loom waste; (B) Waste slittings; and (C) Virgin E-glass fibre.

The clean filament winding process was used to produce glass-fibre and waste fibre tubes as discussed in an earlier section of this report. In addition to these tubes, conventional filament wound tubes were also manufactured; these were fabricated in order to allow for a comparison of the modified CFW process in relation to its conventional predecessor.

5.3 Composite Filament Wound Tube Assessment Methods

5.3.1 *Physical Properties*

Presented in Table 2 are the physical property results for the six tube variations tested in this study.

Table 2: Fibre volume fractions and void contents for the six types of tubes manufactured in this study. Five samples were tested from each of the six tubes to generate the results presented below.

Tube Type	***In-house CFW***	***Conventional***	***On-site CFW (7 m/min)***	***On-site CFW (21 m/min)***	***Waste Slittings (CFW)***	***Direct-loom waste (CFW)***
Void Content	0.496 (±0.04)	1.19 (±0.52)	0.93 (±0.63)	1.06 (±0.59)	3.049 (±0.93)	1.04 (±0.11)
Fibre Volume Fraction	68.10 (±2.57)	48.86 (±2.51)	60.07 (±1.46)	60.59 (+0.94)	40.12 (±2.77)	26.01 (±1.47)

From reviewing Table 2, it is evident that the tubes produced in-house and on-site had a higher fibre volume fraction and lower void content than the conventional filament winding process. These desirable properties were achieved as the impregnation process in the clean filament winding technique is assumed to be more efficient (Pandita et al., 2007).

Nevertheless, the conventional tube's low fibre volume fraction could be attributed to the excess resin used in the process, where a resin film builds up on the tubes surface. In contrast a conventional filament wound tube would normally be expected to have a fibre volume fraction similar to those reported by Wait, 2010 and Shotton-Gale *et al*., 2010. Both authors reported a conventional filament wound tube in their result comparisons with a fibre volume fraction of 63% and 70% respectively.

The higher void content evident in the waste slittings was due to the fact that they were difficult to impregnate, due to the presence of a resin sealant coating on the

surface of the fibres. The resin sealant together with the rigid structure of the fabric may have contributed to the poor wetting and impregnation of the waste slittings.

With regards to the direct-loom waste, the poor process-ability of the direct-loom waste meant a slower winding method was adopted. This resulted in lower tension in the fibre, preventing the fibres from becoming closely packed; consequently a lower fibre volume fraction is evident. This can also be attributed to the non-uniform spatial distribution of the fibres, highlighted in the scanning electron microscopy images earlier in this report (Figure 34)

The results from Table 2 show the benefits of utilising the clean filament winding method to reduce void contents and increase fibre volume fraction. The results also show that it is possible to replicate the process on-site.

5.3.2 Mechanical Properties

5.3.2.1 *Inter-laminar Shear Strength*

Figure 38 presents a summary of the inter-laminar shear strength (ILSS) results for the tubes manufactured by the CFW and conventional processes.

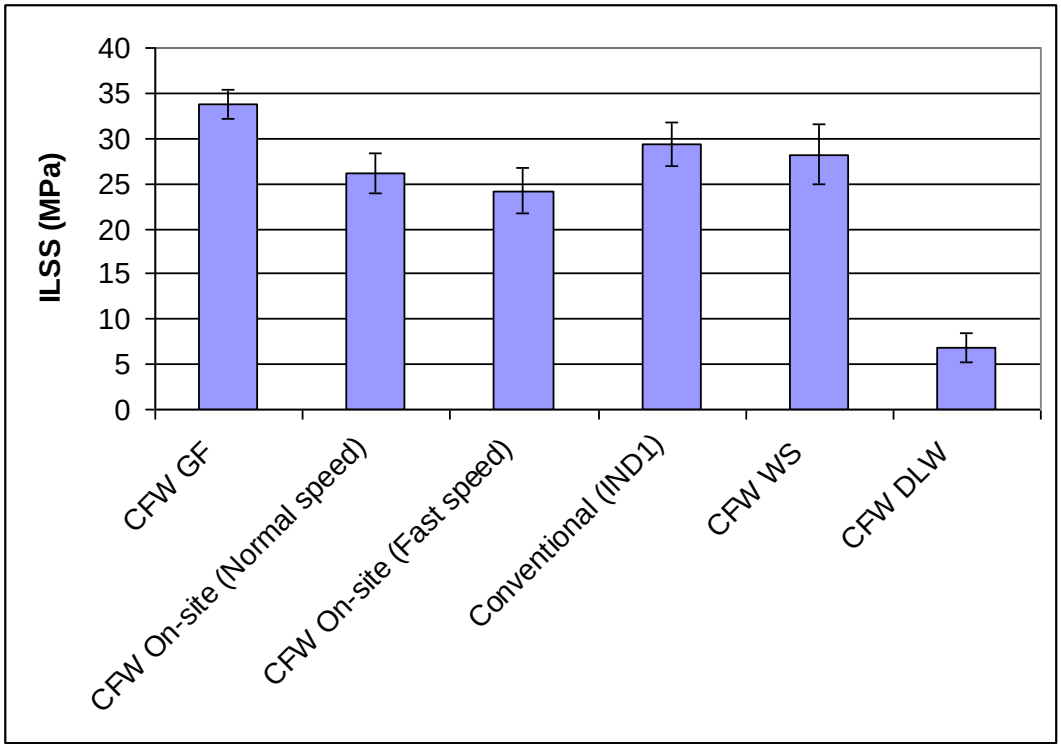


Figure 38. Inter-laminar shear strength test results for six of the tube variations tested in this study.

With reference to Figure 38, it its evident that the in-house CFW tube yielded the highest ILSS for the tubes manufactured and tested in this study. The ILSS of 33.81 MPa may be due to its low void content as there 'is a general agreement that voids have a detrimental effect on the matrix dominated properties, such as inter-laminar shear strength' (Zhu *et al.,* 2009). The tubes produced on-site via the retrofitting of the CFW process were the weaker of the virgin glass fibre tubes, with an increase in processing speed appearing to affect the inter-layer bonding. The conventionally produced tube was able to show an ILSS of 29.29 MPa as the conventional filament winding process ensures that the fibres are completely wetted out and impregnated.

The waste slittings showed a comparative ILSS to the virgin glass fibre tubes. The resin sealant does not seem to have hindered the impregnation of the fabric in this instance.

The poor ILSS in the DLW can be attributed to (i) the poor spatial orientation of the weft fibres; (ii) the inability of the cotton threads to stitch the weft yarn together and; (iii) the variability in the weft yarns leading to resin rich areas within the composite.

The ILSS results clearly show the benefit of clean filament winding however the processing speed and a sufficient volume of resin is clearly a factor in determining the ILSS of the tube.

5.3.2.2 *Hoop Tensile Strength*

Figure 39 presents a summary of the hoop tensile (split-disk) strength results of the clean filament winding method in comparison to conventionally produced filament wound composite tubes.

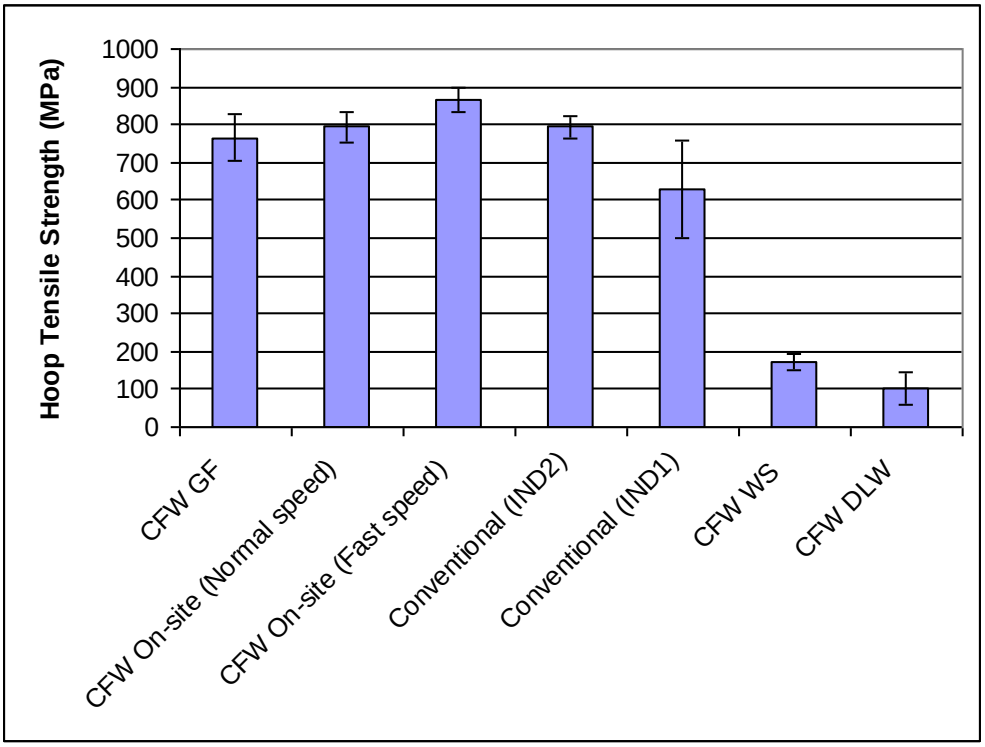


Figure 39. Summary of hoop (split-disk) tensile results for the six tube variations manufactured in this study. An additional data set is presented for the filament wound tubes that were produced on-site (conventional (IND2)). The conventional IND2 tube was manufactured at an undisclosed venue using an industrial 2-axis filament winding machine.

It is clearly apparent from Figure 39 that the hoop tensile strengths for the tubes manufactured in the laboratory (CFW GF), on-site (CFW on-site (normal speed)) and conventional (IND2) are comparable. Filament wound tubes manufactured at the undisclosed site (conventional (IND1)) had the lowest hoop tensile strength when compared to the tubes mentioned above. Currently the reason for this disparity is unknown but it does highlight that the tubes produced using a conventional resin bath can show a relatively large disparity in their properties.

The CFW tube recorded a hoop tensile strength of 764 MPa compared with the 793 MPa for the conventional tube. It is also evident how the increase in speed and therefore tension aids the hoop tensile strength. This is possibly because (i) the higher winding speed (21 m/min) resulted in a higher tension in the E-glass fibres; and (ii) the higher tension would have meant that the relative orientation of the reinforcing fibres would be better.

The hoop tensile (split-ring) data presented in Figure 39 shows that the tubes manufactured using the waste slittings and direct-loom waste were the lowest when compared to other reinforcing fibres used in this study. This appears to be rational for the following reasons.

The structure of the direct-loom waste principally consists of weft E-glass fibres which are held in place with cotton 'stitching' threads. These reinforcing fibres run transverse to the primary loading direction which is illustrated in Figure 40.

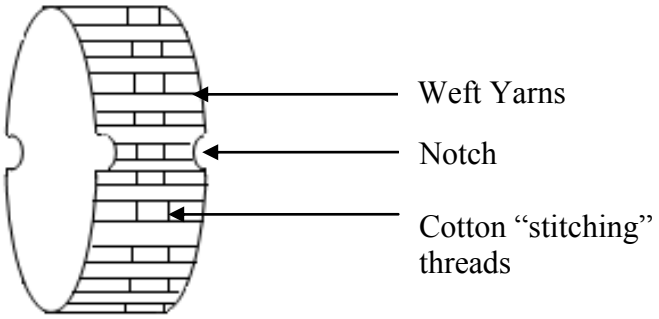


Figure 40. A schematic illustration of a direct-loom waste hoop tensile test specimen. Highlighted within the illustration is the structure and orientation of the material.

In addition to the structure of the hoop tensile samples highlighted in Figure 40 a further complication with these samples is that a variable number of overlapping layers will contribute to the materials strength. In the current study six layers were used. In summary, in the case of the direct-loom waste it is the transverse properties of the composite tube that are being evaluated.

With reference to the hoop tensile strength data for the waste slittings material, it just out-performs the direct-loom waste material but again does not compare to the virgin E-glass fibre tubes tested in this study. The apparent weakness within the composite tubes may be due to the following reasons:

(i) The fabric was coated with a proprietary resin system and its compatibility with the epoxy system used in the study is currently unknown. Therefore the load transfer between the layers in the composite may have been poor.

(ii) The woven fabric meant that the fibre in the hoop direction were undulated and;

(iii) The "frill" caused by the weft fibres created a variable thickness within the filament wound tube.

In summary the presence of a resin "coating" may have adversely affected the load transfer between the layers of the reinforcement within the filament wound tube.

5.3.2.3 *Lateral Compression Strength*

Presented in Figure 41 are the lateral compression strengths comparing tubes produced using the clean filament winding method and those produced using conventional methods.

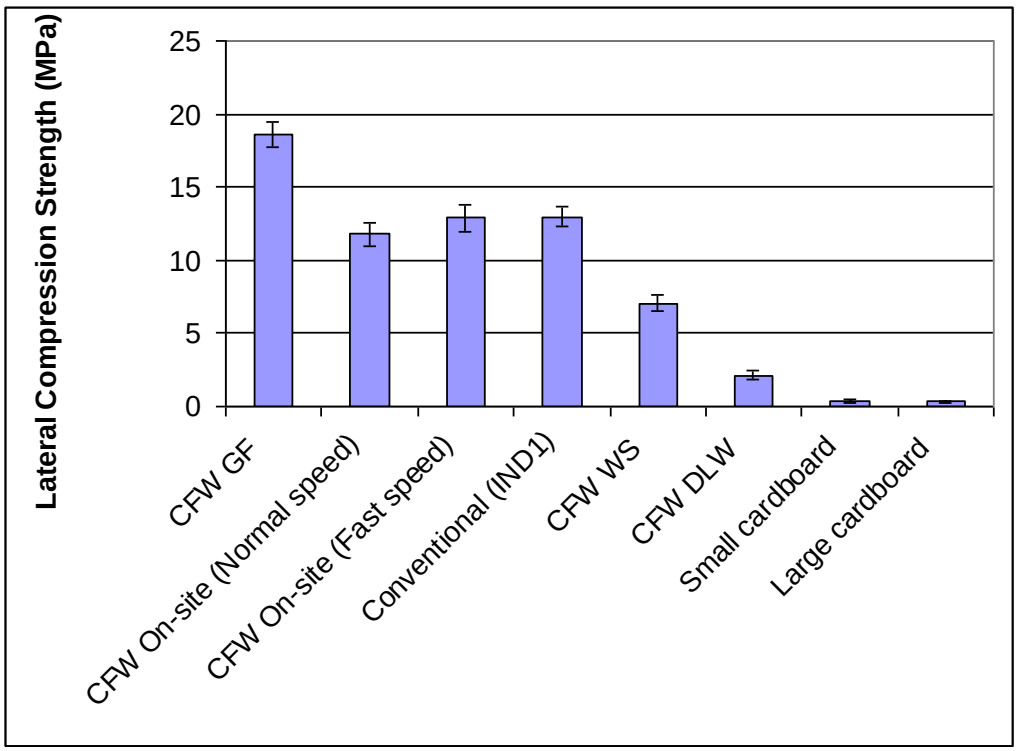


Figure 41. Graph comparing the lateral compression strengths of tubes produced from conventional and clean filament winding techniques. Also included within the comparisons are two variations of cardboard inner tubes currently used by an industrial partner. These are included to create a direct comparison and evaluate the potential for replacement of these tubes.

The results presented in Figure 41 give further evidence for the benefit of the clean filament winding method. As well as its clean credentials discussed earlier, the apparent strength of the tubes in comparison to those produced conventionally again offers credibility. The clean filament wound tube produced in-house showed the highest lateral compression strength of 18.49 MPa with the other three virgin glass fibre tube variations not being significantly different from each other at 11.76 MPa, 12.87 MPa and 12.97 MPa (left to right).

The waste slittings tube was the stronger of the waste fibre two tubes at 7.07 MPa, approximately 38% of the strength of the CFW E-glass fibre tube. The results of this lateral compression test appear similar to that of the ILSS.

The direct-loom waste was not impregnated as effectively due the large nature of the fibre passing through the injection unit. This resulted in dry spots within the composite and created weakness points due to poor bonding. However, the waste slittings were able to pass through the injection unit more freely and were uniformly coated with resin. This resulted in significantly fewer dry spots in the filament wound tube as well as a sufficient amount of resin to bond the interfacial layers together. The glass fibre tube is much stronger as the glass-fibre filaments were sized to bond well with the resin and had no resin sealant coating on its surface. This enabled effective impregnation of the glass fibre. The weakness in the cardboard tubes was a result of weak glue bonding the layers of the cardboard which has a significantly lower strength than resin.

The crucial result for the mechanical testing of the filament wound tubes would come from the lateral compression test. This is due to the application for which the tubes would be used i.e. to replace cardboard inner tubes currently used by the industrial partner to wind on woven fabrics. In this type of application the tube would be under a considerable amount of lateral compression due to the load of the material wound around it. Both types of waste-fibre tubes outperform the cardboard tubes and clearly show the potential to replace them in this application.

5.4. *Image Analysis*

This section presents a series of micrographs to indicate the quality of the filament wound tubes manufactured using specified reinforcements and the CFW or conventional techniques.

5.4.1 Conventional Filament Wound Tubes

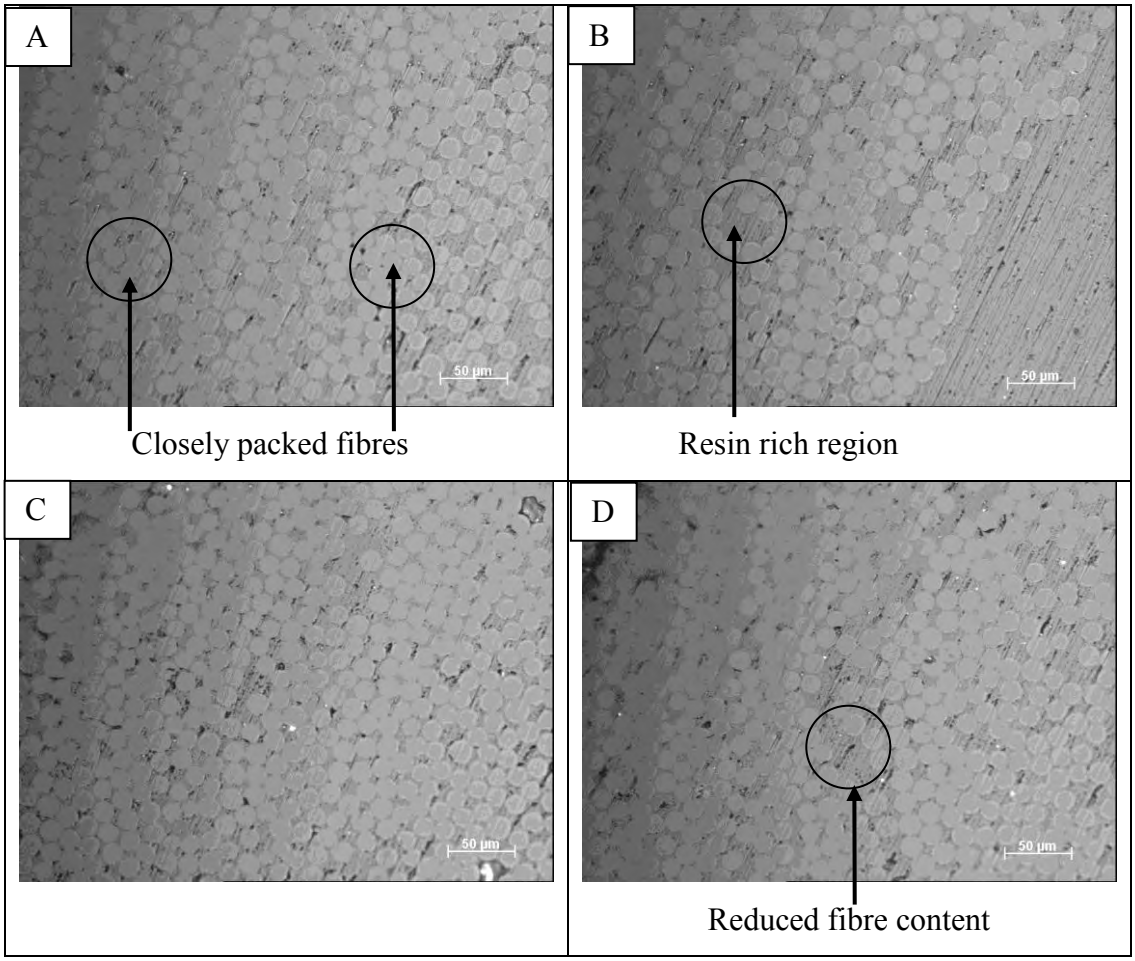


Figure 42 (A-D). Image analysis micrographs of conventional filament wound tube sections. The images present areas within the conventional filament wound tube sample that have high fibre content. There are areas within the same sample however, that have a lower fibre content as indicated.

The conventional tube was taken as the baseline sample as the CFW technique is still a relatively new process. Figure 42 presents images of the conventionally produced filament wound tube. Demonstrated in Figure 42A are images where the fibres appear to be closely packed but also highlighted resin rich regions. This explains the lower fibre volume fraction when compared to the other tubes in Table 2.

5.4.2 Laboratory Produced Clean Filament Wound Glass-fibre Tube

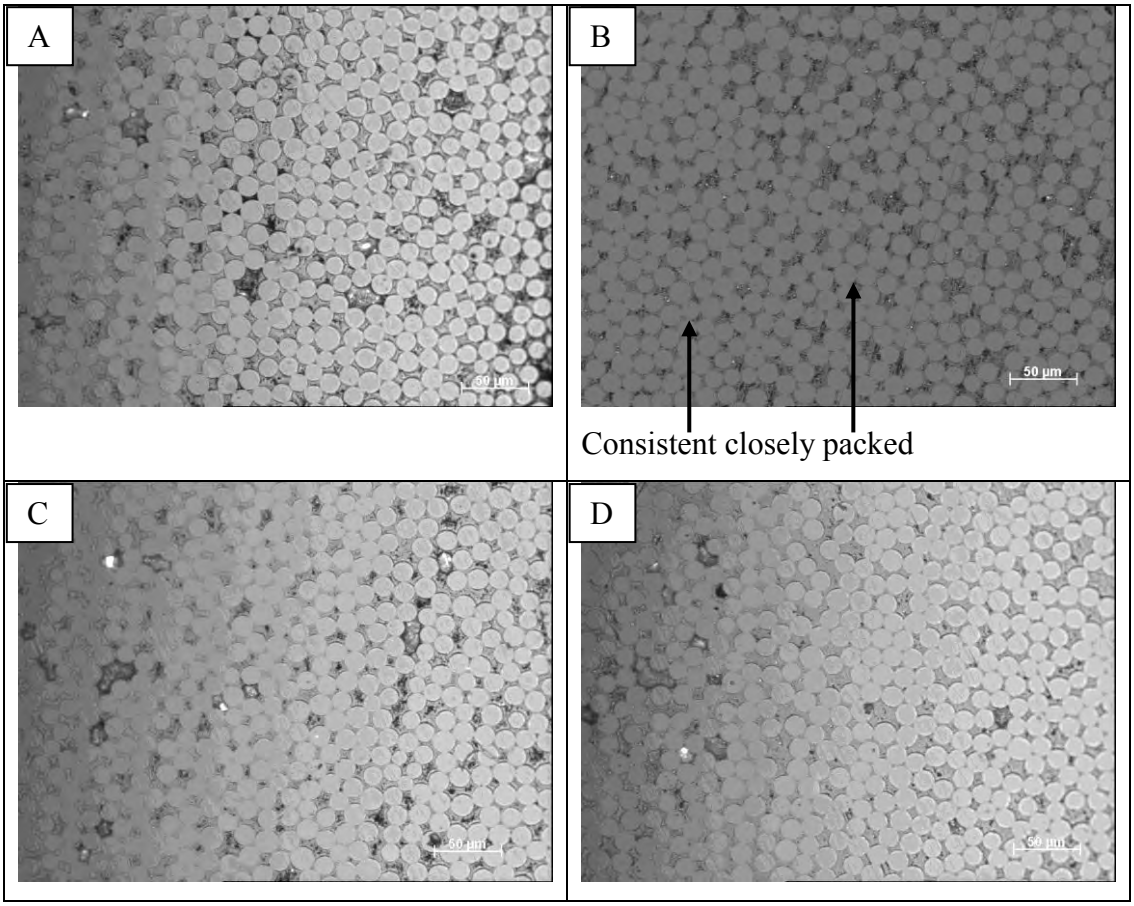


Figure 43(A-D). Image analysis micrographs of laboratory clean filament wound tube sections. The micrographs depict closely packed high fibre content samples with minimal areas of resin concentration.

In contrast to the conventional tube, the CFW images are completely filled with closely packed fibres which correlates with the higher fibre volume fraction results presented earlier in Table 2.

5.4.3 Clean-filament Winding On-site

5.4.3.1 Normal Speed

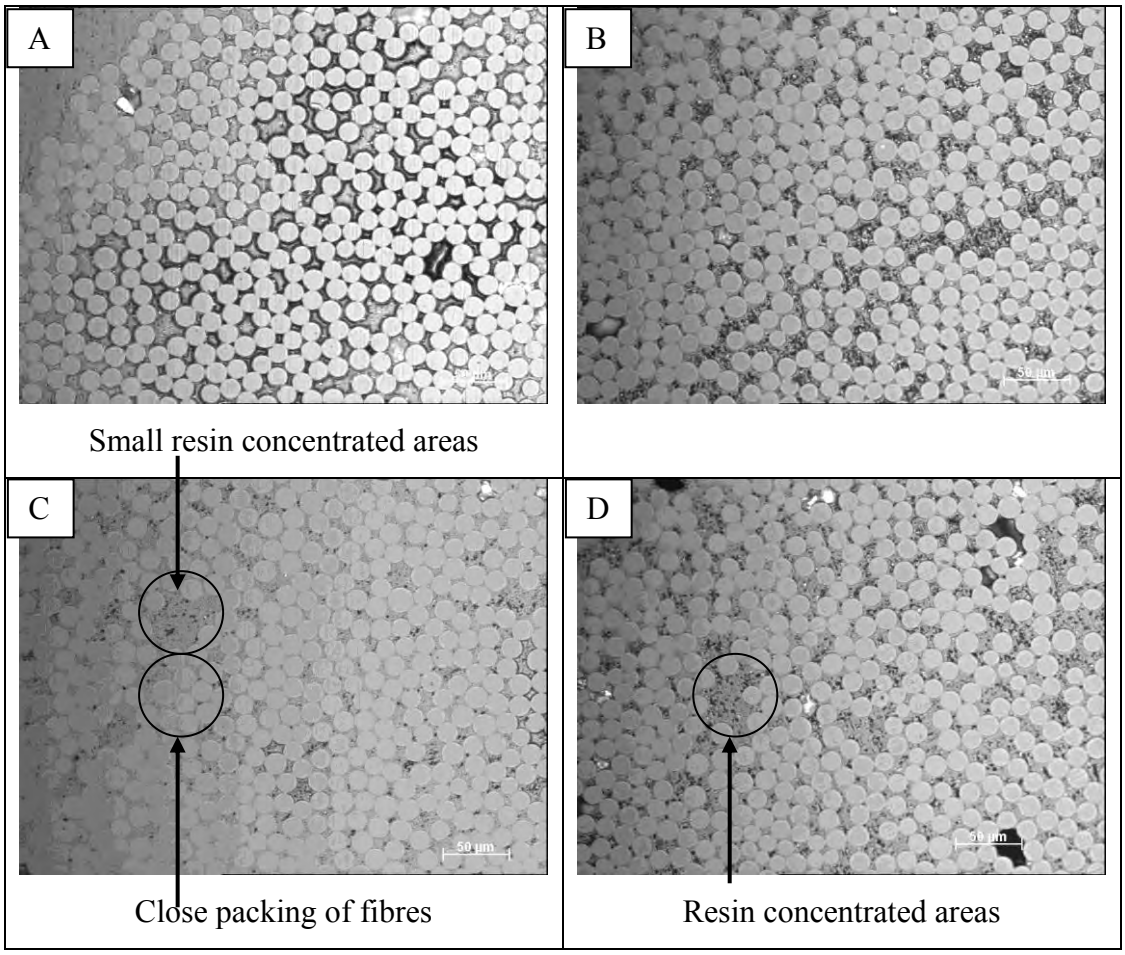


Figure 44 (A-D). Image analysis micrographs of on-site clean-filament wound tube sections: 7 m/min winding speed. Areas of high fibre content and concentrated areas of resin are indicated.

The images presented for the tubes wound on-site at the normal speed again show these areas of resin concentration and packing of fibres (Figure 44C and D). From these images it appears that the clean element of the filament winding helps to retain a high fibre volume fraction in the on-site tubes. This can be validated in Table 2 with the conventional tube producing the lowest fibre volume fraction value.

5.4.3.2 Faster Speed

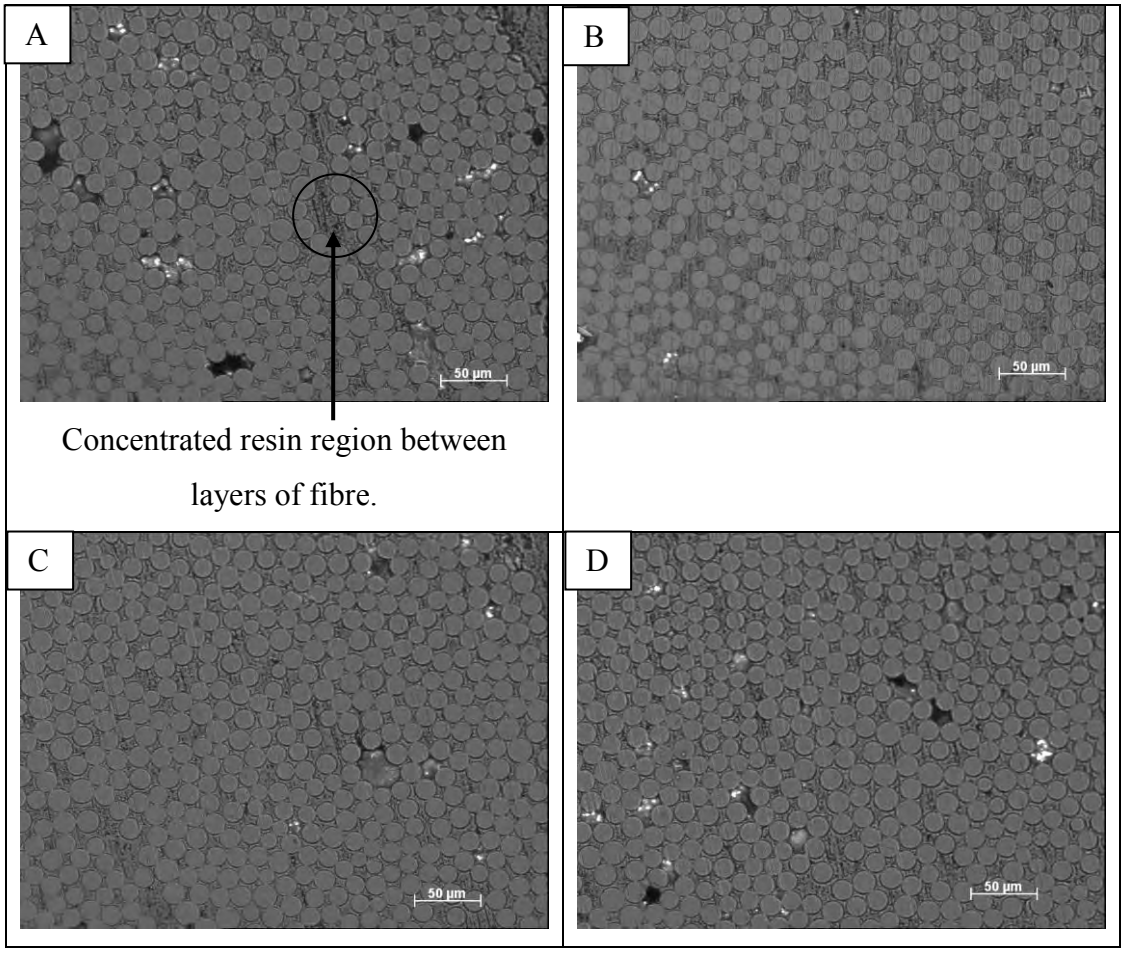


Figure 45 (A-D). Image analysis micrographs of on-site clean-filament wound tube sections: 21 m/min. Concentrated resin areas between layers of the fibres can be found, however the images generally show a high fibre content.

Small pockets of resin appear again in the faster on site winding method (Figure 45A) and account for the lower fibre volume fraction presented earlier within this report. The higher speed may have increased the tension within the fibre and may be the cause of the lower fibre volume fraction. The higher speed however did appear to have a positive effect on the hoop tensile results presented earlier so this lower fibre volume fraction is not necessarily an issue.

5.4.4 Waste Slittings

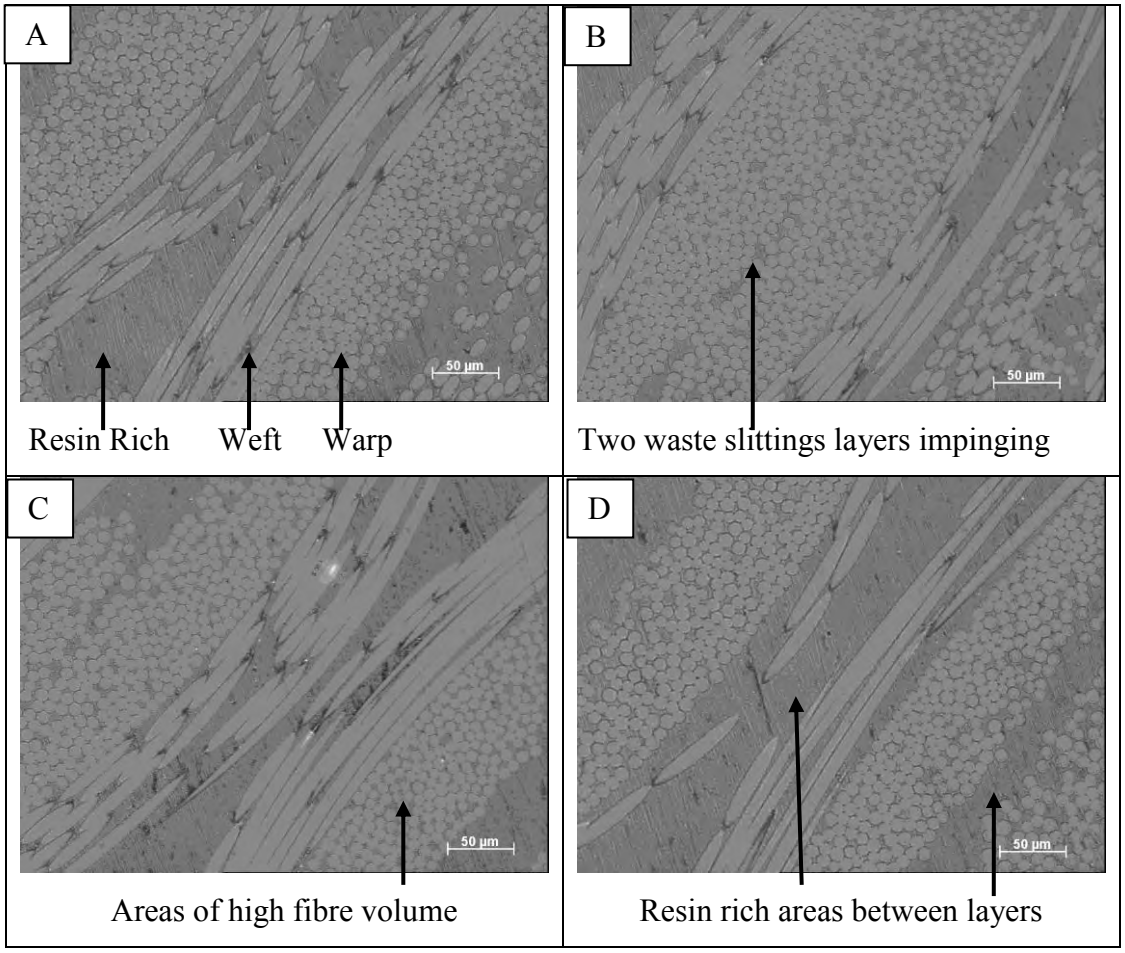


Figure 46 (A-D). Image analysis micrographs of waste slittings' tube sections. The weaving of the fabric is indicated with the direction of the warp and weft fibres. Areas of significance within the micrographs are also indicated.

Presented within Figure 46(A-D) are figures of the waste slittings where in Figure 46A the warp and weft fibres are clearly indicated. Generally large resin rich areas sit between the layers of the fibres (Figure 46D), which could be due to the difficulty in impregnating the fibre due to the presence of a proprietary resin coating. Figure 46B indicates that this impregnation may be possible in areas with the two layers appearing to impinge. The large resin regions again account for the low fibre volume fraction.

5.4.5 Direct-loom waste

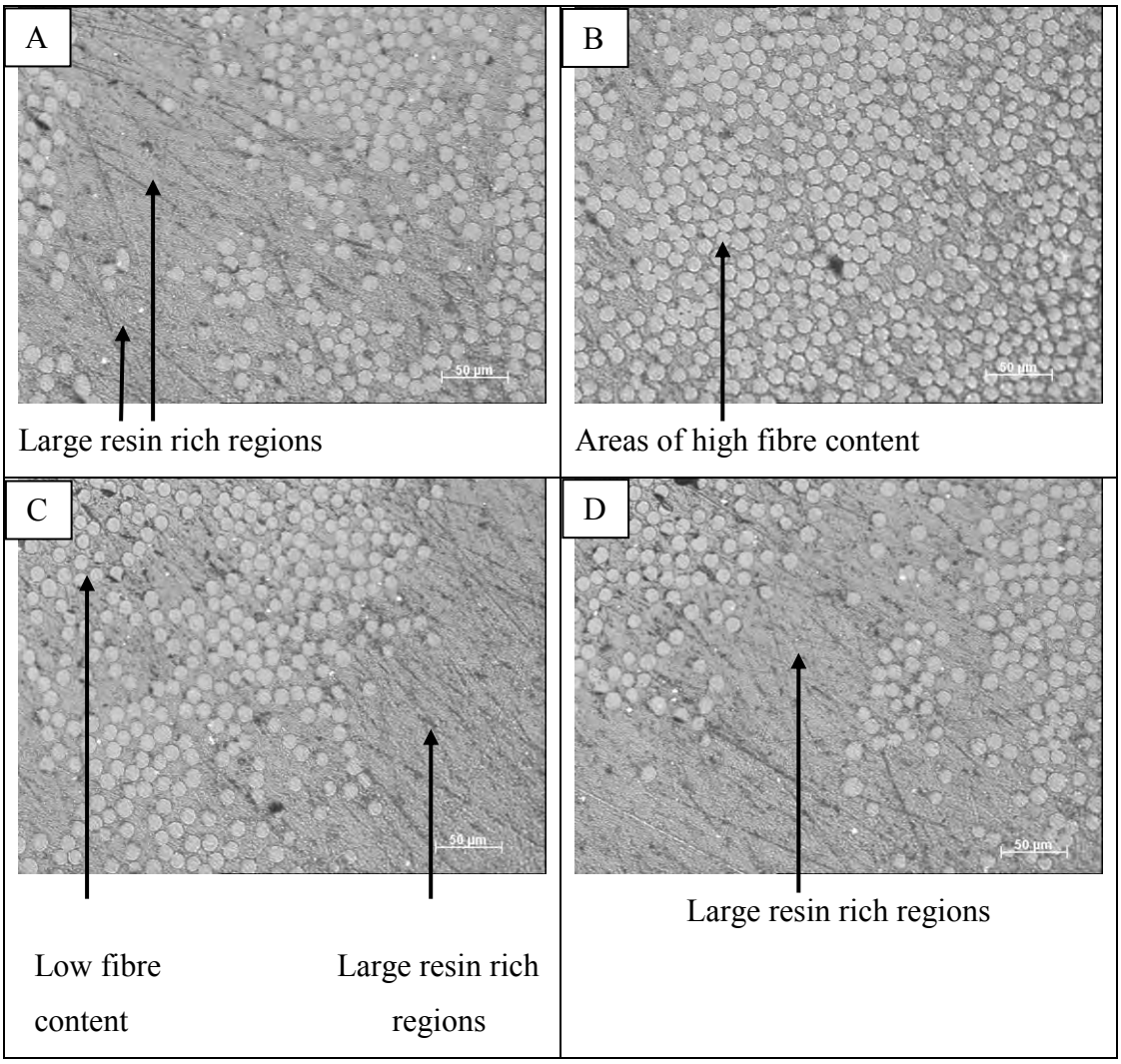


Figure 47 (A-D). Image analysis micrographs of clean filament wound direct-loom waste tube sections. Areas of high and low fibre content are indicated with the large resin content apparent in the majority of the samples.

From viewing Figure 47, immediately apparent is the low fibre volume fraction when compared to the other tube variations. Highlighted within the images are areas of fibre concentration which are the fibre strands within the DLW structure. These were heavily impregnated to try and manage the "frilly/fluffy" nature of the fibre and resulted in the low fibre volume fraction clearly apparent. The images analysis

supports the results presented in Table 2 and provided further evidence for the weakness of the DLW and WS.

5.5 *Scanning Electron Microscopy*

5.5.1 Fracture Surfaces

Fracture surface of the waste slittings and the direct-loom waste are presented in Figure 49 through to Figure 54. The images are taken from the fracture surfaces of the hoop tensile test samples; the area from where this is taken is highlighted in Figure 48.

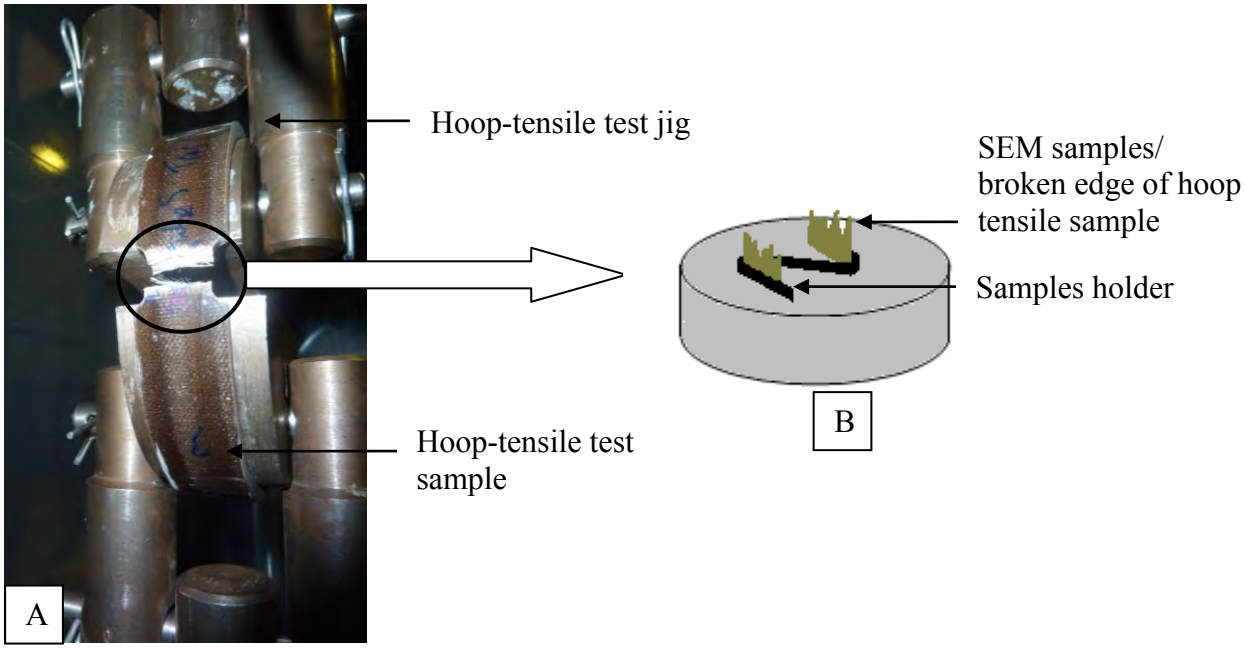


Figure 48. (A) A photograph of a failed hoop tensile specimen following the spilt disk test. (B) A schematic illustration of a steel SEM stub containing the broken edges of the failed hoop tensile test sample.

5.5.2 Waste slittings

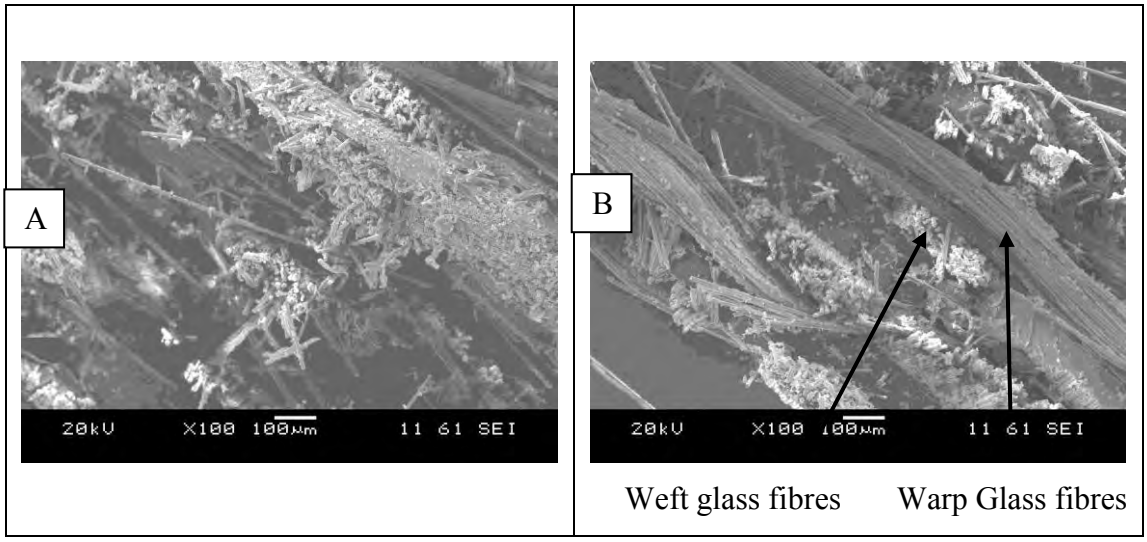


Figure 49 (A and B). SEM micrographs of waste slitting fracture surfaces at x100 magnification.

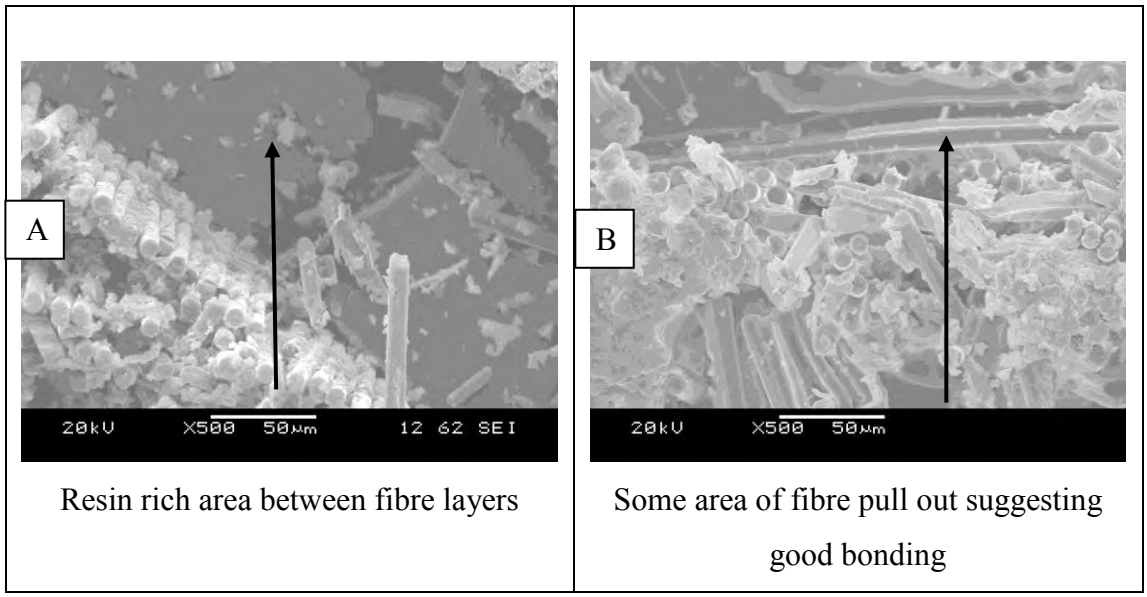


Figure 50 (A and B). SEM micrographs of waste slitting fracture surfaces at x500 magnification.

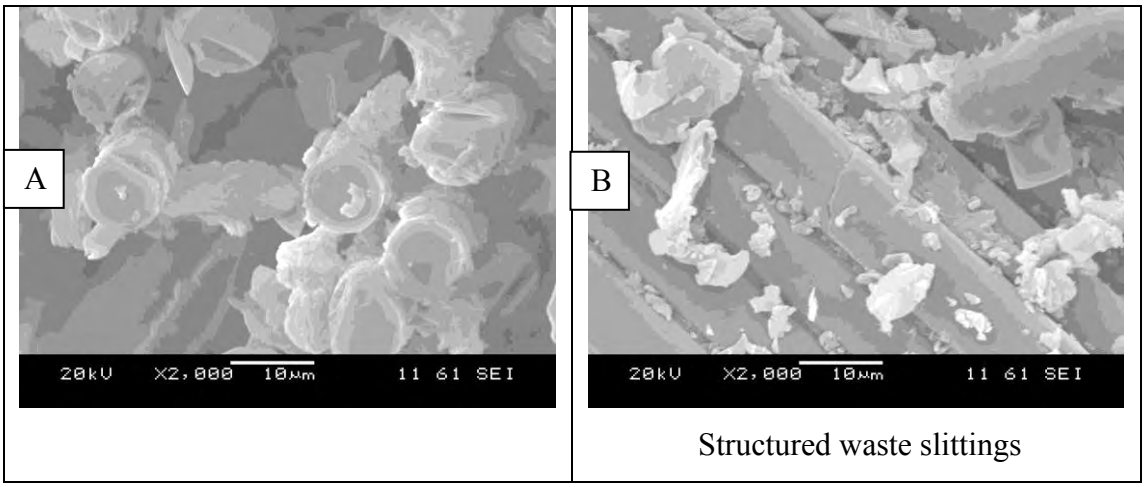
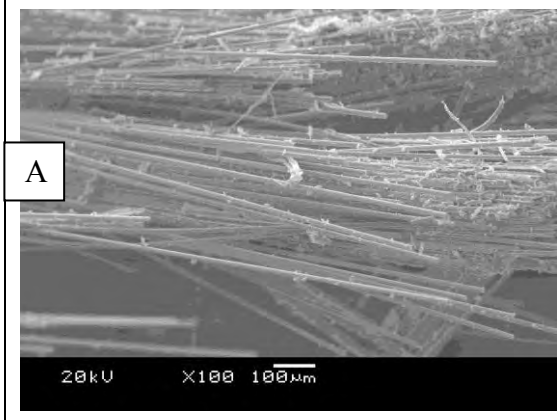


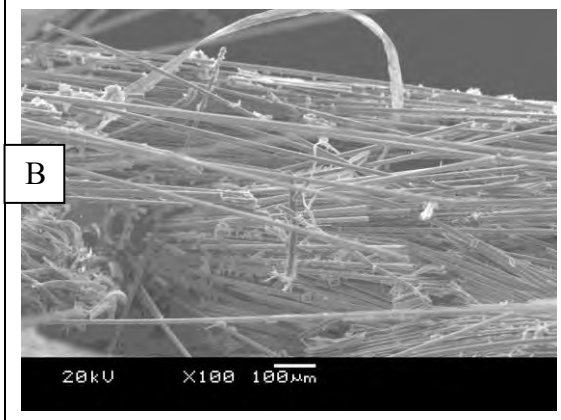
Figure 51 (A and B). SEM micrographs of waste slitting fracture surfaces at x2000 magnification

The results of the SEM tie in well with those of the image analysis. The weave of the warp and weft fibres within the waste slittings structure are clearly visible in Figure 49B. Figure 50B indicates an area of fibre pull out from the matrix to suggest that effective bonding was achieved which is also highlighted within the ILSS results. Again evident in Figure 51 is the aligned, structured fibres within the waste slittings. Dry areas within the material were also apparent on initial visual analysis of these fracture surfaces contributing to the low hoop tensile strengths presented earlier.

5.5.3 Direct-loom waste

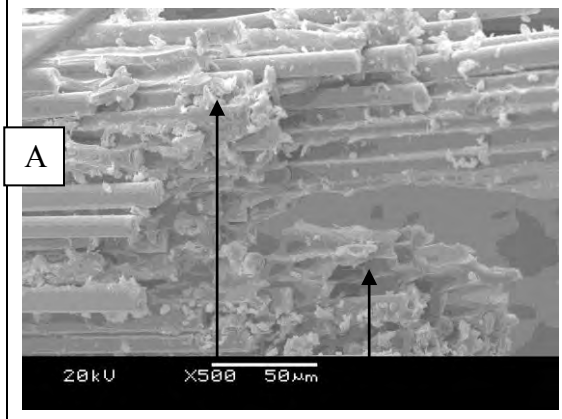


Some order with the majority of the fibre running in parallel



Loosely held fibre within composite

Figure 52 (A and B). SEM micrographs of direct-loom waste fracture surfaces at x100 magnification.



Large concentration of resin around fibre bundle

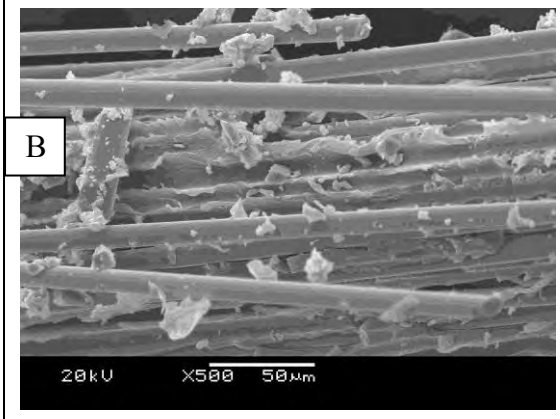
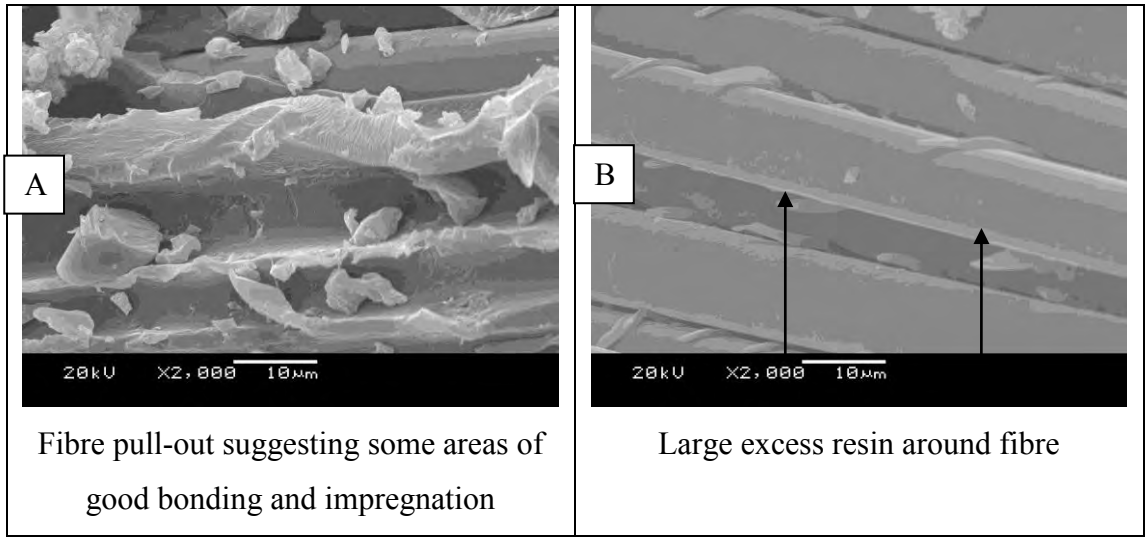


Figure 53 (A and B). SEM micrographs of direct-loom waste fracture surfaces at x500 magnification.



Fibre pull-out suggesting some areas of good bonding and impregnation	Large excess resin around fibre

Figure 54 (A and B). SEM micrographs of direct-loom waste fracture surfaces at x 2000 magnification.

The image analysis results of the direct-loom waste also support the results of the fracture surfaces shown in Figure 52, Figure 53 and Figure 54. Figure 52A and B again highlight the loose bonding around the glass fibres as there is some order, but the fibres are splayed unlike the waste slittings. This again highlights why the direct-loom waste was so difficult to process.

Large amounts of excess resin are evident in Figure 53 and Figure 54; this was used due to the "frilly/fluffy" nature of the material. As the direct-loom waste was considerably more delicate and loosely structured, significantly more resin was used to try and control/impregnate the large mass of fibres. The fibre pull-out found in Figure 54 suggests that this was successful in some areas however the large excess resin and loss of fibre in areas resulted in the lower mechanical strengths found in the split-disk, ILSS and compression tests.

5.6 Short-fibre vibration-based delivery system

5.6.1 *Evaluation of Composites*

5.6.1.1 Degree of Alignment

5.6.1.1.1 Prototype-I

Initial trials with Prototype-I were conducted using adhesive tape to assess the initial orientation through this set-up. Figure 55 shows the six strips of 4.5 mm short E-glass fibre created using Prototype-I.

Figure 55. Photograph of the initial attempts to align short fibres using Prototype-I.

With reference to Figure 55, it is evident that the orientation and control over fibre deposition improved from trial one to trial six, predominantly due to the lower amount of fibre present in the strips. The orientation relied on the operator manually depositing the fibre on to the aligning flute and could not be controlled accurately. The movement of the system was also controlled manually using the Hounsfield tensometer and introduced some human error into the system. The flute angle was also significant in achieving fibre alignment because if the drop distance from the flute to the aligning plate was too large, the fibre would fall vertically and bounce off the hard surface resulting in misalignment. This prototype was clearly shown to be dependent on the operator's competence.

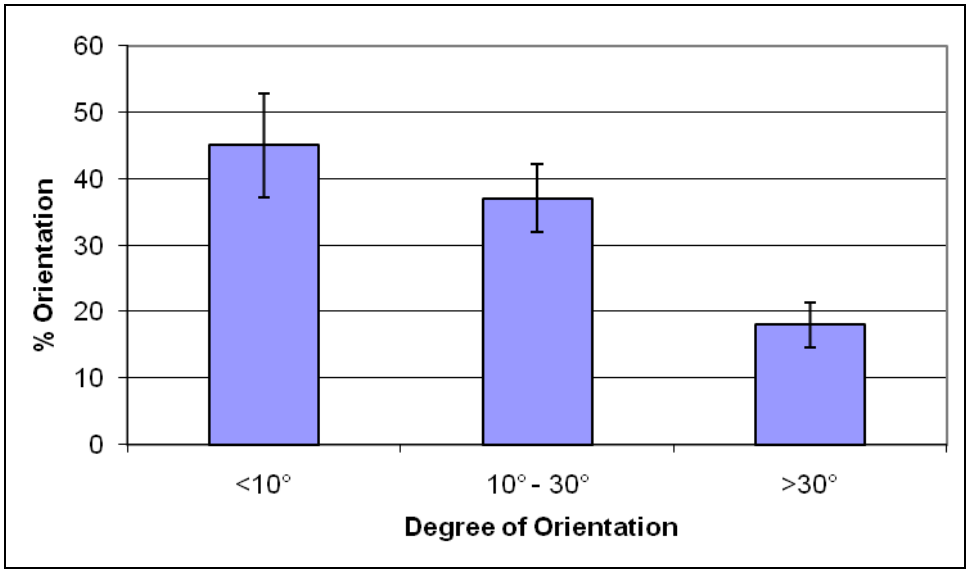


Figure 56. Orientation results from the initial trials strips using Prototype-I.

Following the production of the short-fibre strips the task was then to complete a mat of fibres. This was to represent the production of short-fibre pre-pregs. The mat of fibres produced a similar result to the strips.

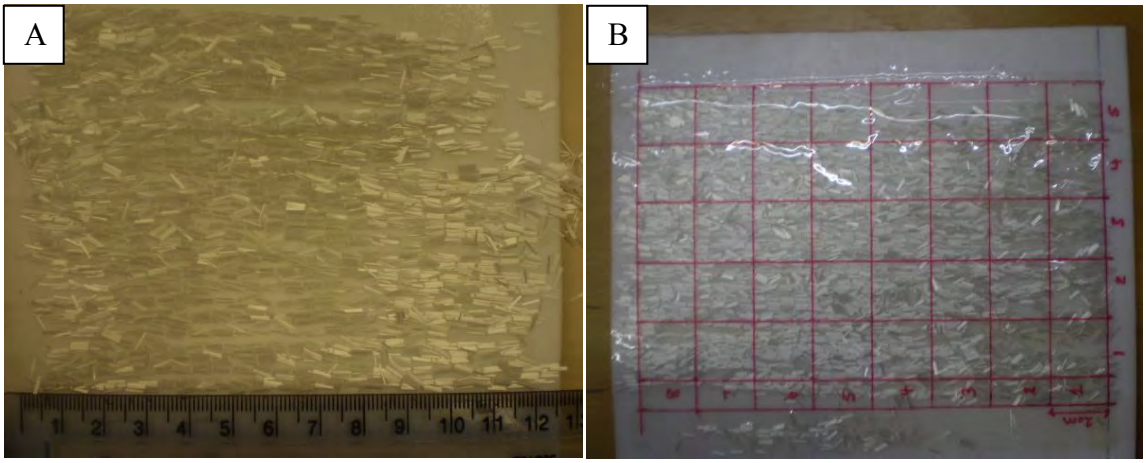


Figure 57: (A) The fibre mat after initial processing. (B) The fibre mat with a protective covering a segregated into squares for orientation analysis.

It is evident from Figure 57 that the mat of fibres is more consistent in its orientation than the original strips of orientated fibre depicted in Figure 55. The majority of fibres are seen to be aligned in the horizontal direction although there are areas of misaligned fibre. This occurs as the new row of fibres is laid and the new fibres bounce off the previous line causing misalignment. This was also found by Harper et

al., 2009, who stated three reasons for fibre misalignment, the second of which 'occurs as tows contact the preform screen or previously deposited tows'. The degree of alignment calculated from this mat of fibre can be found in Figure 58.

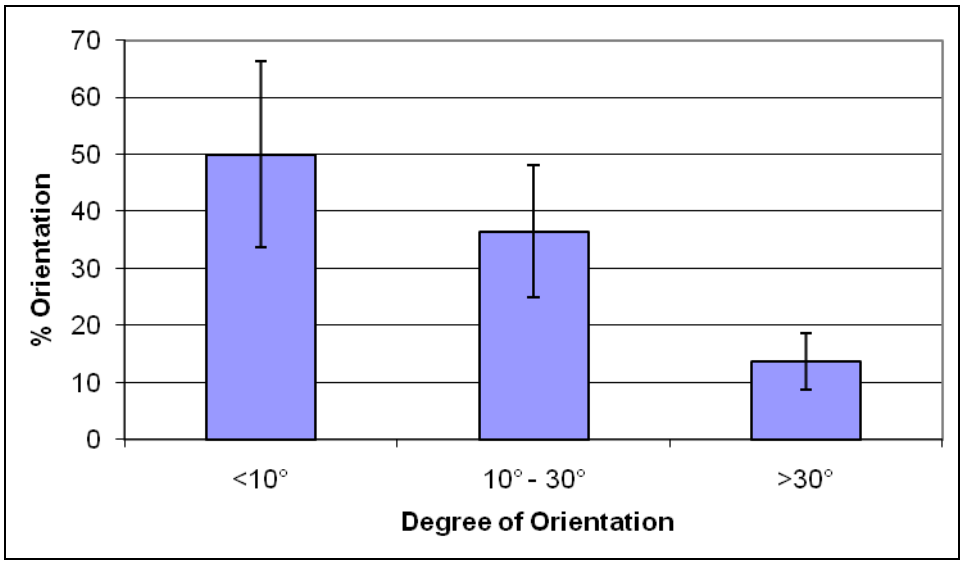


Figure 58. Graph showing the percentage orientation of the short-fibres within the fibre mat produced using Prototype-I.

Although the fibres between 0 to 30° appear to be orientated, the fibres must lie between 0 to10° as used by Harper et al., 2009, in order to be accepted as aligned. Therefore the mat of fibres was able to show a 50% degree of alignment similar to that shown by electrostatic methods (Kim et al., 2009).

5.6.1.1.2 Prototype-II

Due to the aforementioned problems associated with Prototype I, efforts were made to make the process more reliable and consistent. This included the use of a linear translation stage, a solenoid and new speaker housing. Following initial calibration, the new prototype was then employed to align strips of short E-glass fibres on the Hexply® 913 resin film. Results of the initial trials are shown in Figure 60.

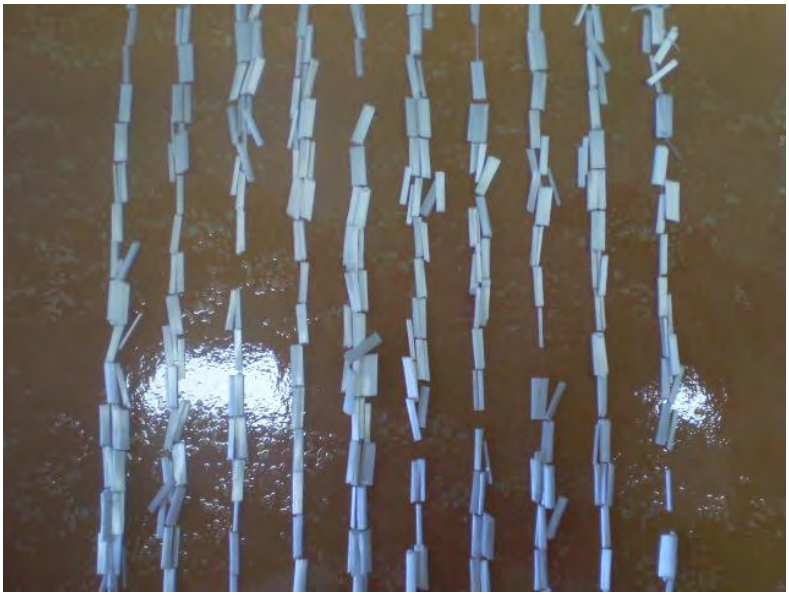


Figure 59. Photograph of the initial trials of Prototype-II on to the Hexply® resin film.

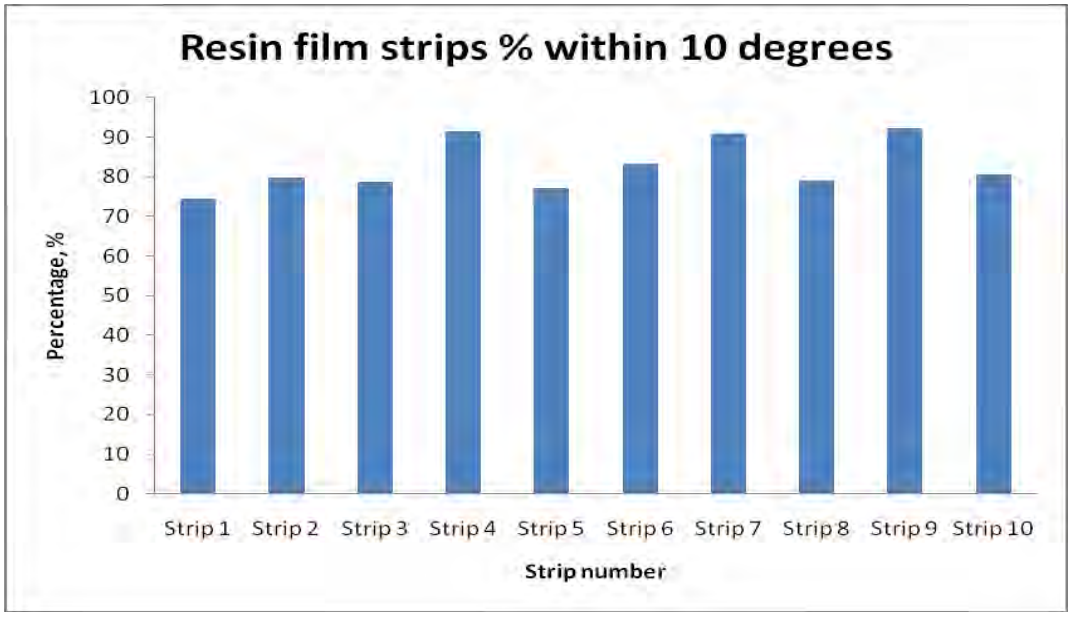


Figure 60. Graph showing the percentage orientation of the strips produced using Prototype-II.

Prototype-II was able to show a higher percentage of orientation than the previous prototype, as indicated by Figure 60. The percentage of orientation varies between 70% and 90%, within the given ±10°, with an average 83% alignment achieved. Prototype-II allowed a more consistent fibre flow from the primary aligning flute to the custom made secondary aligning flute. A more consistent flow allows for the

fibres to be orientated and channelled down the flute and deposited in time with the vibration of the speaker.

Following the success of the single fibre strips, a complete mat of fibres was processed as a short-fibre composite. Using Prototype-II, 4.5 mm E-glass fibres were deposited on to a resin film strip and then covered with a top layer of resin film. An additional layer of resin was added to each side of the composite to ensure a sufficient amount of resin was present and that the composite could be effectively processed. The final composite preform can be found in Figure 61 A. A close up view of this preform can also be found in Figure 61B, showing that a high degree of orientation can be achieved. An image of the final composite is shown in Figure 62 after the initial preform was processed via conventional autoclaving procedures.

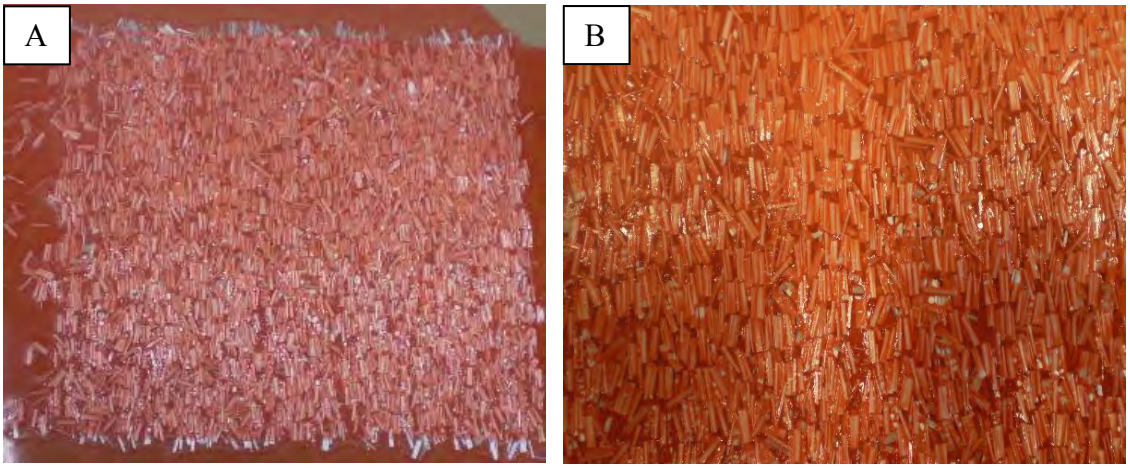


Figure 61. (A) A short-fibre composite preform ready to be processed using standard autoclaving procedures to produce a final composite. (B) A close-up view of the preform.

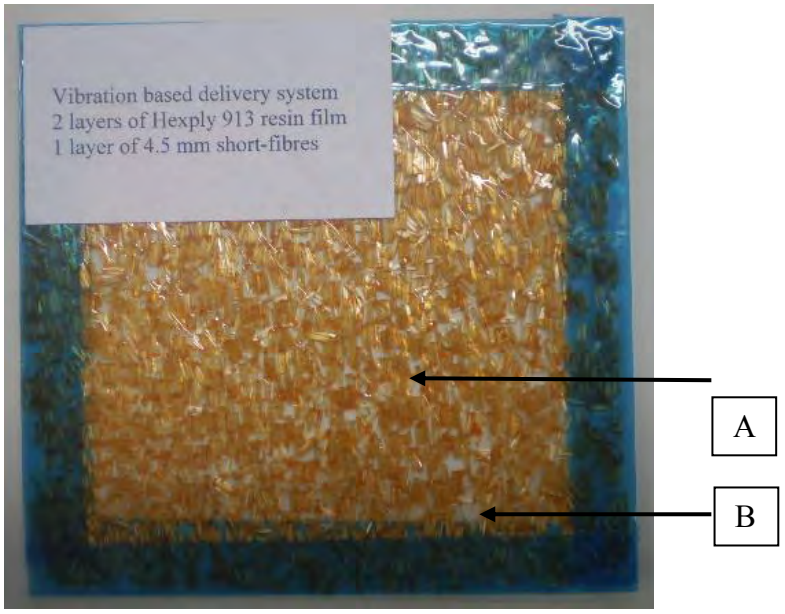


Figure 62. The final cured composite containing two layers of Hexply® 913 resin each side of one layer of 4.5 mm E-glass fibres. Highlighted within the image are resin rich areas (A) and dry fibre (B) within the composite.

Figure 62 shows a series of resin-rich areas within the final composite produced. The short-fibres within the structure are heavily bound and with the vast amount of fibres within this deposition it was difficult to achieve uniform resin coverage. The dry spots indicated in Figure 62 would result in a vastly reduce strength within the composite as areas are not effectively supporting the construction. To combat this issue a more substantial layer of resin was built up prior to the fibre being deposited on it. This is demonstrated in the following section on Prototype-III.

5.6.1.1.3 Prototype-III

Although a highly aligned composite was produced using Prototype- II, it was evident that the size of the composites produced were not large enough to provide a conclusive outcome. Therefore, Prototype-III was developed using the same equipment but with a modified set-up to enable production of a more substantial 30 cm by 30 cm preform. Eight sequential preforms were produced using this Prototype-III, to produce a final eight layer uni-directional short-fibre composite plate. Images of the preforms after the initial fibre alignment can be found in Figure 63.

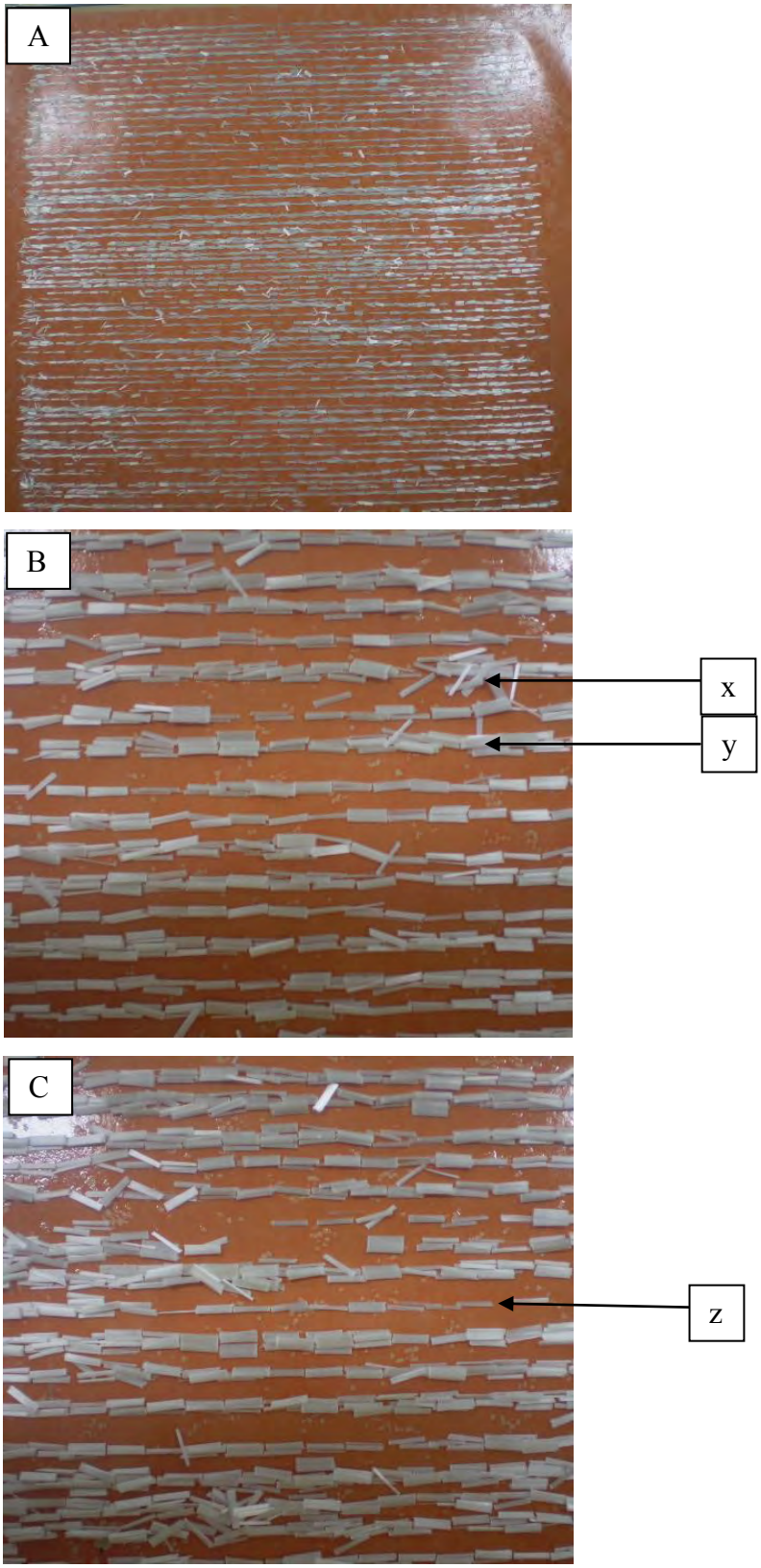


Figure 63 (A, B and C). (A) Photograph of a short-fibre preform immediately after aligned fibre deposition. (B and C) Close-up images of short-fibre preforms highlighting the high degree of orientation.

Highlighted within Figure 63 area areas of misalignment (x), a straight run of large fibres (y) and a well orientated run of thin fibre (z). The misalignment clearly indicated with 'x' has been previously discussed by Harper *et al*., 2009. Harper stated that one of three reasons why tows misalign is that "tows slide as they make contact with previously deposited tows" which appears to be the instance in this scenario. Label 'y' is able to show an area where a consistent run of aligned fibre has been created with the system however areas such as 'z' indicates areas where the fibre is less consistent. This could be caused by the solenoid delivery system which provides a consistent rocking motion to deliver the fibres. This may require fine tuning or more constant delivery in conjunction with the translation of the speaker system. A section such a 'z' required a further run to complete this area which resulted in time delays and would have to be rectified in a more industrial system.

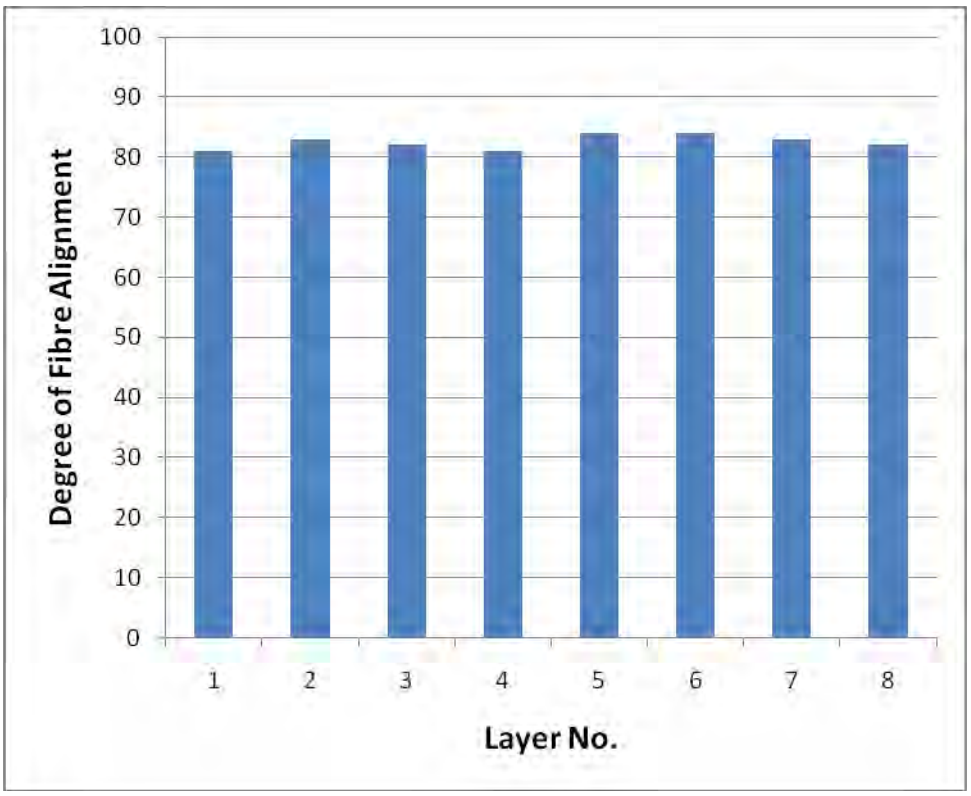


Figure 64. Degree of alignment across eight layers of a short-fibre composite produced from Prototype-III

Digital photographs were taken of the preforms, following the initial fibre deposition to allow for orientation analysis. The results of this orientation analysis can be found in Figure 64.

Following the orientation analysis layers of Hexply® 913 resin film were applied to form the finished preforms. An example of a finished preform can be found in Figure 65.

Figure 65. Photograph of a preform after the additional layers of resin and 'debulking' had been performed in the autoclave.

The combination of the eight individual preforms to form an eight ply uni-directional short E-glass fibre composite can be found in Figure 66.

Figure 66. The final processed composite composed of the eight layers of composite preforms, processed using conventional autoclaving procedures.

The eight ply short-fibre plate was then cut to the aforementioned specific sample sizes to be mechanically and physically tested. The short-fibre plate was tested against a short-fibre plate produced in-house by other authors, a uni-directional E-glass fibre plate produced in-house and a uni-directional E-Glass fibre plate produced from a conventional pre-preg. A photograph of the UD E-Glass plate produced in-house can be found in Figure 67.

Figure 67. The final uni-directional E-Glass fibre composite plate produced in-house.

5.6.1.2 *Physical Properties*

5.6.1.2.1 Fibre Volume Fraction

Following the aforementioned resin burn-off technique, the fibre volume fraction of the short-fibre composite plate was calculated. The results of this test are presented in Figure 68.

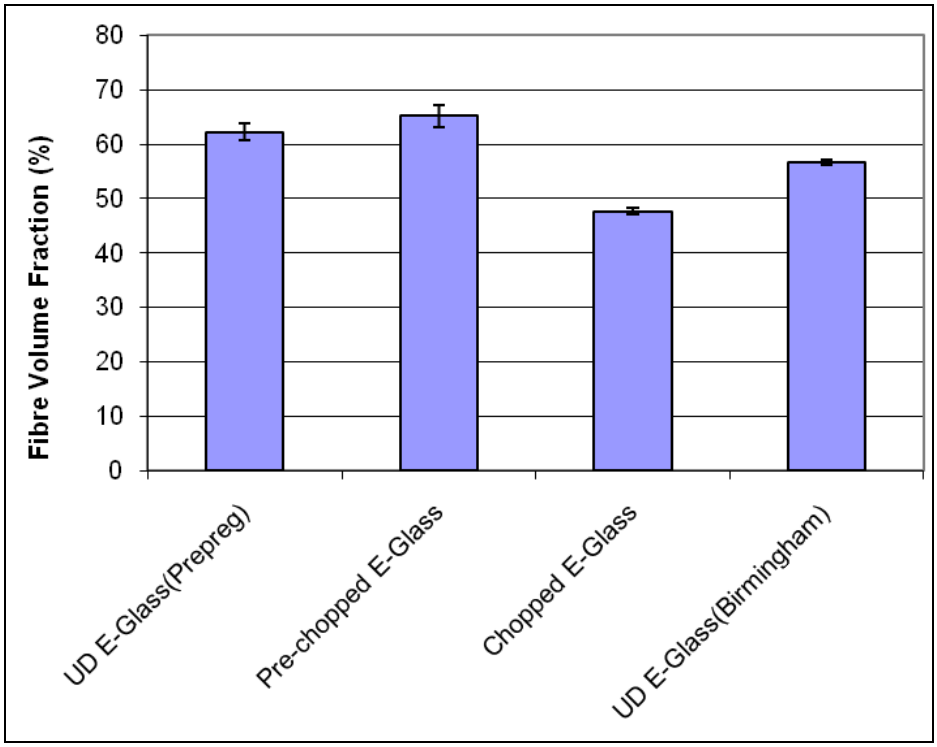


Figure 68. The fibre volume fraction results of the short-fibre composite plate (Pre-chopped) compared to a uni-directional E-Glass pre-preg, an in-house chopped and orientated E-glass fibre composite plate and a uni-directional E-Glass fibre plate produced in-house

With reference to Figure 68, the pre-chopped E-glass plate produced using Prototype-III was shown to have the highest fibre volume fraction of 65%, which would suggest a higher strength composite. The UD E-glass plate made in-house shows comparable strengths to a conventional pre-preg. It should be noted that all three types of samples sit at the top end of the commercial composite range of 30-70% for fibre volume fraction (Jones & DiBenedetto, 1993). The chopped E-glass was shown to have a lower volume fraction of 48% due to the fluffy nature of the fibres, whereas the pre-chopped E-glass fibres were able to be more closely packed due to them being heavily bound individual entities.

5.6.1.2.2 Void Content

The same burn-off procedure used for the calculation of the fibre volume fraction also produced results for the void content present within the samples. The results of this calculation can be found in Figure 69.

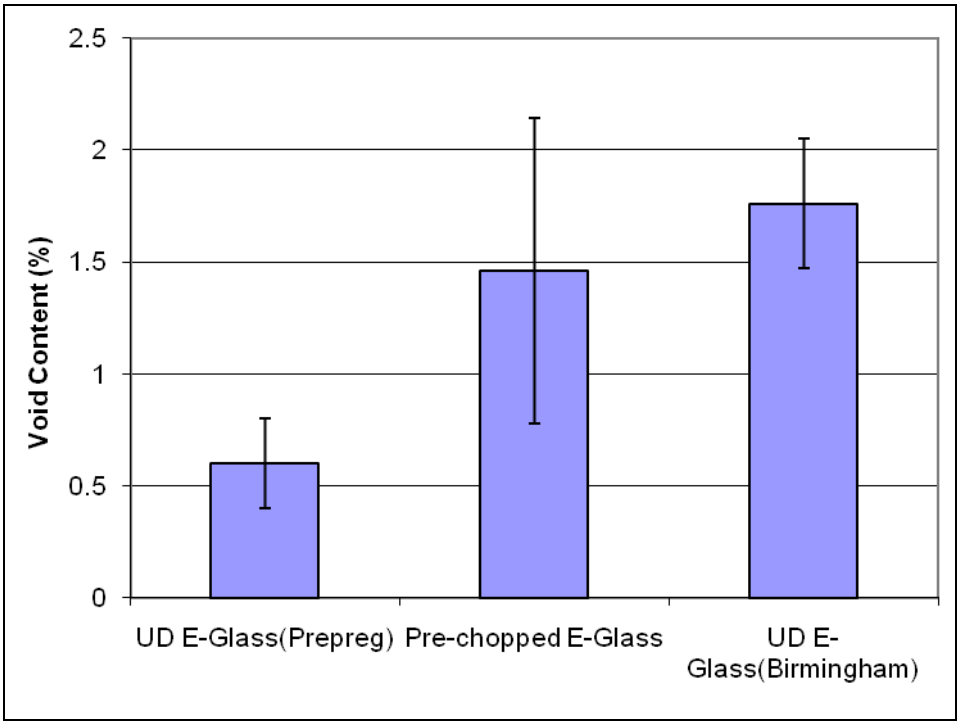


Figure 69. Graph comparing the void contents of the composite produced from pre-preg, pre-chopped E-glass fibre and continuous E-glass.

As expected, the E-glass pre-preg showed the lowest void content at an impressive 0.6%, over 1% less than the E-glass composite produced in-house. The pre-chopped E-glass fibre composite showed a comparatively lower void content than the UD E-glass. This is due to the smaller fibres being able to fill the areas that could represent voids. However, the values presented still fall within acceptable values since less than 1% is acceptable in aerospace applications and some automotive and marine applications accept voids greater than 5% (Suhot & Chambers, 2007).

5.6.1.3 *Mechanical Properties*

5.6.1.3.1 Tensile Testing

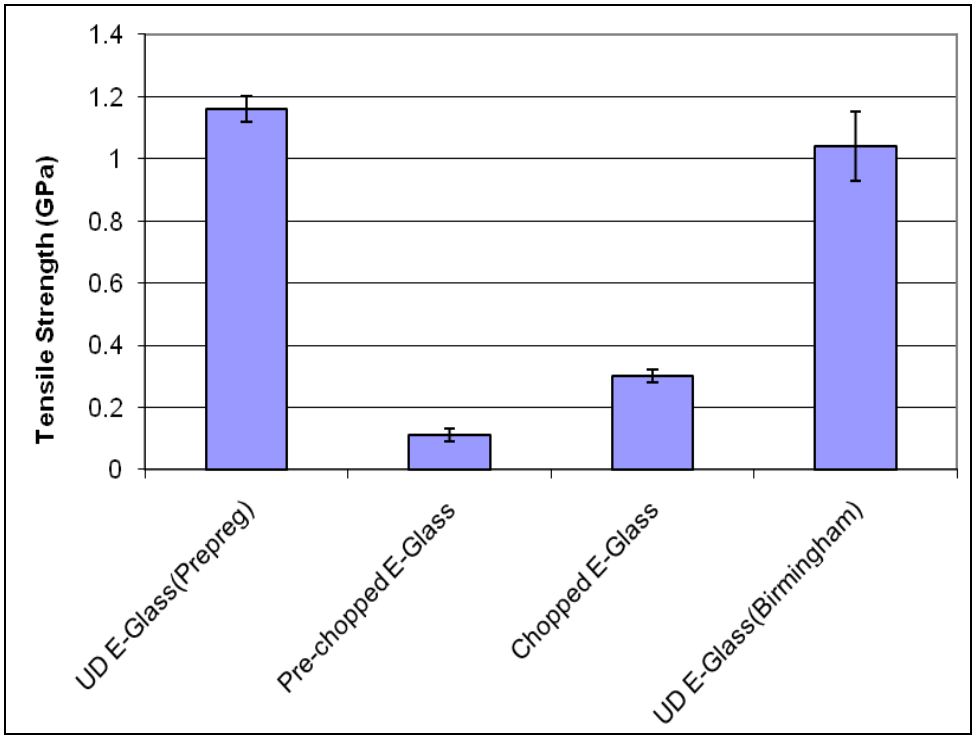


Figure 70. Graph comparing the tensile strengths of the four samples produced.

Figure 70 shows the result of tensile testing of the short-fibre composites, a UD E-glass composite produced in-house and an E-glass fibre composite produced from prepreg. As expected, the composite produced from pre-preg shows the highest tensile strength due to its high void content and considerably high fibre volume fraction. The pre-chopped fibre composite has a high fibre volume fraction and low void content but it is still a weak composite. The primary reason for this is due to the resin binder system present on the pre-chopped fibres. The resin binder was designed for use with thermoplastic resins and hence is not necessarily compatible with the fibres used. Nevertheless this was not a directly relevant issue as the focus of the study was to develop the short-fibre alignment and deposition technique.

This short length of the reinforcing fibre within the composite also directly affects the strength of the composite. The short-fibres therefore created stress concentrations at the end of the fibres when the composite was placed in tension (Maclaughlin & Barker, 1972). These stress concentrations resulted in a composite with a vastly reduced strength when compared to a continuous fibre composite (Kardos, 1985). The

slightly higher strength in the chopped fibre composites was due to the better impregnation achieved with this type of fibre as the binding in the pre-chopped fibres slightly hinders the impregnation process.

5.6.1.3.2 Flexural Strength

Due to several of the samples not being able to reach a failure point because of their high flexibility, Figure 71 shows the comparison of the samples flexural modulus for alternative strength comparison.

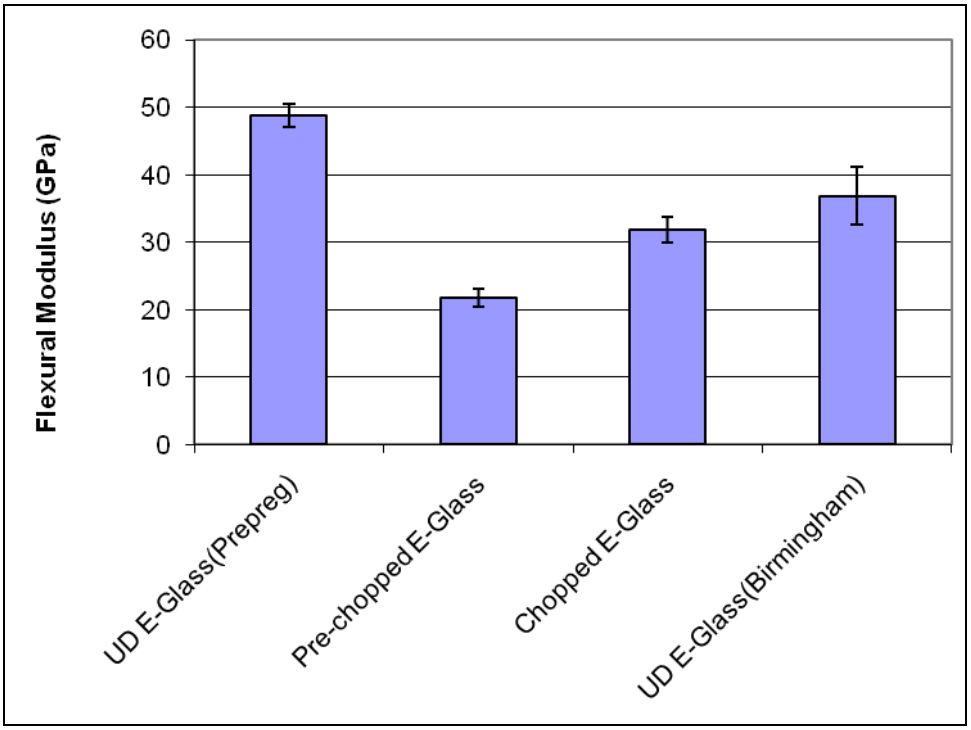


Figure 71. Graph comparing the flexural modulus of the four composite samples produced.

The same principles can be used to explain the differences in the flexural moduli for the four composite samples. The high fibre content and low void content in the pre-preg enabled it to be the strongest of the four composite samples produced. The pre-chopped E-glass sample was the weakest due to the considerably shorter reinforcing fibres. The fact that these were also more difficult to impregnate than the chopped fibres adds to the effect of the discontinuous reinforcement. The UD E-glass produced

in-house was again weaker due to its lower fibre volume fraction and higher void content than the pre-preg version.

6. Conclusions

This was a feasibility study to investigate two potential methods to manufacture composites from waste glass fibres and fabrics materials. Two forms of glass fibre waste materials were used namely, direct-loom waste (DLW) and waste slittings (WS). The waste glass fibres and fabrics were manufactured into composites using the recently developed clean filament winding (CFW) process, since is a low-tension process that is ideally suited for delicate preforms such as the DLW. The composites tubes compared in this study were therefore the direct-loom waste, waste slittings, virgin E-glass fibre tube produced in-house and two clean filament wound composite tubes produced on-site. The E-glass fibre tube produced in-house served as a reference material to which the waste fibre tubes would be compared whilst the on-site tubes would examine the feasibility of incorporating the clean filament apparatus on to a conventional filament winding machine. The primary end-use application for the filament wound tubes that were manufactured from the waste glass fabrics was to replace the cardboard tubes, currently used as the inner core for over-winding the woven glass fibre fabrics. The potential replacement of the cardboard tubes using filament wound tubes produced from waste glass fibre would help the industrial partner assess their waste management issues whilst providing a more durable cost effective inner tube. It was therefore necessary to compare the various tubes to determine their suitability for this application. These composites tubes were evaluated using the following techniques: inter-laminar shear strength (ILSS), hoop tensile strength, lateral compression strength, fibre volumes fraction and void content. Visual evaluation was also acquired through image analysis and scanning electron microscopy.

Upon physical property evaluation the CFW E-glass fibre tube produced in-house was shown to have the most superior fibre volume fraction and void content at 68.1% and 0.496% respectively. This makes even more impressive reading when compared to a conventional filament wound tube with a fibre volume fraction and void content of 48.86% and 1.19%. The tubes produced on-site at the faster and normal speeds were also able to show a comparative void content to the conventional tube however higher fibre volume fractions of approximately 60%. These results clearly show the benefit of clean filament winding and also indicate that it is possible to retrofit this apparatus

onto a conventional filament winding machine, to enhance its products and green credentials.

The waste fibre tubes however were shown not to be comparable to the E-glass fibres tubes with fibre volume fractions of 40.12% and 26.01% in the waste slittings and direct-loom waste. The lower fibre volume fractions were attributed to the difficulties in processing both types of waste fibre in terms of processing tensions, impregnation and poor spatial orientation of the fibres. The presence of a resin sealant of the surface of the waste slittings and hence poor impregnation was the cause of the highest void content discovered at 3.049%.

Upon mechanical property evaluation the CFW in-house tube was shown to be the most superior across ILSS and lateral compression strength whilst the CFW tubes produced on-site were superior across hoop tensile strength, with an increase in processing speed seeming to aid strength in the test. The waste slittings tubes was actually comparable to the virgin E-glass fibre tubes across ILSS with the resin sealant and excess resin not appearing to effect the bonding between composite layers. However, the key result from the mechanical evaluation was the waste fibre tubes performance in a lateral compression test. As stated previously the CFW glass-fibre tube was clearly superior in this test with the WS achieving a strength of 7.07 MPa approximately 38% of the strength of this CFW tube. However it was the waste fibres performance in comparison to the cardboard tubes currently used in the industrial application which was key. Both types of waste fibre tubes clearly outperformed the cardboard tubes and due to the superiority in strength and surface finish of the WS tubes it was this tube that was selected for deployment into the industrial application.

The recycling method of production was developed in order to orientate short glass fibres to replicate the form in which the recycled composite fibres would be supplied. A short-fibre vibration-based delivery system was presented and shown to be effective in orientating short (4.5mm) glass fibres in the production of a short-fibre composite. The short-fibres in this composite were shown to have a fibre alignment of over 80%. This short-fibre composite was also able to show 1.5% void content and 65% fibre volume fraction making this comparable to a continuous fibre reference. Nevertheless the short nature of the fibre and difficulty impregnating this fibre resulted in the weakest composite in the comparisons of flexion and tension.

7. Future Work

Initially the next step for this project would be to place the composite tubes into service and evaluate its performance. Based on the service life of the composite tube an LCC (life cycle cost) and LCA (Life cycle analysis) could then be completed to really appreciate the benefits attained from using this replacement tube. The results of this LCA and LCC could also open the opportunity for further cost savings with the investigation of different resin systems.

Another stage would be to further investigate methods to process the direct-loom waste. Based on the properties attained from a slow filament winding method there does seem to be a potential to use this material in some form. However in order to make a viable tube it would have to be produced at higher speeds and easier to incorporate into the clean filament winding process. The management of this type of fibre with another waste material may be a possibility.

As the orientation of short-fibres appears to be possible, further investigation into the optimisation of the composites properties appears to be the next stage for this area of work. The prospect of combining the two processing methods to create a hybrid composite of both short and continuous fibre would also be a very interesting concept to pursue.

8. Publications

1. “An investigation into techniques to fabricate highly aligned short-fibre prepregs”, Shotton-Gale, N., Paget, M. A., Smith, C., Jameson, N., Wang, L., Malik, S. A., Burns, J. M., Biddlestone, F., Prasad, A., Harris, D., Machavaram, V. R., Mahendran, R. S. and Fernando, G. F., SAMPE Europe, April 2010, Paris, France. (2010).
2. “Manufacture and evaluation of filament wound tubes from loom (weaving) waste”. Smith, C., Shotton-Gale, N., Wait, C., Paget, M., Harris, D., Machavaram, V. R., Wang, L., James, J., Price, R. and Fernando, G. F., SAMPE Europe, April 2010, Paris, France. (2010)

9. References

Asokan P, Osmani M. and Price A.D.F. (2010) Improvement of the mechanical properties of glass fibre reinforced plastic waste powder filled concrete. Construction and building material, 24, 448.

Asokan P, Osmani M and Price A. D. F. (2009) Assessing the recycling potential of glass fibre reinforced plastic waste in concrete and cement composites. Journal of cleaner production, 17, 821.

Bartl A, Hackl A, Mihalyi B, Wistuba M and Marini L. (2005) Recycling of fibre materials. Trans IChemE, Part B, Process Safety and Environmental Protection, 8, 351.

Bernasconi A, Rossin D and Armanni C. (2007) Analysis of the effect of mechanical recycling upon tensile strength of short glass fibre reinforced polyamide 6, 6. Engineering fracture mechanics, 74, 627.

Boeing Environmental Technotes (2003). Composite Recycling and Disposal. An Environmental R&D Issue, 8, 4.
http://www.boeingsuppliers.com/environmental/TechNotes/TechNotes2003-11.pdf - Accessed 13/10/2010.

Chu J and Sullivan J.L. (1996) Recyclability of a continuous E-Glass Fiber Reinforced Polycarbonate Composite. Polymer Composites, 17, 556.

Cunliffe A.M. and Williams P.T. (2003) Characterisation of products from the recycling of glass fibre reinforced polyester waste by pyrolysis. Fuel 82, 2223–2230.

Conroy A, Halliwell S, and Reynolds T (2005). Composite recycling in the construction industry, Composites: Part A 37 (2006) 1216–1222

Council Decision of 19 December 2002 establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 of and Annex II to Directive 1999/31/EC (2003). Official Journal of the European communities 11/27.

Darling, A. (2010) Chancellor extends landfill escalator until 2014. http://www.letsrecycle.com/do/ecco.py/view_item?listid=37&listcatid=5504&listitemid=54931 – Accessed 02/02/10

Directive 2008/98/EC of the European Parliament and council of 19 November 2008 on waste and repealing certain directives. (2008) Official Journal of European Union 312/3.

Directive 2008/1/EC of the European Parliament and council of 15 January 2008 on waste and repealing certain directives. (2008) Official Journal of European Union 24/8.

Directive 2000/53/EC of the European Parliament and of the Council of 18 September 2000 on end-of-life vehicles, (2000) OJ L 269/34.

Flemming, T., Kress, G. and Flemming, M. (1996) A new aligned short-carbon-fiber-reinforced thermoplastic prepreg. Journal of advanced composite materials, 5, 151.

Fu, S-Y and Lauke, B. (1996) Effects of fiber length and fiber orientation distributions on the tensile strength of short-fiber-reinforced polymers. Composite science and Technology 56, 1179.

Fu, S.-Y., Lauke, B., Mäder, E., Yue, C.-Y. and Hu, X. (2000) Tensile properties of short-glass-fiber and short-carbon-fiber-reinforced polypropylene composites. Composites part A: Applied science and manufacturing, 31, 1117.

Guell D. C. and Graham A. L. (1996) Improved mechanical properties in hydrodynamically aligned, short-fibre composite materials. Journal of composite materials, 30, 2.

Gupta, N. K and Abbas, H. (2000), Int. J. Imp. Eng. 24: 329

Harper, L. T. Turner, T. A. Martin J. R. B. & Warrior N. A. (2009) Fiber Alignment in Directed Carbon Fiber Preforms – A Feasibility Study. Journal of Composite Materials, 43, 57.

Hine P. J, Tsui S.-W, Coates P. D, Ward I. M. and Duckett R. A. (1997) Measuring the development of fibre orientation during the melt extrusion of short glass fibre reinforced polypropylene. Composite A, 28, 949.

Itoh T, Masuda S, and Gomi F. (1994) Electrostatic orientation of ceramic short fibers in liquid. Journal of Electrostatics, 32, 71.

Jiang G, Pickering S.J, Lester E.H, Turner T.A, Wonga K.H and Warrior N.A (2008) Characterisation of carbon fibres recycled from carbon fibre/epoxy resin composites using supercritical n-propanol. Composites Science and Technology 69, 192.

Jones K.D and DiBenedetto A.T. (1993) Fibre fracture in hybrid composite systems. Composites Science and Technology, 51, 53.

Kardos J.L. (1985) Critical issues in achieving desirable mechanical properties for short fiber composites. Pure & Applied Chemistry, 57, 1651.

Kanari N, Pineau, J.-L. and Shallari S. (2003) End-of-life vehicle recycling in the European union. JOM, a publication by The Minerals, Metals & Materials Society, 15.

Kennerley J.R, Kelly R.M, Fenwick N.J, Pickering S.J, Rudd C.D. (1998) The characterisation and reuse of glass fibres recycled from scrap composites by the action of a fluidised bed process. Composites Part A, 29, 839.

Kim Y. K, Langley K. D, Lewis A. F. and Seyam A. Electro-static web formation. NTC Project: F03-MD01 National Textile Center Annual Report, 2006.

Kouparitsas C.E, Kartalis C.N, Varelidis P.C, Tsenoglou C.J. and Papaspyrides C.D. (2002) Recycling of the fibrous fraction of reinforced thermoset composites. Polymer composites, 23, 682.

Kilde and Larsen (2000) as cited by the European Environment Agency. Number of cars to be scrapped in Europe. In *UNEP/GRID-Arendal Maps and Graphics Library*. Retrieved 15:54, October 13, 2010 from http://maps.grida.no/go/graphic/number_of_cars_to_be_scrapped_in_europe

Larsen, K. (2009). Recycling wind turbine blades. Renewable energy focus, 9, 7.

Lester E, Kingman S, Wong K. H, Rudd C, Pickering S and Hilal N. (2004) Microwave heating as a means for carbon fibre recovery from polymer composites: a technical feasibility study. Materials Research Bulletin, Volume 39, 1549.

MacLaughlin, T. F. and Barker, R. M. (1972) Effect of modulus ratio on stress near a discontinuous fibre. Experimental mechanics, 12, 178.

McConnell V.P. (2010) Launching the carbon fibre recycling industry. http://www.reinforcedplastics.com/view/8116/launching-the-carbon-fibre-recycling-industry - Accessed 13/10/2010

McKechnie and Wegman (2008) ECRC heads search for composites recycling solutions. http://www.reinforcedplastics.com/view/1089/ecrc-heads-search-for-composites-recycling-solutions/ - Accessed 13/10/2010

Mouhmid, B., Imad, A., Benseddiq, N., Benmedakhène, S. and Maazouz, A. (2006) A study of the mechanical behaviour of glass fibre reinforced polyamide 6, 6: Experimental investigation. Polymer testing, 25, 544-552.

Oliveira G.H, Guimaraes V.A. and Botelho E.C. (2009) Influence from the Temperature on the Mechanical Behavior of Pei/Glass Fiber Composites. Polimeros-ciencia e Technologia, 4, 305.

Otheguy M. E, Gibson A. G, Findon E, Cripps R. M, Ochoa Mendoza A and Aguinaco Castro M. T. (2009) Recycling of end-of-life thermoplastic composite boats. Plastics, Rubber and Composites, 38, 9/10.

Palmer J, Ghita O.R, Savage L, Evans K.E. (2009) Successful closed-loop recycling of thermoset composites. Composites: Part A 40, 490.

Pandita, S. D., Smith, S., S-Gale, N., Paget, M., Allen, J. M., Fernando, G. F. (2007), "Clean" Filament Winding: A New Manufacturing Process

Papathanasiou T.D, Ingber M.S, Guell D.C. (1995) Stiffness enhancement in aligned, short-fibre composites: A computational and experimental investigation. Composites science and technology, 54, 1.

Peng J, Lin T. L. and Calvert P. (1999) Orientation effects in freeformed short-fibre composites. Composite A, 30, 133.

Perrin D, Clerc L, Leroy E, Lopez-cuesta J.M. and Bergeret A. (2007) Optimizing a recycling process of SMC composite waste. Waste management, 28, 541.

Pinero-Hernanz R , Dodds C , Hyde J , Garcia-Serna J ,Poliakoff M , Lester E , Coceroa M.J, Kingman S ,Pickering S and Wong K.H.(2008), Chemical recycling of carbon fibre reinforced composites in nearcritical and supercritical water. Composites: Part A 39, 454–461.

Pinero-Hernanz R, Garcia-Serna J, Dodds C, Hyde J, Poliakoff M, Cocero MJ, Kingman S, Pickering S and Lester E. (2008) Chemical recycling of carbon fibre composites using alcohols under subcritical and supercritical conditions, Journal of Supercritical Fluids 46, 83–92

Pickering S.J, Kelly R.M, Kennerley J.R, Rudd C.D and Fenwick N.J. (2000). A fluidised-bed process for the recovery of glass fibres from scrap thermoset composites. Composites Science and Technology 60, 509-523.

Sanomura, Y & Kawamura, M. (2003) Fiber orientation control of short-fiber reinforced thermoplastics by ram extrusion. Polymer composites, 24, No. 5.

Sarasou, J.R. & Pouyet, J (1997). Recycling effects on microstructure and mechanical behaviour of PEEK short carbon-fibre composites. Journal of Materials Science 32 533 - 536.

Sarioglu A, Hagstrand P-O. and Manson J-A. E. (2004) Fibre orientation in multilayer tubes processed by a conical extruder. Polymer composites 25 (3), 331-41.

Shotton-Gale, N., Harris, D., Pandita, S. D., Paget, AM. A., Allen, J. A. and Fernando, G. F. (2010) Clean and environmentally wet-filament winding Chapter 13. Management, recycling and reuse of waste composites, Edited by V. Goodship, Woodhead Publishing Limited. ISBN 978-1-84569-462-3.

Steenkamer, D.A. and Sullivan, J.L. (1998). On the recyclability of a cyclic thermoplastic composite material. Composites Part B, 29, 745.

Suhot M. A. and Chambers A.R. (2007) The effects of voids on the flexural fatigue performance of unidirectional carbon fibre composites. 16th International conference on composite material, Japan.

Taib, R.M. (1998) Cellulose fiber-reinforced thermoplastic composites: processing and products characteristics. Thesis. Virginia Tech, Blacksburg. pp. 123.

Torres A, De Marco I, Cabellero B.M, Laresgoiti M.F and Chomon M.J. (2009) Recycling of the solid residue obtained from the pyrolysis of fibreglass polyester sheet moulding compound. Advances in polymer technology, 28,141.

White F.G. and Abdin E.M.Y. (1985) Dynamic properties of aligned short carbon fibre-reinforced plastics in flexure and torsion. Composites, 16, 293.
www.matweb.com (accessed 30/05/10)

Wait C (2010) The reuse and recycling of glass fibre waste. A University of Birmingham Masters thesis.

Yamashita S, Hatta H, Sugano T. and Murayama K. (1989) Fibre orientation control of short fibre composites: experiment. Journal of composite materials. 23 32-41.

Yip H. L. H, Pickering S. J, and Rudd C. D. (2002). Characterisation of carbon fibres recycled from scrap composites using fluidised bed process. Plastics, Rubber and Composites, 31, 6.

Yuyan L, Linghui M, Yudong H & Lixun L. (2006) Method of Recovering the Fibrous Fraction of Glass/Epoxy Composites. Journal of Reinforced Plastics and Composites; 25; 1525

Zhang T, Evans J. R. G. and Bevis M. J. (1997) Control of fibre orientation in injection moulded ceramic composites. Composites Part *A* 28, 339.

Zhu H, Li D, Zhang D, Whu B and Chen Y. Influence of voids on interlaminar shear strength of carbon/epoxy laminates. Trans. Nonferrous Met. Soc China, 19, 470.