Hybrid Composite Structures: Multifunctionality Through Metal Fibres

An exploratory investigation

Proefschrift

ter verkrijging van de graad van doctor
aan de Technische Universiteit Delft,
op gezag van de Rector Magnificus prof. dr. ir. J.T. Fokkema,
voorzitter van het College voor Promoties,
in het openbaar te verdedigen op vrijdag 17 april 2009 om 10:00 uur

doors

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This research was carried out as part of the innovation program of the Materials innovation institute (M2i) (formerly, the Netherlands Institute for Metals Research) on "Hybrid Structures - Metal Fibres, Yarns and Textiles for polymer Composite Structures", project number "MC1.03165"


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Cover design: T.J. Ahmed
Photography: F. Oostrum

Printed in the Netherlands by Ipskamp Drukkers B.V.
"He who laughs, lasts"
To my family...
The introduction of fibre reinforced polymer composites into the wings and fuselages of the newest aircraft are changing the design and manufacturing approach. Composites provide greater freedom to designers who want to improve aircraft performance in an affordable way. In this quest, researchers are looking to the area of multifunctional structures, which represents a new manufacturing and integration methodology. Composite materials are ideally suited to achieve multifunctionality because the best features of different materials can be combined to form a new material that has a broad spectrum of desired properties.

Along these lines, this thesis explores the idea of structural multifunctionality of reinforced polymer composites through the addition of metal fibres. In this way, a new family of hybrid composite materials may be produced. Through their unique inherent properties, metal fibres offer the opportunity to expand the design envelope to incorporate tuneable properties such as ductility and electrical conductivity, for example.

In this undertaking, the research was conducted in three main phases: (i) to define the properties of the metal fibres themselves, (ii) to determine the extent to which these properties contribute to the overall mechanical behaviour of the composite, and (iii) to manipulate the properties of the fibres to add functionality to the composite.

Out of the many metal types, stainless steel was chosen for its superior mechanical properties. Three forms of fibres were identified; fibre bundles, fine wires and meshes, and mechanically tested. It was found that there were variations in properties dependant on fibre diameter, treatment and extent of surface flaws.

Improvements in metal fibre-matrix adhesion were also demonstrated to be possible using traditional sheet metal surface treatments such as anodising; however, these results are limited by fibre diameter. Below a certain diameter, such surface treatments become too aggressive resulting in damage to the fibres. Emerging non-aggressive surface techniques may provide an answer to this shortcoming, but require further development for this application.

Changes in mechanical behaviour in terms of low velocity impact and flexure were demonstrated for the hybrid composites with metal fibres compared to those without. A complex interaction between the matrix and the metal fibres has been identified as the mechanism responsible. Through intelligent placement of the metal fibres in the hybrid composite to exploit this interaction, various improvements in mechanical behaviour can be realised.

Finally, innovative applications for hybrid composites were investigated, exploiting the electrical and thermal properties of the metal fibres. The first was aimed towards aiding thermographic non-destructive evaluation, and the second to provide an integrated ice protection system concept, thereby opening up new applications for the novel material concept through adding functionality to the structure.
Samenvatting

De introductie van vezelversterkte kunststoffen in de vleugels en rompen in nieuwe vliegtuigen zorgt voor verandering in de ontwerp- en productiemethodes. Composieten verschaffen een grotere vrijheid voor ontwerpers die vliegtuigprestaties willen verbeteren op een betaalbare wijze. Ontwerpers zoeken hiervoor naar multifunctionele constructies die leiden tot nieuwe productie en integratie methodologien. Multifunctionaliteit kan heel goed met composieten bewerkstelligd worden omdat de beste eigenschappen van verschillende materialen gecombineerd kunnen worden tot een nieuw materiaal met een breed spectrum van wenselijke eigenschappen.

Met dit in gedachte, wordt in deze thesis het idee van structurele multifunctionaliteit van vezelversterkte kunststoffen door toevoeging van metaalvezels onderzocht. Door hun unieke intrinsieke eigenschappen bieden metaalvezels de gelegenheid om de ontwerpruimte uit te breiden en aanpasbare eigenschappen zoals taaheid en het elektrische geleidingsvermogen in composieten te verweven.

Het onderzoek was in drie hoofdfases verricht: (i) het bepalen van de eigenschappen van de metaalvezels, (ii) vaststellen in welke mate deze eigenschappen bijdragen aan het gehele mechanische gedrag van composieten en (iii) de eigenschappen van de vezels te manipuleren om multifunctionaliteit aan het composiet toe te voegen.

Van alle beschikbare metaalsoorten was roestvrij staal gekozen vanwege zijn superieure mechanische eigenschappen. Drie verschillende vezelvormen werden gedetermineerd en getest; vezelbundels, fijne draden en gazen. De variaties van de eigenschappen die werden vastgesteld waren afhankelijk van de vezeldiameter, de behandeling en de omvang van oppervlaktegebreken.

Verbeteringen in de hechting van de metaalvezel met de matrix, bereikt door gebruik te maken van traditionele oppervlaktebehandelingen voor metaalplaten, zoals anodiseren, konden ook aangewezen worden. Deze resultaten zijn echter afhankelijk van de vezeldiameter. De gebruikte oppervlaktebehandelingen worden te agressief beneden een bepaalde vezeldiameter, wat leidt tot schade aan de vezels. Nieuwe niet-agressieve oppervlakte technieken kunnen een uitkomst bieden voor deze tekortkoming, maar vereisen nog verdere ontwikkeling voor deze toepassing.

Het is aangetoond dat in geval van buiging en impact onder lage snelheid, hybride composieten met metaalvezels een ander mechanisch gedrag vertonen in vergelijking met composieten zonder metaalvezels. Een complexe wisselwerking tussen de matrix en de metaalvezels is gedetermineerd als het verantwoordelijke mechanisme voor deze verandering. Verschillende verbeteringen in mechanisch gedrag kunnen door deze wisselwerking gerealiseerd worden door intelligente plaatsing van metaalvezels in composieten.

Tot slot werden innovatieve toepassingen voor hybride composieten onderzocht die gebruik maken van de elektrische en thermische eigenschappen van metaalvezels. De eerste toepassing was het ontwikkelen van een thermografische, niet destructieve evaluatie voor composieten en de tweede toepassing was de integratie van een systeem voor ijsbescherming in composieten. Hierdoor worden nieuwe toepassingen voor revolutionaire materiaalconcepten gecreëerd door het toevoegen van functionaliteit aan de constructie.
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Chapter 1

Introduction

Metals have been dominating materials in structural applications throughout history and only recently have fibre reinforced composites become a serious contender to this title. The ease of processing of metals mainly relies on their ductility and without this deformation ability, metal usage would not have played such a large part in history. In contrast, continuous fibre reinforced polymer composites (CFRPCs) are hindered by their inability to greatly plastically deform due to the nature of the brittle reinforcing fibres. However, more lightweight structures can be produced and it is for this reason that the aerospace and automotive industries have realised their importance. In the same way, short fibre reinforced polymer composites (SFRPCs) display poorer mechanical properties compared to their heavier metal counterparts. If a medium between CFRPCs and metal could be found, it would be possible to have the benefits of both materials.

In current design and manufacturing of lightweight composite structures the engineer is not only designing the shape and functionalities of the structure but also the mechanical behaviour or response to mechanical loads within an envelope of sufficient resistance to temperatures, chemical and physical degradation. The structural design nowadays is limited to mechanical and environmental loading and the design envelope needs an extension to parameters with respect to tuneable electric and magnetic properties, thermal conduction, thermal expansion behaviour and, last but not least, tuneable ductility when appropriate. The contemporary families of organic and inorganic reinforcement fibres provide a limited extension to the design envelope. However, metals in fibre form provide and interesting way to further increase the possibilities for added functionality.

When metal fibres of any nature and morphology become available and selected with respect to mechanical, chemical and physical properties, a new range of materials and structure design possibilities emerge. By hybridising contemporary organic and inorganic fibres with metal fibres of different but compatible nature, we extend the possibilities through mix-and-match. In addition, metal fibres can be placed in specific locations within the composite structure for localised property enhancement.

Metal fibres are available in various forms, and for this thesis three different forms have been defined: fine wires (single filaments), fibre bundles (fibre tows) and meshes (woven filaments), Figure 1.1. Previous studies on the use of metal fibres in composites have mainly focused on use for electromagnetic shielding. Rarely has a study been performed where the continuous metal fibre is the second reinforcing component in a composite, and therefore has yet to be fully realised.
Chapter 1. Introduction

1.1 Research goals

The research goals are as follows:

• To provide an overview of the current state-of-the-art for metal fibres, specifically focusing on use for polymer composite materials.

• To discover the added value of the addition of metal fibres to the composite structure. In particular, the scope for added functionality is to be determined.

In order to realise these goals, aside from the metal fibres, other reinforcing fibres were restricted to glass and carbon. One thermoset and two thermoplastic polymers were used to determine, if any, compatibility issues.

The thesis begins with a literature review in Chapter 2 on the background of metal fibres, production methods and previous research. In this chapter, an explanation is provided for the use of stainless steel fibres in this thesis. Three forms of stainless steel fibres are tested for their material properties in Chapter 3, and efforts to improve metal/matrix adhesion is detailed in Chapter 4. Chapters 5, 6 and 7 deal with the mechanical properties of composites hybridised with metal fibres. Finally, four novel ways of adding functionality to the composite is outlined in Chapters 8, 9, 10 and 11. Finally, conclusions and recommendations for future research is presented in Chapter 12.
Chapter 2

Literature review

This literature review collates all the information that has been gathered on metal fine wires, fibres and textiles and their use in composites to date. The processing techniques and properties of various metal fibres are first described followed by a review of previous research and applications.

2.1 Metal fibre production

A number of methods exist for producing metal fibres, which can be divided into continuous and discontinuous processes. The material properties of the bulk metal determine which of the methods can be used, which in turn depends on the application. Since it is the focus of the research to apply metal fibres to advanced composites, short, discontinuous fibres were not of interest. Hence, only the continuous methods to produce long fibres will be considered here.

2.1.1 Fine wire drawing

Drawing is the action of pulling a section of metal through a wear-resistant die, resulting in a reduction of cross-sectional area and an increase in length. Due to the die, the wires are perfectly round in cross-section with a smooth surface. A drawing apparatus often consists of a line of dies, each slightly smaller in diameter than the preceding one. A complete drawing apparatus may include up to twelve dies in a series sequence, each with diameter 20-30% smaller than the preceding one [1]. In multiple-die machines, each stage results in an increase in length and therefore a corresponding increase in speed is required between each stage. This is achieved using capstans which are used both to apply the tensile force and also to accommodate the increase in the speed of the drawn wire, which are typically around 40m/s but may reach up to 100m/s [2]. Figure 2.1 shows an example of a drawing die and a schematic of the drawing process.

The mechanical properties of the final wire depend on the chemical composition of the starting metal, the metal treatment and the final diameter reduction, or end-reduction. The metal wire workhardens during plastic deformation and the ductility is reduced while the tensile strength and hardness increases. As with any metal that has been drawn, the increase in tensile strength arises from elongation and alignment of the crystalline grains along the wire axis, as shown in Figure 2.2(a). Generally, the smaller the grains, the stronger the metal. To continue wire drawing to very small diameters, intermediate annealing is necessary to restore ductility. Hence the end-reduction is of importance because the greater the diameter reduction, the more work-hardening can occur, and therefore the greater the mechanical properties [3]. Depending on the application, a final stage of annealing is also
Chapter 2. Literature review

Figure 2.1 Cross-section of a typical die (left) [4] and a schematic of the wire drawing process (right) [5].

applied to the wire to restore full ductility albeit at the expense of strength and hardness, Figure 2.2(b).

In theory, almost all types of metal may be drawn and differ only in the possible size of the final wire diameter, which in turn depends on the initial chemical composition. Stainless steel, for example, may be drawn to a final diameter of 18\(\mu\)m, whereas titanium may be drawn to 40\(\mu\)m. The production process for wire drawing is costly and increases with decreasing fibre diameter due to the additional dies and subsequent drawing and annealing steps.

2.1.2 Taylor-wire process

As an alternative to the expensive wire-drawing process, other techniques exist to offer lower-cost fine-wire production. Of these, the Taylor process has received the most attention and has been previously reviewed by Donald [6]. The Taylor-wire [7], or microwire, process is a production method that produces the fibres directly from the melt in a single step, thereby reducing cost. Metal is held in a glass tube that is sealed at one end and induction coils provide the heat necessary to melt the metal. Once the metal has been melted, the sealed end of the tube is heat softened concurrently and drawn to produce a metal filament with a glass coating. This glass coating may then be removed and the metal retained if necessary. Drawing speeds of up to 16m/s have been achieved [6] and fibre diameters in the range of 1-100\(\mu\)m have been produced. The resulting fibres are circular in cross-section and have a smooth surface. Figure 2.3 shows a schematic of the Taylor-wire process and an example of a glass-coated wire.

The fibres produced with this method become stronger and more brittle with decreasing diameter [6]. A number of metals may be drawn in this way, as long as there is compatibility with the glass, and the softening point of the glass lies between the metal melting and boiling points [6]. However, industrial application for Taylor-wires is currently rather limited, and the technique has been most commonly used on laboratory scales. The limiting factor may be due to non-uniformity of the glass coating produced during cooling, which in turn affects the properties of the wires [8]. Currently, wires with electromagnetic (EM) properties are produced with the process for use as, for example, EM shielders and anti-shoplifting labels.

2.1.3 Bundle-drawing

The process of bundle-drawing first arose as a way of reducing the cost of individually drawing fine wires, by drawing a number together at the same time [10]. In this process, a metal wire or rod is
2.1 Metal fibre production

(a) Work-hardening

(b) Annealing

Figure 2.2 Effect of metal treatment on the grain size and mechanical properties.

made to undergo a series of drawing and annealing steps. Once a diameter of 1mm has been reached, the wire is bundled and coated with another metal that is able to withstand the drawing and annealing processes, typically iron. The bundle is then in turn drawn and annealed to the desired diameter. In the final step, the bundles are leached to dissolve the coating material, releasing the individual metal fibres. In this way, depending on the chemical composition of the initial metal, fibres with a diameter down to less than 1µm may be produced. The process is schematically shown in Figure 2.4.

As with the fine wires, the mechanical properties of the fibres depend on the chemical composition of the metal, metal treatment and end-reduction. As before, the tensile strength of the fibres increase with decreasing diameter as the grain size elongate and become highly aligned. However, the extent of the tensile strength increase is of yet unknown of the fibres produced with this method. Due to the coating and leaching processes, the fibres are irregular in cross-section and have a very rough surface as shown in Figure 2.5.

Not all metals may be bundle-drawn and consist mainly of alloys containing nickel, such as stainless steel, nichrome and Hastelloy X, and also titanium [10]. Although producing bundles of metal fibres would appear more cost-effective than wire-drawing, the same amount of subsequent drawing and annealing steps are the same and it is not possible to extract individual fibres from the bundle. The additional leaching step is also an expensive process and therefore cost reduction achieved by drawing a number of fibres together is negated.

2.1.4 Fabrics

The geometry of fabric materials can be thought of as a hierarchical structure. Metal fibres are twisted in bundles to produce yarns and yarns are knitted or woven to produce fabrics. The knitting of metal in staple yarn form into a fabric is not an easy process and many staple yarns are also very expensive. To compensate, the yarns have often been blended with polyester fibres. In some cases, not only is the cost lowered, but the overall material has a lower bending rigidity and coefficient of friction than
Chapter 2. Literature review

Figure 2.3  Taylor-wire process (left) and a glass-coated wire (right) [9]

Little damage to the metal fibres has been found due to the lubricating effect of the polymer binding yarns. Uncommingled yarns can be produced with a polymer matrix fibre used as a binding yarn, which is wrapped around bundles of metal, and other, fibres. Under high temperature and pressure, the polymer yarns melt and impregnate the fibres resulting in knitted-fabric-reinforced thermoplastic composites [12, 13].

Figure 2.4  Wire bundle drawing [11].

HDJ Isomuvotherm b.v. have found a way to incorporate stainless steel fibres into woven textile fabric to produce a material that can be very uniformly heated even over a relatively large area. Although the fibres are not blended with another material, the metal is simply wound around the textile yarns in a similar way as the polypropylene fibre in Figure 2.6, which not only produces a large surface area in which heat is emitted, but also facilitates ease of weaving. If a matrix can be incorporated

Figure 2.5  Longitudinal (left) and cross-sectional (right) microscope images of a bundle-drawn metal fibre.
into this system in the same way as with knitted-fabric-reinforced thermoplastic composites, then this fabric can also be made into a composite with both electrical and mechanical potential.

The two methods mentioned so far are based on the fact that the metal fibres would be wrapped within, or around, another fibre. Another potential method would be if the metal was woven, or knitted, individually either as twisted fibres or fibrous strips into the existing fibre material.

2.2 Metal fibre types

A number of metals are available in various fibre form, in theory providing a pool from which desirable properties may be extracted for any application. However in reality, a number of practical factors limit the number of fibre types that are feasible, especially for polymer composite applications, where mechanical properties are particularly important. The most important of these are:

- Quantity - while almost every metal can be made into a wire or fibre, the abundance of the metal is one factor that determines availability. In addition, a relatively large quantity must also be available to determine the feasibility of application.
- Form - certain metal fibres are only available in a small range of diameters and cross-sectional form. This can be due to difficulties in producing the fibre because of its intrinsic properties, or due to a limited state-of-the-art production process.
- Existing usage - a number of commercial applications already exist for metal fibres but usage for more advanced engineering applications, such as load-bearing structures, has not been fully explored. The quantity of available metal fibres is therefore also dependent on the existing use.
- Cost - the cost of metal fibres is determined by a number of factors such as fibre type, abundance, production procedure, diameter, form and so on. The use of the metal fibre is driven by the application, which in turn is also affected by the cost of the materials. Therefore the price of the metal fibre can limit the feasibility for a given application.

With these factors in mind, and before considering polymer composite application, it is possible to reduce the number of potential metals in fibre form to a relative few.

2.2.1 Traditional primary metals and their alloys

Aluminium is one of the key materials used in aircraft structures and has been used in ever increasing quantities since the first powered flight over a century ago. Aluminium alloys are used in a large number of aerospace applications, ranging in complexity and performance requirements from simple components right through to primary load bearing structures in aircraft. Generally, these alloys are characterised by their low density, high electrical and thermal conductivities and good resistance to
chemical corrosion. Aluminium is also recyclable and properties do not degrade with the recycling process.

First mined over 5000 years ago, the use of copper dates back to prehistoric times and has been used in numerous applications, ranging from printed circuits to fasteners. It is tough, ductile, malleable and has a conductivity that is rivalled only by silver. Generally resistant to corrosive environments, copper is nevertheless susceptible to oxidising agents.

Although in its pure state, nickel is hard, ductile, malleable and slightly ferromagnetic, it is mainly used in alloys such as stainless steels and other corrosive resistant materials such as the super-alloys Inconel and the Hastelloys, and Nichrome.

Pure tantalum is a heavy, dense, malleable and ductile metal. Add to this the exceptional resistance to corrosion and acid attack and the properties allow the metal to be used as a substitute for platinum in laboratory equipment in the chemical industry and for surgical implants. Finally, tantalum is used as an alloying element to produce alloys of good corrosion resistance, strength and ductility [14].

Tungsten has the benefit of having the highest elastic modulus and melting point of all the pure metals. In the fibre form, tungsten has been used as reinforcements for superconductor filaments due to its resistance to large tensile stresses produced by the electromagnetic force during operation [15]. Much research on these fibres in composites have been focused on their use in metal matrices for improvement in toughness [16, 17].

All these metals are available in fine wire form, but only nickel and its alloys are available as bundle-drawn fibres.

2.2.2 Stainless steels

As will be described in the next few sections, stainless steel fibres have shown the greatest potential in terms of availability, application and material properties. The presence of chromium produces a self-healing oxide film responsible for the "stainlessness" of these metals and results in a high resistance to corrosion. Further improving the resistance to corrosion is achieved by the inclusion of nickel and molybdenum. A wide range of mechanical properties obtainable from these alloys, along with their corrosion resistance, and in some cases, resistance to oxidation at elevated temperatures, makes them extremely versatile in their application [18].

Stainless steels represent a huge class of metals, all of which can be grouped into four classes; austenitic, martensitic, ferritic and precipitation hardened. Due to its ease of use, ease of production and mechanical properties only austenitic steels, represented by the AISI300 series, are readily available in fine wire and fibre forms.

2.2.3 Titanium

A relatively new metal in comparison with other metals being considered, titanium possesses a remarkable set of properties. The combination of extremely high strength and corrosion resistance, high melting point and low density has found its use in a number of structural applications, ranging from aircraft structures to chemical plants. At elevated temperatures, titanium becomes chemically reactive with other materials resulting in special manufacturing processes to be developed, which increases the cost of the metal [18]. Nevertheless, it is as strong as steel but 45% lighter and 60% heavier than aluminium but twice as strong [19]. Currently only fine wires of titanium and its alloys are available.
2.3 Electrical Properties

With enormous annual growth in the defence, electrical and electronic industries and with electrical equipment being produced, there is an ever increasing need to provide shielding from external electromagnetic radiation, radio frequency interference (RFI) and lightning strike while preventing any emission from known sources. The best electromagnetic interference (EMI) shielders are ferrous but most metals will provide an adequate shield. However, polymers are transparent to such radiation yet are still used to house electrical components due to their low weight, cost and electrical conductivity. Therefore, in recent years, more attention is being paid to produce a polymer-based composite that incorporates the EMI shielding benefits of metals for such applications. These conductive polymer composite materials exhibit desirable characteristics with regards to electrostatic discharge, EMI, RFI, thermal expansion, weight, impact and fatigue properties, and chemical and corrosion properties [20, 12].

Four ways are established in making polymers conductive. LeBlanc and Reinhard [21] amongst others have described surface treatment approaches such as plating, spraying and painting. However, some of these processes are expensive, can easily be rendered ineffective through any kind of damage to the surface, and are hence limited to interior surfaces. Conductive carbon black is a filler material that has good EMI shielding capabilities but is difficult to work with, has an undesirable effect on the mechanical properties of the plastic and the moulded part is destined to be black [20], although carbon nanotubes are overcoming these problems [22]. Metal-coated glass fibres have also been suggested for integration into the polymer matrix but have accompanying processing difficulties [23]. Finally, metallic fillers may be used in a polymer matrix and this has been the subject of interest for much work.

2.3.1 Metal fibres for electrical conduction

Metal fibres and particles have been investigated in numerous studies for the purpose of EM shielding. It has been found that metallic fillers with higher aspect ratios result in better conductivity as the distance an electron must travel before reaching the next fibre is reduced [20]. Hence, contrary to using particles, fibres need not be present in high volume fractions for the same amount of EMI shielding to be produced [24]. Requiring such a low concentration of these fibres has many other advantages such as minimal alteration of base resin properties and shrinkage similar to unfilled resins [25]. Fibre orientation is also important in shielding effectiveness. Fibres arranged predominantly in one direction will only shield strongly against one radiation polarity and less against radiation of opposite polarity, leading to reduced shielding. Uniform dispersion ensures that there is no penetrable area of the overall structure.

A number of composites have been investigated all with the aim of improving the electromagnetic shielding effectiveness (EMSE) of the material. Bigg’s [20] work included both thermoset and thermoplastic polymer resins used with varying lengths of short aluminium fibres. Typically for this type of application however, stainless steel fibres have been used with the conclusion that an increased presence of metal fibres produces an increased degree of shielding [12, 13, 26, 27, 28]. Increasing the concentration of the fibres increases the probability of fibre contact within the composite, forming a conductive network and hence increasing its shielding effectiveness. Yet, at low fibre concentrations, EMSE is satisfactory over a large frequency range [29]. As Table 2.1 shows, steel exhibits high absorption and low reflection of EM energy resulting in low interference and high deflection of EM waves. Not shown is that steel also has a high resistance to corrosion and oxidation and does not increase in resistivity with time, nor does surface damage to the fibres decrease the shielding effectiveness. For
Chapter 2. Literature review

<table>
<thead>
<tr>
<th>Materials</th>
<th>Conductivity</th>
<th>Permeability</th>
<th>Absorption loss*</th>
<th>Reflection loss+</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silver</td>
<td>1.05</td>
<td>1</td>
<td>1.05</td>
<td>1.05</td>
</tr>
<tr>
<td>Copper (annealed)</td>
<td>1.00</td>
<td>1</td>
<td>1.00</td>
<td>1.00</td>
</tr>
<tr>
<td>Aluminium</td>
<td>0.61</td>
<td>1</td>
<td>0.61</td>
<td>0.61</td>
</tr>
<tr>
<td>Steel (SAE 430)</td>
<td>0.02</td>
<td>500</td>
<td>10</td>
<td>$4 \times 10^{-5}$</td>
</tr>
<tr>
<td>Nickel</td>
<td>0.20</td>
<td>100</td>
<td>20</td>
<td>$2 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

* taken as the product of conductivity and permeability of the metal
+ taken as the ratio of conductivity to permeability of the metal

Table 2.1 Electrical properties of some shielding materials relative to copper [12]

these reasons, stainless steel is the metal of choice for EMI applications.

Another area in which metal fibres are particularly useful is in lightening strike applications. Nearby strikes generate a strong EM field while target strikes create pulsed current flow within the structure. These current flows induce voltage pulses that affect nearby circuits or components and are of much concern to both structural and electrical engineers [30]. Already various applications use stainless steel and copper fibres that are woven together with graphite in an attempt to protect against lightning [25] although in aircraft, materials such as metal mesh and strips are generally used to direct the lightning away from vulnerable areas.

2.3.2 Knitted metal fabric for EM shielding

In order to improve the EMSE, research into knitted-fabric-reinforced composite laminates has been undertaken. By incorporating metal into the knitting yarn itself, a higher degree of metal wires may be present thus improving both mechanical properties and EMSE. This is the impetus behind the work of Cheng et al. [12]. Glass fibres were knitted together with stainless steel fibres and impregnated with a polypropylene matrix. It was found that the EMSE of this composite was affected by the concentration of the metal fibre, the thickness, or number of plies and the fabric knit structure. The added benefit of using such knitted composites over simple fibres was that impact resistance was also markedly increased without any loss of conductivity and hence EMSE [12].

2.4 Mechanical Properties

In the fabric industry, metal fibres have been used to improve the durability of clothing and reduce wear of carpets [31]. On an industrial level, compared to other fillers, metal fibres impart lower wear rates at high loading levels on moulds and machine parts, for example [25, 32]. This section highlights past work that has been done of the effect of various metal fibres on mechanical properties.

2.4.1 Physical ageing and creep

It is well known that although polymer composites display creep properties, they recover partially or completely when the applied stress is removed. Such viscoelastic deformation of polymer composites is dependent upon stress, temperature and physical ageing time. Fibre-reinforced plastics are used in primary and secondary structural applications on aircraft and so high durability and reliability over long-term periods with regards to deformation is important [33]. During loading, two processes occur:
creep deformation and progression of physical ageing. Physical ageing is a time-dependant characteristic of polymers in the amorphous glassy state. Products of glassy polymers are usually shaped when they are in the molten state and are fixed into shape through rapid cooling to a temperature below the glass transition temperature. However, this period is too short for full solidification and stiffening of the polymer to take place. The process continues during the service life of the finished product and mechanical properties change with time, such as increased stiffness. Most importantly, physical ageing causes problems with long- and short-term creep behaviour. Hence, the rate of cooling plays a role in determining the properties of the plastic. The addition of reinforcing fibres changes and helps to reduce these effects.

In order to observe whether metal fibres behave differently to other composite fibres, Biswas et al. [34] studied the effect on stainless steel fibres arranged randomly within poly-phenylene ether (PPE). With an increase in the fibre volume fraction, suppression of viscoelasticity occurs and the composite is able to withstand higher temperatures and last a longer time before significant changes in deformation take place [33]. In addition, Biswas and Somiya [35] had found that by increasing the physical ageing time of the polymer, the progression of physical ageing changes the resin matrix towards a more stable state and this phenomena is still seen with the addition of steel fibres. An increase in isothermal physical ageing treatment with time can decrease the amount of creep deformation, which in turn is arrested by physical ageing and are able to stave off deformation for longer periods [34, 35, 36]. In this way, the metal fibres behave in the same way and with the same predictability as other fibre reinforcements.

### 2.4.2 Strength

Reinforcement of any material with metal fibres potentially serves two purposes. The first is to strengthen a material against static and dynamic loads while the second is to strengthen against impact. In a study where titanium fibres were introduced into a glass ceramic matrix, the strength was found to increase linearly with increasing fibre volume fraction, and a composite composed of 20% titanium fibres sustained stresses five times higher than the fracture stress of the matrix alone [37]. In addition, Hoffman et al. [38] investigated and modelled the stress-extension behaviour of ductile aluminium fibres embedded in alumina matrix and found that the high constraint provided by the matrix raises peak stresses in the fibres. Higher strength resulted from this constraint with little detriment to the toughness.

### 2.4.3 Fracture Toughness

Much work on metal fibres regarding fracture toughness has focused on ceramic matrix materials [39, 40, 41, 42] and it has been found that the best results can be reached by using continuous reinforcements [43]. The major mechanism of improving this toughness is through impeding fracture with the use of intact fibres. These fibres bridge the cracks thus delaying failure of the material. Figure 2.7 shows the crack path of a brittle composite in tension. Cracking through the matrix is favoured as opposed to along the fibre/matrix interface and ductile fibres are able to bridge the cracks and avoid early catastrophic failure. Most theories suggest that the ductile phase, acting as a crack bridger, deflects energy built up within the brittle matrix and releasing it through plastic deformation. However, with a very restrictive and rigid matrix, this ductile deformation is inhibited by a very strong fibre/matrix interface.

Ceramic foam cores have recently been a topic of much interest due to their thermal and acoustical insulating properties. Microwave radiation is used to manufacture these foams but the porosity of the
foam has been found to be uncontrollable and fracture properties as a result worsen [44]. Minay et al. [45] have investigated the addition of metal fibres as reinforcements and have found that the ductility and crack bridging resulting from the fibres not only improved fracture toughness, but also were able to produce even distribution and sizing of the pores during microwave radiation.

In general with brittle matrix/brittle fibre composites, a weak interfacial strength is required which allows frictional sliding of the fibres against the matrix following failure at the interface [41, 46]. The work done at the interface increases and fracture toughness is improved. Conversely, strong interfacial bonding is necessary in the case of ductile metal fibres. The increase in work done in this case is due to the plastic deformation of the bridging fibres as the distance between the crack surfaces enlarges.

Heating pre-strained titanium-nickel alloy, also known as a shape memory alloy (SMA), in the form of fibres in an epoxy matrix causes a change of phase of the alloy resulting in a compressive stress within the fibre. Shrinkage of the fibres decreases the stress concentration around any crack tip present in the vicinity of the fibre thus reducing the stress intensity factors and improving fracture resistance [47]. The use of SMAs as crack bridgers have been investigated further and modelled in [29, 48, 49]. Along similar lines, high shrinkage of the matrix material is able to grip the reinforcing fibres tighter thereby increasing the energy needed to open a crack, also resulting in increased toughness [50].

Improved fracture toughness is not restricted to such brittle composites. Stainless steel fibre/plastic matrix systems display improved fracture toughness, provided that the relatively hydrophobic steel has been surface treated, or that interfacial reactive agents are added [27].

2.5 Acoustic Behaviour

Sound absorbing materials are necessary in order to avoid the release of disruptive noise. Stainless steel has been investigated by Unix Co. Ltd and have found that sintering at high temperatures produces three dimensional interconnecting pores which are highly effective at dissipating sound energy. The acoustical behaviour of sintered and non-sintered stainless steel fibre mats were also investigated by Albracht and Lotze [51]. Highly porous metal fibre structures were used to investigate whether disruptive noise could be successfully filtered whilst being structurally sound in terms of strength and toughness. In contrast to those findings from Unix, it was found that sintering the fibres reduces the surface roughness and resulted in a loss of absorbency. Non-sintered bundle-drawn fibres were more effective as lightweight sound absorbers. Although the use of metal fibres as sound absorbers have not been investigated in the context of composite materials, as stand-alone structures, they have shown to be extremely effective.
2.6 Concluding comments

A number of metals are available in continuous fine wire and fibre-bundle forms, yet it has been shown that continuous metal fibres introduced as a third component in composite systems have not been thoroughly examined. Based on results from the literature, further examination of processes such as lightning strike protection and electromagnetic shielding is recommended. The potential for improvements in various mechanical properties remains virtually unknown, and therefore is of interest for further investigation.

As yet, property characterisation of metal fibres has not been performed, even with the vast range of metals available. Instead, it has been common practice to describe the properties in terms of the sheet metal. A comparison is presented here of the metals mentioned in this chapter.

<table>
<thead>
<tr>
<th>Metal</th>
<th>Density (g/cm³)</th>
<th>Tensile strength (MPa)</th>
<th>Elongation at break (%)</th>
<th>Resistivity (x10⁻⁶ Ω·cm)</th>
<th>CTE (µm/[m·°C])</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium (7000)</td>
<td>2.8</td>
<td>76</td>
<td>50</td>
<td>2.7</td>
<td>25.5</td>
</tr>
<tr>
<td>Copper</td>
<td>9.0</td>
<td>221-455</td>
<td>55</td>
<td>1.7</td>
<td>17.7</td>
</tr>
<tr>
<td>Nickel</td>
<td>8.9</td>
<td>317</td>
<td>30</td>
<td>6.4</td>
<td>13</td>
</tr>
<tr>
<td>Stainless Steel:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>304</td>
<td>8.0</td>
<td>505-840</td>
<td>&gt;40</td>
<td>72</td>
<td>17.8</td>
</tr>
<tr>
<td>316</td>
<td>8.0</td>
<td>460-860</td>
<td>&gt;60</td>
<td>70-78</td>
<td>17.8</td>
</tr>
<tr>
<td>Tantalum</td>
<td>16.7</td>
<td>310</td>
<td>-</td>
<td>13</td>
<td>7</td>
</tr>
<tr>
<td>Titanium</td>
<td>4.5</td>
<td>241-552</td>
<td>15-25</td>
<td>60</td>
<td>9.2</td>
</tr>
<tr>
<td>Tungsten</td>
<td>19.3</td>
<td>530-1920</td>
<td>8</td>
<td>5.4</td>
<td>4.5</td>
</tr>
</tbody>
</table>

Table 2.2 A summary of various metal properties [52, 53, 54, 55].

Figure 2.8 shows the sheet properties of a selection of these metals compared to carbon, glass and aramid fibres. The stiffness and ductility properties of stainless steels offer some of the most interesting opportunities for use in composite materials, and in fact will be used throughout this research. The major drawback arises when the density is considered and using high volume fractions of metal fibres in composites becomes futile. Therefore metal fibres can only be used for very specific applications, where the high strain to failure would be advantageous, for example. In addition, metal fibres could find a place in applications that also take advantage of their electrical and thermal properties, adding functionality to the composite.

Bibliography


Figure 2.8 Comparison of various sheet metals and fibres.


B.C. Gabrielson. An introduction to the emp and lightning effect.


Chapter 3

Properties of metal wires, fibres and meshes

3.1 Introduction

Metal fibres have been used in previous research and are currently being used in various applications. However, material mechanical property data is not readily available and is obviously very important for finding areas of potential application. In addition, while the mechanical behaviour of metal sheets, bars and rods under tensile loads, for example, are well-known in research, the behaviour of metal fine wires, fibre bundles and meshes are only generally known by the manufacturer.

A variety of manufacturers produce fine metal wires, fibres and meshes in a variety of metals but all three forms can be conveniently found in stainless steel. For every manufacturer, the method of production varies according to the desired properties and cost of the final product. Hence it cannot be assumed the same properties will be found for the same product that has been obtained from different manufacturers. Therefore, this chapter investigates the tensile behaviour of metal fibres, fine wires and meshes.

3.2 Method

3.2.1 Tensile tests of metal wires

Fine metal wires ranging between 30-100µm diameter and of AISI304 type were obtained from two different companies, Companies A and B. The wires were all annealed, the precise procedure of which is confidential information of the supplier.

Roller clamps were used to grip the fine wires as shown in Figure 3.1(a) and were placed in a 1 Ton testing machine equipped with a 1kN load cell. A number of gauge lengths were pretested from 50-100mm, with no difference in results found. The gauge length was therefore set to 50mm allowing for more space for high wire strains. The wires were tested at a constant rate of extension of 100mm/min, and were only accepted if failure did not occur in the grips. As the wire diameters were small, strains in the elastic region of deformation could not be measured accurately and hence the stiffness was not evaluated.
3.2.2 Tensile tests of metal fibre bundles

End-drawn (as-produced) and annealed fibre bundles of AISI316 type were obtained in diameters ranging from 8-30\(\mu\)m. The end-drawn fibres were drawn such that, according to the manufacturer, the maximum fibre strength could be obtained. Annealed versions of these fibres were performed directly from the draw at 1050\(^\circ\)C in a continuous furnace followed by rapid cooling. All fibre bundles consisted of 1000 filaments.

Radiused clamps of diameter 25mm were used to grip the bundles and set 250mm apart in a 25 Ton tensile testing machine. Figure 3.1(a) shows one of the clamps. Extensometers were used to measure the displacement of the bundles under loading and the grips were equipped with rubber bands so as not to cause damage to the specimens, Figure 3.1(b). The gauge length was set at 100mm. A pretension was applied manually to straighten the bundle. Metal fibre bundles were tensile tested according to ISO 3341, at a constant rate of extension of 200mm/min.

In order to calculate the tensile properties of the fibre bundles, it was assumed that the fibre statistics could be described using the two function Weibull distribution:

\[
F(\sigma) = 1 - \exp\left[ -L \left( \frac{\sigma}{\sigma_o} \right)^m \right]
\]

where \(F(\sigma)\) is the cumulative probability of failure of a fibre subjected to a load no greater than the equivalent strength, \(\sigma\), \(L\) is the gauge length and \(m\) and \(\sigma_o\) are the Weibull shape and scale parameters, respectively. Assuming random failure of the fibres and that the applied load is uniformly distributed amongst the remaining intact fibres, it has been shown that the load, \(P\), on a bundle may be given as [1]:

\[
P = AN_oE\sigma_o\epsilon o\exp\left[ \left( -\frac{\epsilon}{\epsilon_o} \right)^m \right]
\]

where \(A\) is the average cross-sectional area of the fibres, \(N_o\) is the initial number of fibres, \(E\) and \(\epsilon\) are the bundle modulus and strain respectively, and \(\epsilon_o = \sigma_o/E\). Differentiating Equation 3.2 with respect to \(\epsilon\) and rearranging for \(E\), the following is obtained [1]:
3.2 Method

<table>
<thead>
<tr>
<th>Mesh name</th>
<th>Wire diameter (mm)</th>
<th>Mesh opening (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M40</td>
<td>0.140</td>
<td>0.495</td>
</tr>
<tr>
<td>M54</td>
<td>0.112</td>
<td>0.495</td>
</tr>
<tr>
<td>M70</td>
<td>0.112</td>
<td>0.250</td>
</tr>
<tr>
<td>M320</td>
<td>0.035</td>
<td>0.050</td>
</tr>
</tbody>
</table>

Table 3.1 Mesh dimensions.

Figure 3.2 Geometry of the mesh for tensile testing.

\[(3.3)\]

\[E = \frac{(dP/d\epsilon)_{\epsilon=0}}{AN_o}\]

Therefore the bundle modulus may be calculated using the initial slope of the \(dP/d\epsilon\) curve. Finally, it follows from Equation 3.3 that the bundle strength, \(\sigma_b\) may be given as:

\[(3.4)\]

\[\sigma_b = \frac{P_m}{AN_o}\]

It should be noted that there was no exact value for the fibre diameter due to their irregular cross-sections. Therefore the manufacturer’s measurements were taken as the average value for the calculations.

3.2.3 Tensile tests of metal meshes

Stainless steel meshes of annealed AISI304 type were obtained of various wire diameters and openings, as detailed in Table 3.1. Rectangular tensile specimens were cut from the meshes using scissors, ensuring that the number of longitudinal fibres were the same for each specimen from the same mesh. After cutting, the specimens were degreased in an acetone bath for 15 minutes, and then rinsed in water and cleaned with pressurised air. Aluminium tabs were prepared to ensure even gripping during the test. Scotchweld 9323 B/A (3M) was used to bond the tabs to the mesh, and left overnight to cure. Figure 3.2 describes the specimen geometry.

Extensometers were set at a gauge length of 100mm, with the grips equipped with rubber bands. The specimens were tensile tested in a 25 Ton tensile testing machine according to ASTM D3039. The test speed was set at 2mm/min until yield point, and increased 6mm/min after this point was reached. Only those samples that failed outside of the tabs were accepted. The stress values were calculated using the actual area of fibres carrying the load, that is, only considering those in the 0° direction.
Chapter 3. Properties of metal wires, fibres and meshes

<table>
<thead>
<tr>
<th>diameter (µm)</th>
<th>(\sigma_{uts}) (MPa)</th>
<th>(\epsilon_{max}) (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Company A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>672.4 (18.3)</td>
<td>0.23 (0.02)</td>
</tr>
<tr>
<td>50</td>
<td>919.6 (29.3)</td>
<td>0.31 (0.03)</td>
</tr>
<tr>
<td>70</td>
<td>750.5 (14.3)</td>
<td>0.32 (0.01)</td>
</tr>
<tr>
<td>Company B</td>
<td></td>
<td></td>
</tr>
<tr>
<td>70</td>
<td>746.9 (4.9)</td>
<td>0.36 (0.02)</td>
</tr>
<tr>
<td>100</td>
<td>752.0 (3.8)</td>
<td>0.38 (0.01)</td>
</tr>
<tr>
<td>Sheet metal</td>
<td>-</td>
<td>515</td>
</tr>
</tbody>
</table>

Table 3.2 Ultimate tensile strength and failure strain for metal wires of different diameters compared to the sheet metal. Standard deviations are given in brackets.

3.2.4 Inspection

A selection of fine wires and fibres were chosen for scanning electron microscopy (SEM) inspection. In particular, the austenitic grain sizes were of interest. The samples were wound around a small metal plate and embedded in epoxy. The samples were then carefully ground along the wire/fibre axis using 1µm grit-size paper to roughly half a diameter depth. To expose the grains, an etching process similar to that described in Section 4 was used.

3.3 Results and discussion

3.3.1 Fine wires

Figure 3.3 gives an example of tensile stress-strain graphs produced by the wires obtained from Companies A and B. It is immediately clear that all the curves follow typical stress-strain curves for stainless steel sheet metal. As with most metals, the yield point was not clearly defined but roughly lies between 0.004-0.006 strain. After the yield point, there was a drop in stress due to atomic interactions and dislocations, followed by a period of strain hardening indicated by increasing stress to the ultimate strength. Table 3.2 provides a summary of the tensile test results and also shows typical values for AISI304 sheet metal.

Three different diameters were obtained from Company A. It was expected that there would be an increase in tensile strength with increasing diameter, as the wire became less susceptible, and more resistant, to surface defects and discontinuities. However, the 50µm wire had properties superior to those of the 70µm. In contrast there were little differences in tensile properties between the two diameters obtained from Company B.

The grain size of the wires were analysed to explain the differences in the tensile performance from Company A, and SEM images are shown in Figure 3.4. The irregularity and random grain orientation is typical of annealed steel. The images show that the average grain size of the 50µm wire was smaller than of the other diameters. There were more grains per cross-sectional area that took the load during tensile testing, resulting in a stronger wire. The differing grain sizes indicate that different annealing conditions were used for these wires, highlighting the fact that there is currently no standardisation of fine wire production and adding difficulty in obtaining universal experimental data from these materials.
3.3 Results and discussion

3.3.2 Fibre bundles

Figure 3.5 gives an example of tensile load-strain graphs obtained from fibre bundles of various diameters. Figure 3.5(a) shows the load-strain curves for the end-drawn samples. The low gradient at the initial portion of each of the load-strain curves indicates settling and alignment of the fibres as the load was applied. The curves then become relatively linear up to the maximum load, followed in most cases by sudden failure. With this, the fibres failed in a cotton wool-like manner. The fibres therefore individually failed at different positions within the fibre bundle, implying that there was no stress concentration at the grips.

Figure 3.5(b) shows the load-strain curves for the annealed samples. The initial portion of the curves display the same fibre settling and alignment as with the end-drawn bundles. The curves also display a plateau region where there is a very small increase in load for a large increase in strain. This behaviour imitates that of sheet metal and may be evidence of strain-hardening. After the maximum load was reached, the bundle failed gradually, as the fibres necked and then yielded in sections within the bundle.

The effectiveness of annealing in restoring ductility to the metal at the fibre level is clearly displayed in the failure strain: the end-drawn fibres were an order of magnitude lower than those of the annealed. The bundle strength of the end-drawn fibres were on average around 40% higher. In both the end-drawn and annealed cases, the tensile properties increased with increasing fibre diameter. Figure 3.6 summarises the calculated values of bundle strength and modulus for each fibre diameter.

For both the end-drawn and annealed fibres, the modulus and strength increased with increasing fibre diameter. For the strength, the presence of surface irregularities allowed for premature yielding and failure of individual fibres. In addition, friction within the fibre bundle would have contributed to early failure.

The apparent increase in modulus can be explained through Equation 3.3. This equation assumes
that every fibre within the bundle was intact before testing and each carried an equal quantity of load. In reality, these conditions were not applied, as the bundles had visibly loose, i.e. broken, fibres as well as slight twist, which was a result of the production process. Both of these effects became less apparent with increasing fibre diameter. There was also an influence of the fibre diameter on the accuracy of measurement, as a larger diameter meant a larger bundle, resulting in a greater gripping area for the extensometers.

The grain size of the fibres were analysed to explain the increase in strength with diameter. The grains of the end-drawn fibres could not be seen as the grain boundaries were etched away during the etching procedure. However, it can be assumed that the grains were extremely elongated, giving rise to the superior strength properties. Figure 3.7 shows the SEM images of the annealed fibres. After inspection, it was found that the grain size varied between 2-10\(\mu\)m for every fibre diameter. Therefore the larger the diameter, the more grains per cross-sectional area take the load during tensile testing, resulting in a stronger fibre.

### 3.3.3 Metal meshes

Figure 3.8 gives an example of tensile stress-strain graphs for the meshes. The clear drop after the yield stress seen with the fine wires were not evident for the meshes, and this may have been due to the increased testing speed after the yield point was reached. Evident is the difference in strength and strain to failure for each of the meshes, which in most cases were similar to those of the equivalent fine wire. The summary presented in Table 3.3 also shows that the stiffnesses were lower. This was an obvious result of the crimp, or waviness, of the mesh, and was illustrated with meshes M70 and M54. The strength and stiffness was higher for mesh M54, which had twice the opening than M70 and therefore less crimp. In addition, the wire diameter also had an influence if meshes M54 and M70 are compared. Although crimp was slightly greater for M70, the larger diameter resulted in higher tensile properties, as was seen with the fine wires.
3.4 Conclusion

Stainless steel fine wires, fibres and meshes were tested in tension and the following conclusions were drawn:

- Tensile strength and strain increase with diameter. In the case of hybridising with other reinforcement fibres, the diameters of the metal fibres should be greater in order to obtain a notable increase in overall properties of the composite.

- Annealing considerably reduces the strength of the fibres as the grains of austenite produced are randomly oriented and shaped compared to those of the end-drawn fibres. The size of the grains are not dependent on the fibre diameter, but on the annealing conditions.

- If the mesh is considered as a group of individual fine wires rather than a layer, the properties are dependent on the fibre diameter and mesh opening. For the same diameter, a larger opening will result in a stronger and stiffer mesh due to lower crimp. For the same opening, the mesh with a larger diameter will be stronger and stiffer due to the superior properties of the reinforcing wires.

- The mechanical properties of the metal wires of the same type vary with producer. The differences arise mainly from differing chemical composition of the metal, quality of manufacture and differing production processes.

As a final note, it was shown that for lightweight applications, the mechanical advantages of metal fibres in composites decrease when density is considered. In these cases, if the stiffness and ductility properties are desirable, the metal fibres should be hybridised in low volume fractions with other reinforcing fibres to keep the weight increase to a minimum.

### Table 3.3

Ultimate tensile strength, Young’s modulus and failure strain for metal meshes.

<table>
<thead>
<tr>
<th>Mesh name</th>
<th>( \sigma_{uts} \ (\text{MPa}) )</th>
<th>E (GPa)</th>
<th>( \epsilon_{\text{max}} \ (\text{mm/mm}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>M40</td>
<td>819.0 (10.7)</td>
<td>79.1 (7.02)</td>
<td>0.229 (0.014)</td>
</tr>
<tr>
<td>M54</td>
<td>799.5 (95.4)</td>
<td>70.1 (3.4)</td>
<td>0.361 (0.028)</td>
</tr>
<tr>
<td>M70</td>
<td>725.0 (22.1)</td>
<td>66.8 (7.4)</td>
<td>0.195 (0.037)</td>
</tr>
<tr>
<td>M320</td>
<td>723.7 (41.7)</td>
<td>57.5 (4.8)</td>
<td>0.010 (0.024)</td>
</tr>
</tbody>
</table>

3.3.4 Further comments

It was shown in Chapter 2 that the use of stainless steel may have great advantages in fibre form due to its excellent mechanical properties. From the results of this chapter, Figure 3.9 shows where the metal fibres may be placed in comparison to carbon and glass fibres, and the metal sheet. The envelope of properties for the metal fibres is smaller the sheet metal, but there are clear advantages in terms of stiffness and ductility over those of glass fibres, and ductility over that of carbon fibres. In lightweight applications, these advantages would be lost when density is considered, Figure 3.9(c), already highlighting a major limitation of stainless steel metal fibres. Hence it can already be concluded that in cases where weight is critical, and depending on the application, metal fibres should be hybridised in low volume fractions with other reinforcing fibres to gain the benefits of the metal properties.
Chapter 3. Properties of metal wires, fibres and meshes

Bibliography

Figure 3.5 Typical load-strain curves for fibre bundles of various diameter.
Chapter 3. Properties of metal wires, fibres and meshes

Figure 3.6  Variation of bundle strength and stiffness with fibre diameter.

Figure 3.7  SEM inspection of grain size of the annealed fibres.
Figure 3.8  Typical stress-strain curves for meshes of various diameters and openings.
Chapter 3. Properties of metal wires, fibres and meshes

Figure 3.9  A comparison of the tensile properties of stainless steel fibres to other materials.
Chapter 4

Improving adhesion between stainless steel and thermoplastics

4.1 Introduction

The mechanical performance of a composite is mainly due to the stress transfer between the fibre/polymer interface, and therefore adhesion issues of the metal fibres are very important. Different metals and matrix materials have different adhesive properties and in many cases additives must be included into the composite system to enhance adhesion. In order to explore the mechanical capabilities of metal fibre hybrid thermoplastic composites, the issue of adhesion must first be addressed. In this adhesion study, AISI316 stainless steel sheet plates were used instead of fibres as a convenient way to provide an indication of the impact of the surface treatment. Later, the most promising treatment methods can then be applied to the fibres.

A number of surface treatments for stainless steel have previously been studied consisting of the following treatments: solvent and alkaline cleaning, mechanical roughening, acid treatments and anodising. The effectiveness of the treatments have most commonly been assessed through surface characterisation methods such as X-ray photoelectron spectroscopy [1], scanning electron microscopy [2] and water contact angle methods [1, 3]. However, it has been less common to study the effectiveness of these treatments through mechanical tests. This is especially important if the two surfaces to be joined are not readily compatible and hence it is necessary to focus on the metal/polymer interface rather than simply the metal surface. The thermoplastic polymers used in this study are not adhesives and are therefore equally of interest.

This Chapter describes efforts to improve the adhesion properties of AISI 316 stainless steel to two aerospace-grade thermoplastics that are used extensively in this thesis, polyetherimide (PEI) and polyphenylene sulphide (PPS). A number of surface treatments were applied to the stainless steel substrates and lap shear tests were used to help assess the effectiveness of the treatments.

4.2 Experimental

Stainless steel sheet metal was used rather than the fibres themselves to provide an experimentally straightforward method to observe an increase in adhesion. It should be noted that other methods such as the single fibre fragmentation and the short beam shear tests were also used, but data scatter for the former and insensitivity of the latter rendered these tests unpractical. While adhesion tests were performed on sheet material, it was assumed that the results would translate well to the fibres,
Chapter 4. Adhesion

4.2 Materials

AISI 316 stainless steel (SS) sheets, of dimension 120mm x 25mm x 1.5mm, were used as the substrates for surface treatments. Before all surface treatments were applied, the samples were first cleaned in an alkaline bath at 65°C for 1 hour before being rinsed in deionised water and gently dried in an oven at 60°C. PEI and PPS foils of 60µm and 80µm thickness respectively, were used as the bonding polymers. Before use, the foils were first dried in a vacuum oven for at least 24 hours.

4.2.1 Materials

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4.2.2 Surface treatments

The stainless steel samples were first cut to the necessary dimensions before being cleaned and surface treated. The treatments used in the preparation of the samples are summarised in Table 4.1. All chemicals were diluted in deionised water unless otherwise specified. After all chemical treatments, the samples were rinsed in deionised water to remove any remaining weakly bonded surface contaminants.

Two silane treatments were used in the investigation and were chosen for the best compatibility with both the matrix and the metal according to discussions with the manufacturers and literature [4]. The stainless steel samples to were to be bonded with PEI were treated with γ-glycidoxypropyltrimethoxysilane (GPS) and those with PPS treated with bis-[m-(2-triethoxysilyl-ethyl)tolyl]polysulphide (Tolyl). Both silanes were prepared according the the manufacturers’ specifications. Figure 4.1 show the chemical composition of the silanes.

![Silane chemical structures.](image)

Figure 4.1 Silane chemical structures.
<table>
<thead>
<tr>
<th>Name</th>
<th>Treatment</th>
<th>Concentration (% volume)</th>
<th>Time (min)</th>
<th>Temperature (°C)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cleaned</td>
<td>Alkaline solution</td>
<td>-</td>
<td>60</td>
<td>65</td>
<td>final clean with acetone</td>
</tr>
<tr>
<td>Plasma</td>
<td>Open air plasma</td>
<td>-</td>
<td>0.5</td>
<td>-</td>
<td>plasma gun 10mm from surface</td>
</tr>
<tr>
<td>Grit-blast</td>
<td>Aluminium oxide</td>
<td>-</td>
<td>0.5</td>
<td>-</td>
<td>Nozzle 30cm from samples</td>
</tr>
</tbody>
</table>

### Chemical treatments

<table>
<thead>
<tr>
<th>Name</th>
<th>Treatment</th>
<th>Concentration (% volume)</th>
<th>Time (min)</th>
<th>Temperature (°C)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Passivated</td>
<td>Nitric acid</td>
<td>42</td>
<td>30</td>
<td>room temp</td>
<td>ASTM A967</td>
</tr>
<tr>
<td>Anodised-30</td>
<td>Nitric acid</td>
<td>30</td>
<td>50</td>
<td>50</td>
<td>0.5Adm$^{-2}$ SS cathode</td>
</tr>
<tr>
<td>Anodised-42</td>
<td>Nitric acid</td>
<td>42</td>
<td>50</td>
<td>50</td>
<td>0.5Adm$^{-2}$ SS cathode</td>
</tr>
<tr>
<td>Anodised-55</td>
<td>Nitric acid</td>
<td>55</td>
<td>50</td>
<td>50</td>
<td>0.5Adm$^{-2}$ SS cathode</td>
</tr>
<tr>
<td>Etched</td>
<td>Nitric acid</td>
<td>20</td>
<td>10</td>
<td>65</td>
<td>Treatment A1 of Hydrofluoric acid</td>
</tr>
<tr>
<td>Etch-anodised</td>
<td>Nitric acid</td>
<td>20</td>
<td>10</td>
<td>65</td>
<td>etch as above followed by anodising</td>
</tr>
<tr>
<td></td>
<td>Hydrofluoric acid</td>
<td>9.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Nitric acid</td>
<td>20</td>
<td>50</td>
<td>50</td>
<td>0.5Adm$^{-2}$ with a SS cathode</td>
</tr>
<tr>
<td>GPS</td>
<td>silane</td>
<td>0.4</td>
<td>dipped</td>
<td>-</td>
<td>Dow Corning Z-6040</td>
</tr>
<tr>
<td>Tolyl</td>
<td>silane</td>
<td>0.4</td>
<td>dipped</td>
<td>-</td>
<td>Gelest SIB1820.5</td>
</tr>
<tr>
<td>Anodised-tolyl</td>
<td>Nitric acid</td>
<td>55</td>
<td>50</td>
<td>50</td>
<td>0.5Adm$^{-2}$ SS cathode</td>
</tr>
<tr>
<td></td>
<td>silane</td>
<td>0.4</td>
<td>dipped</td>
<td>-</td>
<td>Gelest SIB1820.5</td>
</tr>
</tbody>
</table>

**Table 4.1** Overview of treatment procedures for the stainless steel (SS) samples
Chapter 4. Adhesion

4.2.3 Test method

After treatment, the lap shear samples were prepared with an overlap of 13mm. Three PEI foils or two PPS foils of 13mm width were placed at the bond line to ensure enough polymer was present for bonding. A schematic of this lay-up is shown in Figure 4.2. The samples were then consolidated in a hot plate press at either 320°C for PEI or 310°C for PPS. The pressure applied was kept as low as possible up until the consolidation temperature was reached to allow the polymer to spread over the surface of the bond line. The pressure was then increased to 0.1MPa for 60 secs before cooling commenced to room temperature. Samples were mechanically tested according to ASTM D1002 to obtain the apparent lap shear strength.

4.2.4 Surface inspection

Scanning electron microscopy (SEM) was used to gain greater visualisation of the surface topography and morphology to help understand the nature of the bonding and failure. Static water contact angles of various samples were also measured to monitor any changes at the surface using a Ramé-Hart Contact 100 goniometer. At least 10 measurements were taken per sample 10 minutes after surface treatment was completed, unless otherwise stated, to give a consistent time after treatment.

4.3 Results

All procedures outlined in Section 4.2.2 were first performed on the lap shear samples bonded with PEI foils. As a wide range of anodising and etching treatments were performed, it was wise to first narrow the number of chemical treatments to those that yielded the highest lap shear strength. This was done to conveniently reduce the testing matrix and hence reduce the number of samples to be tested. It was assumed that the chemical treatments that resulted in a weaker bond strength with PEI would show the same trend with PPS as the mechanism of adhesion in these cases was expected to be the same.
4.3 Results

Sample Conditions Contact angle
--- | --- | ---
As-received & - & 86° (3)
Cleaned & alkaline bath & 30° (4)
Plasma & 30s of exposure & 7° (3)
GPS & after curing & 40° (10)
Tolyl & after curing & 83° (8)

Table 4.2 Water contact angles of various SS samples. Standard deviations are given in brackets.

4.3.1 Surface analysis

Water contact angles were taken of the as-received, Cleaned, Plasma and silane-treated samples, and are listed in Table 4.2. It should be noted that a change in contact angle of a treated sample compared to that of the as-received indicates a change in the surface properties of the sample surface with respect to water, and does not necessarily indicate a change in bonding conditions for the polymer. However, it is useful to verify whether the treatment has been applied to the surface.

The hydrophobicity of the surface was shown with the high water contact angle of the as-received specimen. After cleaning the samples in an alkaline bath, the contact angle decreased significantly indicating that a considerable amount of contaminants were effectively removed from the surface. Plasma treatment further reduced the contact angle such that the surface was almost completely wetted, and was in keeping with previous results [5]. An increase in contact angle after cleaning was seen with the GPS and Tolyl treatments, indicating that silanes were bonded to the surface of the metal. The standard deviations show that the surface was not uniformly treated, which may have been due to the sensitivity of the silane application procedure.

SEM images were initially taken of the SS surfaces directly after treatment and the six most interesting are shown in Figure 4.3. Images were taken at an angle to the sample surface to display the differences in topography more clearly. Figure 4.3(a) shows the surface of the Cleaned sample, which remained smooth after cleaning. A similar surface remained after the plasma treatment. Grit-blasting, Figure 4.3(b), by contrast, was superficially aggressive, displaying a random rough flake-like topography, located in the very surface regions of the metal. The etching treatment, Figure 4.3(c), produced a very fine roughened surface with slight exposure of the grain boundaries and a porous-like appearance. After adding an anodising step, Figure 4.3(d), the treatment became much more aggressive, grain boundaries were very exposed and a porous surface was also produced within the grains. The Anodised-30 sample, had a porous surface with slight grain boundary exposure and was similar in appearance as the etched sample. Passivation produced a rough porous sample without grain boundaries exposure, Figure 4.3(e). Using the same concentration of the acid for the anodising procedure produced a very aggressive treatment that deeply corroded the grain boundaries and left a surface that was similar to the Etched-anodised sample, Figure 4.3(f).

The difference between the Anodised-42 and the Anodised-55 samples were made the most clear when the images were taken perpendicular to the surface, as shown in Figures 4.4(a) and (b). The most aggressive treatment performed was that of the Anodised-55, which exposed more grain boundaries, corroded deeper into the material, at roughly 2µm depth, and produced a more porous surface, Figure 4.4(c).
4.3.2 Mechanical testing

PEI bonded joints

Considering the non-chemical treatments first, Figure 4.5 shows a graph of the lap shear strengths with their associated standard deviations of the samples treated as described in Table 4.1. The baseline Cleaned samples failed at 10.4MPa and all other treatments can be compared to this value. The highest lap shear strength was obtained with grit-blasting compared to air-plasma giving the first indication of the effectiveness of mechanical interlocking between PEI and the SS. The reproducibility of the grit-blasted specimens was also very high as shown by the small scatter, and this may have been due to the aggressive nature of the treatment, which will be discussed in more detail later.

Figure 4.6 shows the lap shear strengths of the samples that were chemically treated. Although the measured water contact angles on the GPS specimens indicated the presence of silane, the lap
shear strength decreased to below that of a specimen that was simply cleaned. The first aggressive chemical treatment applied to the SS was a passivation method, which resulted in a 68% increase in shear strength. The Anodised-30 samples increased the shear strength to a value close to that of the Plasma. Of the anodised samples, the highest strengths were obtained with an anodising solution concentration of 55% resulting in a 2.5-fold increase over the baseline Cleaned samples. A similar result was obtained with the Etch-anodised samples, which show that a two-step surface treatment procedure was unnecessary in this case. In comparison, the lower lap shear strength resulting from simply etching shows that there is a considerable contribution that is obtained from the anodising step.

Examples of the failure surface of a few samples are shown in Figure 4.7. It is clear from Figure 4.7(a) that the Cleaned samples displayed adhesive failure, leaving one surface almost completely stripped of the PEI polymer. Similar failure was found for the Plasma samples. From the visual inspection, it would be expected that there was very little adhesion between the SS and the PEI but the results of the lap shear tests show otherwise. Even only after cleaning, there was a relatively substantial amount of interfacial shear strength, and hence adhesion, between the metal and polymer interfaces. Figures 4.7(b) and (c) show typical surfaces for the grit-blasted and anodised surfaces respectively. There appeared to be very little differences in the failure mode and adhesion was strong enough for deformation of the specimens to first occur, Figure 4.7(d).

**PPS bonded joints**

The results of the lap shear tests of the PEI bonded joints showed that of the aggressive treatments, anodising produced some of the strongest bonds, and therefore only this procedure was investigated in addition to the silane treatment. Figure 4.8 shows the experimental results for the PPS lap joints.
Chapter 4. Adhesion

The natural adhesion of PPS to stainless steel was very low compared to PEI, which was expected due to the non-polarity of PPS. Again although the measured water contact angles on the Tolyl specimens indicated the presence of silane, the lap shear strength on average did not change compared to the Cleaned specimens. Anodising produced a 5-fold increase in strength, which decreased slightly when coupled with the Tolyl treatment.

Examples of the failure surface of a few samples are shown in Figure 4.9. Figure 4.9(a) shows that the Cleaned samples displayed very poor adhesive properties such that the polymer was easily released from the coupon surface after testing. In contrast, Figures 4.9(b) shows a typical anodised surface with the polymer firmly attached to both coupons. As with the PEI specimens, deformation of the coupons to first occurred before failure, Figure 4.9(d).

4.4 Discussion

Stainless steel coupons were surface treated and bonded together using either PEI or PPS and tensile tested. From the results, it was seen that the natural adhesion of PEI was far greater than that of PPS. This was due to its more polar nature. The effectiveness of each surface treatment varied greatly and is discussed in more detail in this section.

The potential of the air-plasma as a surface treatment lies mainly in its ability to effectively clean the surface of the stainless steel from organic contaminants [6]. The wettability of the surface increased greatly but did not significantly improve the lap shear strength. Hence it was found that there was no relationship between wettability and compatibility with the matrix.

It has been shown that typical austenitic stainless steels have a surface oxide layer that when properly cleaned, is highly polar and can readily form hydrogen bonds [6]. Silanes with methoxy- and ethoxy-based hydrolysable groups have been shown to bond well with stainless steel surfaces [7, 8, 9, 10]. It has been suggested [11] that silane bond formation with thermoplastics mainly occurs through interpenetrating network formation in the interphase region, where the functional groups Y of the silane diffuses into the polymer, as shown in Figure 4.10. For this to occur, there must be good compatibility between the polymer and the functional group Y of the silane [11].

Previous research focusing on the adhesion of stainless steel to epoxies using silanes had shown significant improvements [10, 12]. It was clear from these results that under the correct conditions,
Figure 4.6  Lap shear results of the chemical treatments with PEI matrix.

(a) Cleaned  
(b) Grit-blast  
(c) Anodised-55  
(d) Deformation in the Anodised-55 coupon

Figure 4.7  Typical failure surfaces of cleaned and treated PEI specimens.

silanes could offer a viable alternative to chemically aggressive treatments such as etching for stainless steel. However, no improvements were found in this study with either of the silanes and were attributed to three reasons. The first and most obvious was that the chemical degradation temperature of each of
the silanes was far below that of the processing temperature of the polymers used. Thermogravimetric analysis (TGA) was employed to measure the degradation temperature of GPS- and Tolyl-treated stainless steel, the results of which are shown in Figure 4.11. The silanes could not withstand the high temperatures employed during lap-joint production, and began to degrade at around 190°C and 200°C for the GPS and Tolyl respectively. The GPS was very quickly degraded to the extent that a weakly bonded silane layer was produced, which was weaker than the natural adhesion of PEI to stainless steel. Contrary to the findings of this investigation, Nguyen and Berg [4] had found a substantial improvement in using tolyl silane to couple PPS to an aluminium substrate, which suggested that the processing temperature used, also 310°C in their case, should not have affected the silane.

The second reason was due to the low affinity of the silanes used to the stainless steel surfaces. The TGA showed that there was very little GPS bonded to the steel compared with the Tolyl, indicating that there was great difficulty in the silanating the samples. The effect of the Tolyl on the lap shear strength was minimal, which may also have been due to the low presence of the silane on the surface.
4.4 Discussion

The final reason may have been due to the lap shear test itself, which may not have been sensitive enough to highlight small differences in adhesion. Lap joints tested in tension experience peel forces as well as shear, which in this case were clearly evident with the deformation of the coupons, reducing the sensitivity of the test.

Mechanical interlocking played the largest part of any bonding mechanism between the SS and the polymer matrices. As the SS coupons deformed, mechanical interlocking provided the adhesion strength and a "gripping surface" for the polymer. The nature of the gripping surface slightly affected the shear strength as was shown with gritblasted and anodised surfaces, which produced average values of 25.4MPa and 27.5MPa, respectively for the PEI matrix. The gritblasting produced a shallow, flaky surface, with the individual flakes ranging between less than 1µm to 10µm in size, producing a very large bonding area. However, anodising exposed grains of 20µm diameter on average, with deep grain boundaries. Each grain had a porous surface, increasing further the bonding area on a much finer level, and accounting for the slightly greater lap shear strength. This was seen further with the increase of acid concentration used in the anodising process and lap shear strength. The most aggressive treatment produced the greatest bonds as a rougher surface was produced.

As a practical issue, applying these treatments to small-scale metal fibres is only possible to a certain diameter. In the case of gritblasting, this is because of the size of the grit particles, and is also more practical if the fibres are in a more rigid form, such as a woven mesh. With anodising the limiting factor is the arrangement of the fibres in the anodising bath as the process is not continuous. It was shown in Chapter 3 that the grain size does not change with decreasing diameter in the case of annealed fibres. Therefore beyond a certain diameter, anodising would weaken the integrity of the fibre thereby lowering the mechanical properties. Hence the most practical method for improving adhesion of stainless steel metal fibres to thermoplastic matrices would be to roughen the metal surface during fibre production thereby increasing the susceptibility of the polymer to bonding.

Surface roughness provides sufficient mechanical bonding for static applications but durability has not been discussed. In their investigations, Haak and Smith [1] found no correlation between surface roughness and durability and an influence between surface chemistry and durability. Improving the chemical bond between the metal and the polymer is still an important issue that needs further work.
4.5 Concluding comments

Various surface treatments on AISI 316 type stainless steel were investigated to improve adhesion to PEI and PPS. Using the lap shear test, the apparent shear strength was measured and the following conclusions were made:

- Silane treatments were rendered ineffectual due to the high processing temperatures used, which resulted in chemical degradation. The silanes used also had a very low affinity to stainless steel.

- Mechanical interlocking was the most effective bonding mechanism between the stainless steel and the polymer matrices.

- Anodising was a far more aggressive treatment than etching for the stainless stainless steel, show-
ing that typical two-step etch-anodising procedures are unnecessary for this metal.

In terms of applying the outcomes of this chapter to improving adhesion of stainless steel fibres in general, there is still much work to be performed, as clearly anodising is not a universal solution. Efforts need to be placed improving the chemical bond between the metal and the polymer, and although steps were taken in this direction, a full investigation was not possible within the time-frame of this research. However, anodising provides a good solution for the work of this thesis.

**Bibliography**


Chapter 5

Impact properties of glass-fibre reinforced epoxy with metal wires

5.1 Introduction

Aerospace composite structures are often subjected to low velocity impacts which can result in extensive damage. Even at relatively low impact energies many damage modes are possible, such as matrix crushing, cracking, delamination and fibre breakage. The point at which damage begins to occur within the composite laminate significantly reduces residual mechanical properties, and hence it important to keep the level of damage to a minimum by increasing damage resistance.

Several methods have been employed to improve the impact behaviour of fibre reinforced polymer composites, such as matrix toughening, fibre hybridising and 3-D composites, for example. This investigation takes the hybridising approach to improvement in impact properties through the incorporation of metal wires into the laminate. From the properties of the tensile tests in Chapter 3, the ductility of the metal wires is the most obvious property to be exploited. The ability of the metal wires to plastically deform could be beneficial in the event of impact, as plastic deformation is an extra energy absorption mechanism, thus reducing the energy that would otherwise cause delaminations and fibre breakage.

The idea of embedding a more ductile fibre has been investigated in the literature, such as through the addition of aramid or polyethylene fibres, with varying amounts of success. Shape memory alloy (SMA) wires hybridised with carbon fibres have also been investigated [1]. It was shown that only 2.8% volume fraction of embedded SMA wires resulted in a decrease in damage area of up to 25% compared to plain laminates. However, these wires were relatively large at 0.3mm diameter and thus constituted a considerable proportion of the thickness of the overall laminate (1.63mm) in the investigation, making it difficult to differentiate between the effect of the metal inclusion and thickness effects. The impact performance of stainless steel metal wires in epoxy has been investigated previously by Ross et al. [2] and compared to that of glass/epoxy. A difference in failure mechanism was found to exist between the two types of laminate but a direct comparison of impact behaviour in terms of absorbed energy and damage area was not made.

Previous literature has provided good indication that improvements in impact behaviour are possible with fibre hybridising. Therefore, it was the aim of this study to investigate the effect of hybridising glass fibres with metal wires on the overall laminate behaviour under low-velocity impact.
5.2 Experimental details

Glass fibre rovings of 800 tex and AISI304 stainless steel fine wires of 100µm diameter were used as the reinforcement. In addition type 316L stainless steel rovings were Epikote 4908 resin and hardener was used for the epoxy matrix.

5.2.1 Production method

Crossply laminates were filament wound over a stainless steel mould using an automated filament winding machine. Initially, three metal wires were wound together in the 0° direction at a spacing of once every 2.5mm for 160mm, and then once every 1.25mm for 160mm. The mould was then rotated 90° and the procedure repeated. The wires were held on three separate bobbins on a tensioning fixture. In this way the wires were kept slightly taut and therefore were more easily kept in place on the mould. Figure 5.1(a) shows details of the metal wire arrangement, where it is clear that nine 160x160mm sections were produced containing various amounts of wires. In one section, labelled “7” in the figure, there were no wires. It is also clear from the production process and the figure that laminates 1 and 9, 2 and 6, and 4 and 8 contained the same amount of wires and will be verified in the Results section.

Glass rovings were filament wound above the wires, as shown in Figure 5.1(b). Four layers were wound in total with a lay-up of [0/90]_2. The mould and contents were then bagged in preparation for the vacuum infusion process, Figure 5.1(c). Finally, the epoxy resin and hardener were mixed according to the manufacturer’s specifications and vacuum-infused at 20mbars until the contents were fully impregnated. The vacuum was then raised to 500mbars to reduce the size of any voids present and left to cure in this condition for at least 24 hours. A 4 hour post-cure at 80°C completed the production process. In total three laminates were produced in this way.

An electric mini-saw was used to cut the edges of the cured laminate to release two identical plates from the mould, as shown in Figure 5.1(d). Each of the plates were cut using a diamond saw into nine 120x120mm sections.

To compare the laminates, density measurements were performed according to ASTM D792. Volume fraction measurements were also performed according to the burn-off test outlined in ASTM D2584. The metal wires were extracted by hand from the dry glass fibres and weighed separately.

5.2.2 Impact test method

The drop-weight impact tower employed in this study consists of a double column impactor guide mechanism that releases an impactor at the desired height. The system was configured to eliminate friction problems because the impactor is liberated from the carriage holder and is free to fall a few centimeters before impact. The contact force between the impactor and the specimen was measured by a load cell placed in the impactor tup. Figure 5.2 shows the impact apparatus.

In previous studies, a number of methods have been employed in supporting the specimens, from simply supporting [3] to clamped using a number of torques [4, 5], where the clamping device has either been square [6] or circular [7] in geometry. Previous tests [8] performed with the equipment used in this investigation showed that a specimen clamped with a circular test geometry would result in the most satisfying testing configuration. Thus a circular test geometry of 80mm diameter was used, with eight bolts clamped at 30Nm torque, as the holding jig for the impact specimens.

The impactor mass and shape has also seen much variation in previous research. The tup shape strongly influences the amount of energy absorbed by the specimen [9] and therefore there is no standard geometry for impact testing. However, it has been shown that a hemispherical tup allows the
5.2 Experimental details

(a) Metal fibre arrangement on mould plate  (b) Filament winding of glass fibres

(c) Bagging of the mould and contents.  (d) Impregnated plates released from mould.

**Figure 5.1** Details of the production procedure.

**Figure 5.2** The drop-weight instrumentation utilised in impact testing
material to reach the highest peak force and produced the shortest contact duration. A hemispherical
tup of 16mm diameter was used and the entire impactor had a 931.4g mass.

The laminate plates were impacted from one of up to four heights, 95cm, 115cm, 175cm and
215cm, corresponding to 7J, 10J, 13J and 17J respectively, in order to impart varying degrees of
damage. As compression-after-impact tests were to be performed, the highest impact energy was
set to limit the damage area. The laminates were impacted either with the metal wires facing the
impactor (top side) or on the opposite side (bottom side). Force-time and force-deflection were directly
obtained during the impact event. Absorbed energy was obtained from the integration of the force-
deflection plot. In some cases, the force, and hence absorbed energy, could not be measured, due to
instrumentation problems with the impact tower.

5.2.3 Compression after impact test method

Compression-after-impact (CAI) tests based on ASTM D7137 were performed on the damaged spec-
imens. The impacted laminates were cut to 80x120mm dimensions using a diamond saw. During
impact, the damage area always grew preferentially along either the 0° or 90° direction, hence the cuts
were made such that the greatest length of the damage area would be along the axis of compression.
Strain gauges were utilised as outlined in the standard to ensure accurate alignment of the specimen
with the applied load and as a check that the percentage bending was kept to less than 10%. Figure 5.3
shows the experimental set-up and arrangement of a test specimen. Anti-buckling plates were used of
the form shown in Figure 5.3(a). The failure mode was observed and those that did not fail within the
impact damage area were discarded. The maximum force prior to failure was recorded and ultimate
compressive residual strength calculated.

5.2.4 Laminate analyses

Samples were taken from each of the sections for volume fraction analysis using the resin burn-off
method described in ASTM D2584. After the burn-off procedure the glass and metal wires were
weighed together. The metal wires were then hand-picked from the dry glass fibres and were weighed
separately. The individual weights were checked against the original combined weight to ensure that no
glass was inadvertently removed. The weight and hence volume fraction was then calculated according
to the standard.

After impact, the damage area was obtained from ultrasonic c-scanning. In some cases, the
laminates were cut across the damage area and examined using optical microscopy.

5.3 Results

5.3.1 Laminate analyses

After release from the mould the laminates were cut and inspected. As a consequence of the vacuum
infusion method, the mould side of the laminate was smooth whereas the opposite side was very rough
due to the peel ply and bagging materials. The metal wires were wound first on the mould and hence
were on the "smooth side" of the laminate. It will be shown in the results that the surface of the
laminate had an influence on its impact performance.

Cross-sections of the various laminates were examined under the microscope and are shown in
Figure 5.4. During winding, the metal wires could not be kept equidistant from each other as the
smooth surface of the mould allowed some unavoidable movement after placement. This resulted in
the seemingly random distribution of the metal wires seen in the figures.
5.3 Results

(a) Schematic of CAI jig

(b) CAI fixture (left) and close-up of test specimen in fixture (right)

Figure 5.3 Details of the compression-after-impact fixture and test setup.

Figures 5.4(a) and (b) show examples of laminate cross-sections with the metal wires placed unidirectionally. Under greater magnification, the former figure shows two distinct layers, one of the metal wire/epoxy and the other of the glass fibre/epoxy (henceforth called the wire layer and glass layer, respectively). This occurred when the wires were placed perpendicularly to the next layer of glass fibres. The latter figure shows no separate layers, where the wires were laid in parallel with the glass fibres. During the winding process the glass fibres settled around the metal wires, effectively embedding them in the same layer.

Figures 5.4(c) and (d) show examples of laminate cross-sections with a 0/90 arrangement of the metal wires. Again under greater magnification two distinct layers can be seen of the wires and the glass fibres. Both laminates display a thicker wire layer with little visual difference between them.

The thickness of each laminate plate was measured and the metal wire weight and volume fraction calculated. The results are shown in Table 6.1 with laminates 1 and 9, 2 and 6, and 4 and 8 grouped together in order to highlight their similar metal content and wire arrangement. Laminates 1 and 4 were slightly thinner than laminates 9 and 8 due to the settling effect described previously. However, it will be shown that the differences in impact performance were not significantly affected by the different wire arrangement. Laminate 3 was thicker than any of the crossply laminates, which arose due to the closer average spacing of the wires. Waviness of the wires in one layer due to those underneath, and vice versa, was kept to a minimum, hence resulting in a thicker laminate. The density of laminate 3 was also the greatest, and the addition of 1.9% volume fraction resulted in an increase of 3% over that
of the plain laminate 7. The new nomenclature assigned to the laminates is listed in the Table.

5.3.2 Impact tests - metal wires on top face

Figure 5.5(a) shows the absorbed energy versus impact energy graph for the laminates impacted with the metal wires on the top (smooth) face. The plain laminates were also tested with the smooth face towards the impactor. With increasing impact energy, the hybrids generally absorbed more energy compared to the plain laminates. However, the spread of the test data, from 2.05J to 4.54J, also increased with increasing impact energy. From these results, it was unclear of the real influence of the metal wires as the plain laminates absorbed an amount of energy that was within this spread.

The influence of the metal wires was more notable with the impact energy-damage area graph, Figure 5.5(b). The increase in absorbed energy with impact energy was accompanied by an increase in damage area. After impact at 7J, the resulting damage area was notably less with the hybrids than with the plain laminates. Therefore the impact energy was dissipated in a way that restricted the damage area. The discrepancy between the absorbed energy and damage area was due to the
5.3 Results

<table>
<thead>
<tr>
<th>Plate number</th>
<th>Wire</th>
<th>Plate thickness (mm)</th>
<th>Glass volume fraction (%)</th>
<th>Metal volume fraction (%)</th>
<th>Density (g/cm³)</th>
<th>New name</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>UD</td>
<td>2.04 (0.03)</td>
<td>65.48 (1.39)</td>
<td>0.52 (0.05)</td>
<td>2.112 (0.020)</td>
<td>UD_low</td>
</tr>
<tr>
<td>8</td>
<td>UD</td>
<td>2.10 (0.01)</td>
<td>65.18 (1.61)</td>
<td>0.49 (0.05)</td>
<td>2.117 (0.053)</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>UD</td>
<td>2.05 (0.01)</td>
<td>64.36 (0.89)</td>
<td>1.01 (0.12)</td>
<td>2.109 (0.036)</td>
<td>UD_high</td>
</tr>
<tr>
<td>9</td>
<td>UD</td>
<td>2.10 (0.02)</td>
<td>64.11 (0.88)</td>
<td>1.00 (0.09)</td>
<td>2.124 (0.019)</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>CP</td>
<td>2.12 (0.02)</td>
<td>61.18 (0.55)</td>
<td>1.44 (0.12)</td>
<td>2.114 (0.009)</td>
<td>CP_med</td>
</tr>
<tr>
<td>6</td>
<td>CP</td>
<td>2.13 (0.02)</td>
<td>61.20 (0.84)</td>
<td>1.43 (0.12)</td>
<td>2.109 (0.014)</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>CP</td>
<td>2.12 (0.03)</td>
<td>61.20 (0.67)</td>
<td>0.96 (0.07)</td>
<td>2.082 (0.011)</td>
<td>CP_low</td>
</tr>
<tr>
<td>3</td>
<td>CP</td>
<td>2.16 (0.01)</td>
<td>61.00 (1.04)</td>
<td>1.91 (0.22)</td>
<td>2.142 (0.018)</td>
<td>CP_high</td>
</tr>
<tr>
<td>7</td>
<td>-</td>
<td>2.00 (0.02)</td>
<td>66.11 (0.67)</td>
<td>-</td>
<td>2.080 (0.009)</td>
<td>plain</td>
</tr>
</tbody>
</table>

Table 5.1 Plate details with standard deviations given in brackets. UD is unidirectional, CP is crossply.

The presence of the matrix-rich wire layer, which was able to locally plastically deform upon impact hence absorbing energy. A dent in the surface of the laminate resulted. As the impact energy was increased, the difference in damage area between the plain and the hybrid laminates reduced, but the plastically deformed dent on the surface was deeper and more evident. This means that at higher impact energies, the properties of the surface layer became less important than the bottom layer, i.e. beyond a certain impact energy level, the effect of the metal wire layer became minimal.

It was mentioned that there was a difference in the wire direction relative to the adjacent glass fibre direction for laminates UD_low and UD_high. As an example, the UD_high results were separated and highlighted by their original nomenclature of laminates 1 and 9 in both graphs in Figure 5.5. Laminate 1 did not perform consistently better than that of laminate 9, and vice versa, over the range of impact energies tested, highlighting that there was little difference with the wire arrangement. In addition, no particular laminate group performed differently to any of the others and therefore the influence of metal volume fraction was shown to have little influence when placed on the impact face.

Figure 5.6 shows the force-time plots for the plain and one of the hybrid laminates, in this case, that of the UD_high. At 7J, the plain laminates the force on the laminate reached a peak before reducing to zero as the impactor rebounded. The hybrid laminate reached a peak but maintained this force for a longer period of time before returning to zero. A peak plateau therefore resulted, which was due to the wire layer plasticly deforming around the impactor. At 17J, the force plots were more similar in shape.

Figure 5.6(b) shows the force-displacement plots for the same plain and hybrid laminate. At 7J the curves were similar. At 17J, the hybrid laminate showed a more open curve indicating that more energy had been absorbed.

Figure 5.7 shows selected plates after impact at the lowest and highest impact energies. The white area indicates the extent of the damage caused by delaminations and matrix cracking. No fibre damage, i.e. breakage, occurred at any of the impact energies. On the top surface, matrix cracking due to the impactor resulted in permanent indentation for all laminates. At all impact energies, delaminations occurred between the layers of the lower half of the laminate. On the back side, delamination between the last two layers and cracking parallel the fibre direction occurred, forcing push-out of the glass layer, the extent of which was dependent on the impact energy. At the lowest impact energy little differences between the plates were observed. At the higher impact energy differences in damage area were also similar in appearance, as the bottom glass layer offered relatively little resistance to damage.
5.3.3 Impact tests - metal wires on bottom face

Figure 5.8(a) shows the absorbed energy versus impact energy graph for the laminates impacted with the metal wires placed on the bottom (rough) face. The plain laminates were also tested with the rough face towards the impactor. As with the results of the previous section, there was also a spread of test data but the influence of the metal wire layer could more clearly be seen. At the lowest impact energy, the hybrids generally absorbed more energy than the plain laminates, but this behaviour was reversed as the impact energy increased. It should also be noted that the absorbed energy for the plain and hybrid laminates was greater than those presented in the previous section, and was attributed to the surface quality of the laminates. On the rough face, the impact force of the impactor was spread over a greater area while the smooth face was more epoxy-rich, creating a buffer, or cushion, effect. These two effects resulted in greater absorbed energy, and were also found with Ross et al. [2].

The influence of impact energy on damage area also showed a clear trend, Figure 5.8(b). At 7J, there was very little difference in damage area between all of the laminates. However, as the impact energy increased, the damage area became significantly lower than that of the plain laminates. Considering the absorbed energy and damage area, no particular hybrid laminate group could be shown to have superior impact properties compared to the rest. Generally, the difference in impact behaviour within each of the laminate groups was greater compared to the results of the previous section, particularly seen with CP_mid. As the scatter between the plain laminates was small, this effect may be attributed to where the hybrid laminate was impacted and wire/matrix adhesion, and will be discussed in further detail later.

As a check once again whether there was a difference in impact properties within each laminate group, the UD_low and UD_high results were separated and highlighted by their original nomenclature of laminates 4 and 8 and laminates 1 and 9, respectively, in both graphs in Figure 5.8. It was expected that as the greatest damage area occurred on the bottom side of the laminate, placement of the metal wires relative to the adjacent glass layer would be more important, especially with increasing impact energy. In other words, the metal wires would be more effectively placed perpendicularly to the adjacent glass fibres, as was the case with laminates 8 and 9, to prevent crack propagation and push-out of the glass layer. Again laminates 1 and 4 did not perform consistently better than that of laminates 9 and 8, and vice versa, over the range of impact energies tested, and therefore the wire direction had little influence. As with the metal wires placed on the top side, no particular laminate group performed differently to any of the others and therefore the influence of metal volume fraction was shown to have little influence.

The behaviour of the plain and hybrid laminates was further investigated with the force-time and force-displacement plots. Figure 5.9(a) shows an example of the force-time plot for the plain and hybrid laminates, in this case, that of the CP_mid. At 7J the profiles were similar but at 17J were notably different. The plot for the plain laminate increased with time to the peak force, which was lower than that of the hybrid laminate. The lower peak force resulted from the bottom glass layer splitting and was pushed-out through the back of the laminate due to the impactor. A more gradual decrease in slope after the peak force was observed with the hybrid laminate. While the bottom glass layer was also split and some individual wires pushed-out, the damage occurred over a longer period of time. Poorer adhesion of the wire to the epoxy matrix compared with the glass fibres, debonding occurred allowing the wires to slide through the matrix, which may have contributed to preventing sudden damage, and absorb more energy.
Figure 5.9(b) shows the accompanying force-displacement plot for the plain and CP_{med} laminates. At 7J, the curve of the hybrid laminate was more open than that of the plain, indicating that more energy had been absorbed. This may have been due to the greater straining capability of the metal wire layer for the deformation at this low energy. Hence increased absorbed energy was observed because the entire laminate was able to deform more than with the plain laminate, without increasing the damage area. At 17J, the peak force was reached at the same displacement for both the laminates, but more severe damage occurred with the plain laminate as the peak was approached, indicated by the lower gradient initiated at around 4.5mm displacement. After peak load was reached, the plain laminate deflected further, indicating sudden damage, as was seen in the force-time plot and this was not seen with the hybrid. In this case, the wire layer served to restrict the absorbed energy. Crack propagation and delamination was reduced due to the reinforcing effect of the wires, and will be described more fully in the discussion. Hence the residual stiffness of the laminate was greater and more energy was returned to the impactor.

Figure 5.10 shows selected plates after impact at the lowest and highest impact energies. At the lower energy, it was already clear that the presence of the wire layer restricted the damage propagation along the fibre direction in the bottom glass layer. Hence, a more oval damage area was created with the hybrids rather than a cross-shape with the plain laminate. At the higher energy, the restriction of damage area was even more pronounced as delamination and damage to the bottom glass layer was reduced.

5.3.4 Residual strength

CAI tests were performed on the laminates in order to investigate whether the residual compressive strengths (RCS) were affected compared to the plain laminates, and the results are shown in Figure 5.11. Due to the limited number of test samples, undamaged specimens were not compression-tested. A number of laminates failed outside of the impact-damage zone, typically through local crushing, and therefore were discarded resulting in fewer CAI test results in the graphs. This type of CAI failure is typical of thin laminates [10] and occurred even with the anti-buckling plates used in this investigation. Nevertheless, enough samples failed in the desirable manner, within the damage area, for presentation of the results.

As expected for the plain laminates, the RCS decreased with increasing impact energy in both the top- and bottom-impacted cases. The scatter for the hybrids shown in Figure 5.11(a) was large and hence was difficult to draw any concrete conclusions from these tests. In the case of the hybrid laminates with the wires placed on the bottom face, Figure 5.11(b), there was a discernable influence of the wire layer, as the RCS of the hybrids were generally greater with increasing impact energy than those of the plain laminates. This was attributed directly to lower damage area after impact.

5.4 Discussion

It has been shown that at the lowest impact energy investigated, placement of the metal wires on the top of the laminate, impact side, resulted in a smaller damage area for the same absorbed energy compared to the plain laminates. However, the effect of the metal wires reduced with increasing impact energy. Conversely, placement of the metal wires on the bottom of the laminate resulted in increased contribution, particularly in terms of damage area, of the metal wires with increasing impact energy. The reason for the differences in impact behaviour with wire placement was attributed to the contribution of the bottom layers compared to the top plies. It is already known [11] that thin laminates experience the greatest damage at the bottom plies, on the opposite side to the impacted
side, due to the flexibility of the system. Therefore the critical factor in the impact of thin laminates is the mechanical properties of the bottom layer, as this is where damage propagation begins. Hence the metal wires were the most effective reinforcing the bottom layers.

Figure 5.12 shows cross-sections of two impacted hybrid specimens. It is clear that in the impact energy range investigated, the major damage occurred between the 2nd and 3rd and the 3rd and 4th layers in the form of delaminations and cracking of 3rd ply. It was important to note that there were no delaminations evident between the glass layer and the adjacent metal wire layer, which was due to the compliant nature of the wire layer. This means that the metal wire layer was able to strain with the glass layer throughout the impact event. Figure 5.13(a) shows the bottom-side of an impacted hybrid laminate, with the delamination between the 3rd and 4th layer highlighted. The metal wire layer beneath showed no evidence of damage, and hence was not affected by the delamination.

It was expected that the hybrid laminates would allow for greater energy absorption due to the ductility of the metal wires. In particular, this effect was expected to be more pronounced with wires placed on the bottom of the laminates. However, the opposite trend was found as the laminate absorbed less energy compared to the plain laminates. Two reasons was attributed to the lower absorbed energy observed: the first due stiffness and the second due to restriction of damage propagation. The presence of the metal wires increased bending stiffness, as well as the thickness, of the hybrid laminates, increasing impact resistance. Observation of the hybrids with the wires placed on the bottom of the laminate showed that the wires were pulled out due to splitting of the adjacent glass layer, Figure 5.13(b). A small amount of energy was likely absorbed though the pull-out process, and local plastic straining, and the effect was that crack propagation was restricted in the glass layer. In addition, the wires served to reinforce the bottom layer against the high flexural (tensile) stresses experienced during impact. As the splitting of the glass layer was reduced, the stiffness was great enough to prevent further deflection of the laminate. In this way, delamination propagation was also reduced and hence the hybrid laminate was able to return more energy to the impactor, reducing the absorbed energy compared to the plain laminates. This explanation can be applied to both the UD hybrids with the wires aligned perpendicular to the adjacent glass layer as well as those aligned parallel, which were pulled out in the direction of the glass fibres.

The differences in impact behaviour became more distinct with increasing impact energy for the hybrids with the wires placed on the bottom-side, as the metal wires were allowed to strain slightly and hence contribute more fully during the impact event. It has been shown that even with such low volume fractions investigated, the effect of metal wire additions is substantial when placed at the bottom face of the laminate, although little difference was found between the different hybrids investigated. The reason for the apparently random data scatter may have been due to where the impactor hit the laminate in relation to the position of the wires. If the wire was directly underneath the impactor nose, it was able to fully contribute to the strain of the bottom layer. Slight misalignment would have reduced this effect. In addition as the wire/matrix adhesion was poor, the extent of wire pull-out was not controllable, which also contributed to the scatter. If the hybrids are considered as a whole and their results averaged, at the highest impact energy investigated, a drop of approximately 9% and 20% in absorbed energy and damage area respectively was observed for a metal volume fraction of between 0.5-2%.

For further work, it would be interesting to perform impact at higher energies, close to and at perforation, where the properties of the bottom-most layer become even more critical. It is expected that the differences between the metal wire alignment and volume fractions would also be evident as the damage area becomes more localised. Inclusion of a greater volume fraction of metal would discriminate between different wire arrangements and determine whether impact properties could be further improved. Finally, more residual strength tests should be performed to gain a clearer perspective of
5.5 Conclusion

Stainless steel wires were added to the impacted (top) and non-impacted (bottom) side of glass/epoxy laminates and subjected to low velocity impact. Impact performance was measured in terms of absorbed energy, damage area and residual compression strength. Based on the experiments results, the following conclusions were made:

- Better impact resistance was found when the wires were placed on the bottom side of the laminate rather than the top compared to the plain laminates. The wires reinforced the bottom ply against the flexural tensile stress experienced during impact and increased the bending stiffness of the overall laminate.

- Contrary to expectation, absorbed energy decreased when the wires were placed on the bottom side of the laminate compared to the plain laminates. The wires themselves absorbed very little energy through pullout, but instead reinforced the laminate making it stronger and stiffer, resulting in reduced absorbed energy and damage area.

- There was very little difference in impact performance between the hybrid laminates with varying wire volume fractions, as hybrids with 0.5% volume fraction performed similarly to those with 2%. Considering the laminates with the wires on the bottom side, at the highest impact energy investigated, the average drop in absorbed energy and damage area of around 9% and 20% was found. Such a performance increase was paired with an increase in density of less than 0.04g/cm³.

- Compression after impact tests gave relatively little information on the effect on residual strength. Only a small number of specimens could be analysed as many failed undesirably. Therefore more tests need to be performed to gain better insight into how the presence of the metal wires affects the damage tolerance of the laminate.

Bibliography


Chapter 5. Impact of cross-ply laminates


Figure 5.5 Impact energy graphs for plain and hybrid laminates with the metal placed on top. Each graph contains a trend line for the plain laminates.
Figure 5.6  Force graphs for representative laminates impacted with the metal wires placed on top.
Figure 5.7  Visual inspection of specimens impacted with the metal wires placed on top.
Figure 5.8  Impact energy graphs for plain and hybrid laminates with the wires placed on the bottom. Each graph contains a trend line for the plain laminates.
5.5 Bibliography

Figure 5.9  Force graphs for representative laminates impacted with the metal wires placed on the bottom.
Figure 5.10  Visual inspection of specimens impacted with the metal wires placed on the bottom.
Figure 5.11  Residual compression strength after impact for the plain and impact specimens.
Figure 5.12  Cross-section images of impacted hybrid laminates with the metal wires placed either top-side or bottom-side.

Figure 5.13  Images of hybrid laminates impacted at 7J (left) and 17J (right) and with the metal wires on the bottom-side.
Chapter 6

Impact properties of woven glass reinforced polymers reinforced with metal mesh layers

6.1 Introduction

In the previous chapter, it was shown that hybridising metal wires with unidirectional fibres can improve impact performance. It would be interesting to take the idea one step further and apply metal wires to woven fabrics. However, the method of weaving of metal wires directly into the fabric material was not developed within this research. Instead, a metal mesh was used as a reinforcing layer for laminates subjected to low velocity impact.

It was the aim of this investigation to determine the effects of stainless steel metal mesh layers in glass fibre-reinforced polymer composites under drop-weight impact testing. Both thermoplastic and thermoset matrices were utilised to compare the effects of different glass reinforced polymer composites in combination with the mesh.

6.2 Background

As opposed to those tests performed in Chapter 5, a greater range of impact energies were examined, and hence the different levels of damage will be described in this section. One of the main reasons that every researcher must define their own damage modes is because the impact event and damage behaviour depend on many variables, such as specimen lay-up and material, impactor geometry, and so on. A damage mode that may appear in one case will not necessarily reappear under different impact conditions. Hence it is important to define the damage modes which will be used to characterise the behaviour of the composite specimens.

A four-category damage definition was used based on visual observation of the laminate surfaces. Visual observations are more useful than c-scan observations because better discrimination between failure types can be made as well as immediate categorisation after testing. Figure 6.1 schematically shows the four damage categories. BVID describes the damage in which no damage is visible on the impact side but can be barely seen at the back surface, or tensile side. Visible damage is that which can be seen on both sides with very little to no out-of-plane deformation of the laminate. Penetration is the point at which out-of-plane deformation occurs, usually resulting in a dome shape on the tensile surface, and the impact surface is able to move through-the-thickness with increasing impact energy.
The impactor can still get stuck in the laminate or rebound. Finally, perforation describes the point at which the impact tup is able to pass completely through the laminate resulting in the formation of a permanent hole.

6.2.1 Energy profiling

A number of methods have been employed in the literature to display and characterise the data from impact testing. Due to the number of tests performed in this investigation, a clear and concise method was desired and therefore the energy profiling technique described by Liu [1] was used to display the data obtained from the impact tests and is described further.

The absorbed energy is that absorbed by the specimen during the impact event i.e. the energy taken for deformation and inducing damage, and hence is a useful parameter in monitoring the damage progression when the impact energy is increased. The absorbed energies are calculated from the area beneath the load-deflection curves and plotted against the impact energies, an example of which is shown in Figure 6.2.

If an equal energy line is plotted, the curve can be divided into three distinct regions. The first (a) is the region in which there is sufficient energy for the impactor to rebound after strike. The greater the difference between the absorbed energy and the equal energy line, the greater the energy for rebound. The initial portion of region (a) does not imply the extent of the damage, nor whether damage has been initiated at all. However, as the curve tends towards the equal energy line, there is less rebound implying that the stiffness of the laminate has been reduced and thus indicates the presence of damage.

The second region, labelled (b), is the range of impact energies at which all energy is absorbed and has been defined as the penetration region [1]. In a physical sense, the impactor neither rebounds nor continues completely through the specimen, as energy is absorbed through major damage, deformation and friction effects.

The final region, labelled (c), is region in which perforation occurs. The drop of the curve away from the equal energy line is the excessive energy remaining with the impactor as it passes through...
the specimen.

![Figure 6.2 Energy profile of a series of impact performed at a number of impact energies [1]](image)

While the energy profiling methods provides a convenient way to display a large amount of data at once, it is also necessary to consider the "raw" data as well. The standard force-displacement and velocity-time graphs produced by the specimens that are at critical points on the graph (at the limits of each region) can be used to check impact behaviour. In addition, visual and ultrasonic c-scan inspections are also important to determine the extent of damage and to provide an insight to the damage mechanisms that have taken place.

### 6.3 Experimental details

Glass fibre/polyphenylene sulphide (PPS) semipregs and glass fibre/polyetherimide (PEI) prepregs were used as the base material for the compression moulded thermoplastic laminates. Glass fibre and Hexion EPR/H 4908 epoxy resin was used as the base material for the vacuum infused thermoset laminates. For all materials, the glass fabric was an eight harness satin weave. The PEI plain laminates were composed of 14 layers, due to material restrictions, and the rest of 16 layers. The laminates were laid up as $[0/90]_n.s$. A plain woven AISI 304 stainless steel mesh of wire diameter 112$\mu$m and opening 358$\mu$m (Mesh 54), was placed in three different positions within the laminate stack, as shown in Figure 6.3. When a mesh layer was included in the stack, one glass layer was removed in order to keep the overall thickness the same. In the case of the hybrid thermoplastic laminates, two extra polymer foils were included to ensure good impregnation of the mesh layers. Pre-tests had shown that two meshes showed more promising results than only using one mesh layer within the laminate. These pre-tests are outlined in Appendix B. Therefore, for the PPS and epoxy hybrid laminates two mesh layers were used, and one for the PEI as the laminate was somewhat thinner. Before use, the mesh was cleaned as described in Chapter 4. Finally, density tests were performed using the method outlined in Chapter 5.

The impact procedure was the same as that described in Chapter 5 apart from the impactor, which was 15mm in diameter and 2.128kg in mass. All samples were impacted from barely visible impact damage (BVID) to perforation. The results are displayed in terms of absorbed energy, taken from the area underneath the force-displacement graph, versus the impact energy, determined from the tup velocity, and is a convenient way to compare the impact data. Ultrasonic c-scan inspection
Chapter 6. Impact of woven laminates

Figure 6.3 Overview of the laminate stacking sequence.

<table>
<thead>
<tr>
<th>Laminate type</th>
<th>Mesh placement</th>
<th>Plate thickness (mm)</th>
<th>Density (g/cm³)</th>
<th>New name</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEI</td>
<td>0</td>
<td>3</td>
<td>1.96</td>
<td>PEI_plain</td>
</tr>
<tr>
<td>PEI</td>
<td>top, middle, bottom</td>
<td>3</td>
<td>2.04</td>
<td>PEI_(top/middle/bottom)</td>
</tr>
<tr>
<td>PPS</td>
<td>0</td>
<td>3.6</td>
<td>1.96</td>
<td>PPS_plain</td>
</tr>
<tr>
<td>PPS</td>
<td>top, middle, bottom</td>
<td>3.5</td>
<td>2.17</td>
<td>PPS_(top/middle/bottom)</td>
</tr>
<tr>
<td>Epoxy</td>
<td>0</td>
<td>4.3</td>
<td>1.81</td>
<td>ep_plain</td>
</tr>
<tr>
<td>Epoxy</td>
<td>top, middle, bottom</td>
<td>4.4</td>
<td>1.90</td>
<td>ep_(top/middle/bottom)</td>
</tr>
</tbody>
</table>

Table 6.1 Plate details

was also performed to determine the damage area of the samples after impact.

6.4 Results

6.4.1 Laminate analyses

Details of the laminates can be found in Table 6.1. It should be noted that after consolidation, the hybrid thermoplastic samples with the mesh placed in the top/bottom were slightly curved in nature, with the mesh on the inside of the curve. This was due to the differences in coefficient of thermal expansion (CTE) between the mesh layer and the glass layer, leaving the mesh side in compression, and the opposite in tension.

Figure 6.4 shows examples of typical hybrid laminate cross-sections, showing that all the mesh layers were well impregnated. Due to the high consolidation pressure, the mesh layers settled into the glass layers. In the case of the PPS hybrid laminates, packing of the meshes reduced the overall thickness of that layer. This did not occur with the epoxy hybrid laminates, accounting for the greater difference in thickness between the plain and the hybrid laminates.

6.4.2 PEI laminates

The results in this section show that of the energy and damage energy profiles. Vertical lines have been plotted on each curve of the energy profiles to mark the perforation energies of each laminate type.
6.4 Results

![Microscope images of typical cross-sections of the mesh layers.](image)

Figure 6.4  Microscope images of typical cross-sections of the mesh layers.

Figure 6.5(a) shows the energy profile for the PEI, PEI_TOP, PEI_MIDDLE and PEI_BOTTOM laminates. The plain and hybrid laminates performed similarly until impact energies close to perforation were reached. The plain laminate perforated at 82J, which was matched by the PPS_TOP laminate. The PEI_MIDDLE perforated at 77J, which was 6% lower than the plain laminate, whereas the PEI_BOTTOM perforated at 87J, 7% higher.

Figure 6.5(b) shows the damage area profile for the PEI laminates. The PEI_MIDDLE produced the greatest damage area due to a delamination that occurred at the mesh/glass interface.

Figure 6.6 shows the images of the damage at the impact and bottom sides at various impact energies of the PEI, PEI_TOP, PEI_MIDDLE and PEI_BOTTOM laminates. The plain laminate failed through fibre and matrix crushing on the impact side, and finally tensile failure in the shape of a cross on the bottom side. As impact energy increased, the size of the tensile failure grew until buckling occurred in a diamond-like shape surrounding the cross. The PEI_BOTTOM laminate exhibited similar behaviour, but showed a more ductile-type tensile failure on the bottom side at perforation with buckling occurring on only one part of the cross, where the impactor forced through.

6.4.3 PPS laminates

Figure 6.7(a) shows the energy profile for the PPS, PPS_TOP, PPS_MIDDLE and PPS_BOTTOM laminates. The profiles show that the plain laminates absorbed the least amount of energy, especially towards perforation, and the PPS_MIDDLE laminates the most, however the difference in absorbed energy was very small. There were no clear differences in perforation energies between the plain and hybrid laminates, as all perforated around 70J of impact energy.

There were more differences evident in the damage area profile, Figure 6.7(b), where it is clear that the PPS_MIDDLE laminates produced the greatest damage areas. For the rest of the laminates, the PPS, PPS_TOP laminates produced the smallest damage areas.

Images of the damage of the impact and bottom sides at various impact energies are presented in Figure 6.8. The progression of damage on the front and back surfaces of the plain and hybrid laminates were very similar, hence for comparison only the images for the PPS, PPS_TOP laminates are shown. In all cases, the damage appeared to be very localised on both the front and back surfaces. In the plain laminates, at the edges of the damage zone, the glass fibres locally buckled to accommodate the failure, which is evidenced by the wrinkle-like appearance in these areas. The hybrid laminates failed in a similar way but a smaller instance of buckling on the surface mesh layers.

Figure 6.9 shows the cross-section of the plain and hybrid laminates after perforation. The plain laminate revealed fibre and matrix crushing on the impact side with multi-level delamination through the thickness, and finally tensile failure and buckling on the back surface. The PPS_TOP laminate exhibited straining of the mesh layer, which was forced through the thickness by the impactor during perforation. The PPS_MIDDLE laminate had a large major delamination running along the mesh/glass
interface accounting for the large delamination area measured by the c-scan inspection. Finally, the PPS_bottom laminate was similar in appearance to the plain laminate apart from the delamination observed at the mesh/glass interface.

6.4.4 Epoxy laminates

Figure 6.10(a) shows the energy profile for the ep_plain and ep_top, _middle and _bottom laminates. The energy profiles show that a similar trend in absorbed energies before perforation occurred with both laminate types, differences only occurring at the perforation energies. The plain laminate perforated at 82.4J, which was exceeded by 13.5%, 12.1% and 21.0% for the top, middle and bottom configurations respectively. At the perforation energy of the plain laminates, the hybrid laminates had not reached maximum absorbed energy, and enough energy was returned to the impactor for rebound in each case.

The difference in impact behaviour was more discernable in the damage area profile, Figure 6.10(b). At the lower impact energies, the ep_plain and ep_middle laminates produced the largest damage areas, and the ep_top laminates produced the least. Just before perforation a change in failure mode occurred and the ep_bottom laminates produced the largest damage area.

The perforation damage mode on the back surfaces for the plain and the hybrid laminates are presented in Figure 6.11. The plain laminate, Figure 6.11(a), perforated in a diamond shape, with tensile-cracks formed along the diagonals. Buckling occurred along the edges of the damage area from compression failure. The addition of the mesh layers, Figure 6.11(b), changed the failure appearance on the bottom side as buckling was completely eliminated. The result was a more metal-like deformation as the mesh layers allowed the laminate to deform and accommodate the impactor.

Figure 6.12 shows the cross-section of the plain and hybrid laminates after perforation. The plain laminate displays fibre and matrix crushing on the impact side, with multi-level delamination through the thickness, and finally tensile failure and buckling on the back surface. The ep_top laminate displays little evidence of crushing in the mesh regions, where the tough combination of mesh-reinforced epoxy absorbed some of the impact energy. There were also fewer delamination layers. The ep_middle laminate displays the largest delaminations, none of which occurred at the mesh layer. Finally, the ep_bottom laminate showed similar failure to the plain laminate on the impact side, and also displays multi-level delaminations, but is the only laminate to eliminate buckling on the back surface.

6.5 Discussion

The results have shown that the effect of the metal mesh layers depend on the matrix used. This section will discuss and attempt to differentiate between the mechanisms that contribute to the overall impact behaviour of the laminates.

The most common behaviour observed was that with increasing impact energy to before perforation, the absorbed energy was similar between the plain and hybrid laminates, and this was seen with all three matrix types. The damage areas on the other hand, revealed more differences, which increased with increasing impact energy and depended on the matrix type.

The impact velocities used in this investigation were low, and due to the relative flexibility of the laminates, compared to say, carbon fibre laminates, the main response during impact was through bending. This meant that large tensile stresses were generated in the bottom-most ply and hence depended on the properties of this layer.

For the thermoplastic matrices, the hybrids with the mesh layer placed on top resulted in similar damage areas as the plain laminates, meaning that the contribution of the top layer during impact at the lower energies was very small. Placement of the mesh on the bottom of the laminate resulted in
increasing damage with increasing energy because this layer contributed more to the overall impact behaviour. As an illustration, Figure 6.13 shows the force-displacement curves of the plain and hybrid laminates for the PEI and PPS laminates at one impact energy. Two details are of importance. The first is the lower impact stiffness, as the introduction of the mesh layer reduced the overall bending stiffness of the laminate. The second is the greater displacement compared to the plain and hybrid laminates with the mesh placed impact-side. The mesh layer is tougher and more compliant than the glass layers and offers lower resistance to the vertical displacement of the impactor. Both these observations show that the mesh contributed more to the overall impact behaviour of the laminate when placed at the bottom. Finally, mesh layers placed in the middle created the greatest damage areas, and also absorbed slightly more energy, due to delaminations directly at the mesh/glass interface. The damage area was especially large for the PPS laminates due to the poor metal mesh/matrix adhesion, which will be discussed further on.

For the epoxy laminates, different behaviour was observed between the plain and hybrid laminates with increasing impact energy. The hybrids with the mesh placed in the middle and the top produced generally similar damage areas as the plain laminates, while those with the mesh at the bottom produced slightly less. All laminates at lower impact energies produced similar damage areas, suggesting that the impact behaviour seen with the thermoplastic matrices were affected by the presence of a thicker, tougher mesh layer in the epoxy laminates. Figure 6.14 shows the force-displacement curves of the plain and hybrid laminates for the epoxy laminates at one impact energy. The meshes in this case played a greater part during the impact event. The peak force was sustained during a greater deflection when the mesh was placed on top, and a greater deflection was achieved when the mesh was placed on the bottom.

It was mentioned earlier of those thermoplastic laminates with the mesh layers placed on the outside of the laminates were curved due to thermal expansion mismatches, meaning that there was a different distribution of residual stresses through the thickness of the laminate. It could be argued, then, that absence of the residual stresses allowed the damage area of the hybrid epoxy laminates to be lower than that of the plain, which was not observed with the thermoplastic laminates. However, the effect of these residual stresses could not be determined and requires further investigation. The subject of residual stresses shall be dealt with in more detail in Chapter 7.

There was very little difference in perforation energy between the plain and hybrid PPS laminates. This was attributed to poor metal/matrix adhesion. As the laminate deformed during impact, there was low resistance to debonding and crack propagation in the resin rich mesh layer. None of the hybrid configurations were therefore able to delay the laminate from splitting on the tensile side at perforation, weakening the laminate on the tensile face.

An increase in perforation energy was seen with the epoxy laminates with the mesh placed in the bottom configuration. As opposed to the PPS laminates, there was sufficient adhesion between the matrix and the embedded mesh, allowing the mesh layer to strain as the laminate deformed. The resin rich matrix layer on the tensile side of the hybrid laminates was able to deform in more of a membrane-type deformation due to higher achievable strain of the mesh layer compared to that of a glass layer. The mesh reinforced the epoxy, and due to the adhesion properties, received the tensile load from the matrix. This plastic-like energy-absorbing process delayed the perforation energy. A similar effect also occurred with the PEI laminates for the same reasons, although the perforation was not delayed to such a great extent. The volume fraction of the metal may have been too low for the toughness of the mesh layer to have had much influence.

An increase in perforation energy was also seen for the epoxy laminates with the mesh placed on the top and the middle, which was not observed with the PEI laminates. In a similar way with the meshes placed at the bottom, this was likely due to toughness. Lu et al. [2] have shown that...
low-modulus, high-strain layers can alleviate stress concentrations induced under the impactor nose, thereby altering the stress distribution of the laminate, contributing to an increase in impact resistance. In the case of the mesh placed at the top, this would have meant that the impact stress under the loading nose was spread over a greater area. For the mesh in the middle, which essentially acts as a constraining layer for the upper and lower halves of the laminate, this would have meant that it acted as a toughening interleaf layer. Evans et al. [3] have found that damaged interleaved epoxy composites can carry more load than equivalent epoxy laminates. As there was only one layer of mesh in the PEI laminates, such a resistance to perforation did not occur.

The gradient in damage area increased up to perforation, which was generally greater than that of the plain laminates. As the impact energy increased, the mesh layer participated more in the impact event, nominally through plastic straining. Due to a difference in stiffness between the mesh layer and the adjacent glass layers, delaminations were initiated at, and close to, the mesh layer. The delaminations grew as the laminate deflected during perforation.

Finally, it should be noted that the perforation energy of the PEI and epoxy laminates with the mesh placed in the bottom did not increase due to the straining of the metal wires within the mesh, which strained very locally and hence likely did not absorb a large amount of energy in the process, Figure 6.15.

6.6 Conclusion

This study investigated the impact-energy absorbing potential of woven glass fibre reinforced polymers reinforced with stainless steel mesh layers. The following conclusions were drawn:

- The presence of the mesh does not greatly affect the absorbed energy at lower impact energies for any of the matrices investigated. Damage area was a better indicator for changes in impact behaviour and discriminated more between the plain and hybrid laminates.

- Placement of the mesh on top, impact-side, did not affect the damage areas of the thermoplastic laminates, nor the perforation energy compared to the plain laminates. However, for the epoxy laminates, damage areas were smaller and the perforation was delayed. This was attributed to the greater thickness of the mesh layers compared to those in the thermoplastic laminates, which acted as a toughening layer and absorbed more energy during impact through deformation.

- Placement of the mesh in the middle increased the damage area for all the matrices investigated, compared to their respective plain laminates. The extent of the increase was particularly large with the PPS laminates, and this was attributed to poor metal/matrix adhesion. The perforation energy was only delayed for the epoxy laminates as the mesh layer acted as a toughening interleaf.

- Placement of the mesh at the bottom had the greatest effect for the PEI and epoxy laminates, where perforation energies were delayed by 7% and 21%, for an increase in density of 4% and 5% respectively. This was attributed to the toughening effect of the mesh layer, which absorbed more energy than when placed on the top of the laminate, and membrane-like deformation. A greater delay was seen with the epoxy hybrid laminates due to the thicker mesh layer. Little effect in impact behaviour was observed for the PPS laminates.

- For all the hybrids, the damage area became larger than the plain laminates as the perforation energy was approached. This was due to the presence of the mesh layer, which strained greatly to accommodate the impactor, thereby creating larger delaminations, particularly at, or close to, the mesh layers.
Finally, it should be noted that the effect adding metal mesh layers was a combined result of the mesh and matrix. The tests performed with the three matrices revealed that there must be sufficient metal volume fraction, metal/matrix adhesion and matrix ductility to result in an overall toughening effect for the laminate. In this way, at lower impact energies, damage area can be reduced and perforation delayed to a higher energy.

Bibliography


Figure 6.5  Energy and damage area profiles for plain and hybrid PEI laminates.
6.6 Bibliography

Figure 6.6  Images of impacted laminates at various energies.
Figure 6.7  Energy and damage area profiles for plain and hybrid PPS laminates.
Impact side

(i) 24J  
(ii) 55J  
(iii) 72J  
(a) PPS_plain laminates  
Impact side

Bottom side

(i) 24J  
(ii) 54J  
(iii) 70J  
(b) PPS_top laminates

Figure 6.8 Images of impacted laminates at various energies.
Figure 6.9  Microscope images of the PPS laminates’ cross-sections after perforation.
Figure 6.10  Energy and damage area profiles for plain and hybrid epoxy laminates.
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Figure 6.11 Images of impacted laminates at various energies.
Figure 6.12  Microscope images of the epoxy laminates’ cross-sections after perforation.
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Figure 6.13  Force-displacement graphs of plain and hybrid laminates.
Epoxy laminates impacted at 66J

**Figure 6.14** Force-displacement graphs of plain and hybrid laminates.

**Figure 6.15** Local straining of metal wires in hybrid laminate.
Chapter 6. Impact of woven laminates
Chapter 7

Flexural properties of glass-fibre reinforced thermoplastics reinforced with metal mesh layers

7.1 Introduction

The presence of a metal mesh in glass reinforced thermoplastic laminates influences impact properties, as was shown in Chapter 6. It was noted that after the production process, the hybrid impact specimens were curved in shape, due to differences in thermal expansion coefficient between the mesh layer and the glass layers. As a result, greater thermal residual stresses were present in the hybrid laminates than in the flat plain laminates. However, at very low impact energies, i.e. at lower strain rates, the difference between the plain and hybrid laminates was generally indiscernible, indicating little, if any, influence of the residual stresses. Therefore it was of interest to examine more closely the behaviour of the hybrid laminates under low strain rates using a more sensitive test method, the flexural test.

This Chapter investigates the flexural behaviour of glass fibre reinforced thermoplastics with a stainless steel mesh layer. The effect of thermal residual stresses was also investigated and classical laminate theory utilised to help understand the effect of these stresses on the flexural properties.

7.2 Experimental details

7.2.1 Materials

Eight harness satin weave E-glass fibre reinforced polyetherimide (PEI) prepreg and poly-phenylene sulphide (PPS) semipreg were used as the base materials. The prepreg/semipreg was first dried in a vacuum oven for 120°C and 60°C respectively for at least 24 hours before use. AISI 304 type stainless steel mesh of 140μm diameter and 495μm opening was used. The meshes were either cleaned, anodised or grit-blasted as described in Chapter 4. Table 7.1 gives various properties of the materials used in the tests.

Baseline plain laminates were made by stacking 9 plies of semipreg/prepreg in the 0° direction. For the hybrid, one layer of glass ply was replaced by a layer of mesh, which was placed in one of three different positions, either at the outer surfaces of the laminate, or in the middle. Additionally, two extra thermoplastic foils were required to properly impregnate the mesh.

The ply stack was assembled in a picture-frame mould and consolidation took place in a hot
platen press. Figure 7.1 shows the press cycle used for the laminates. After consolidation, laminates were cut to the required dimensions using a diamond saw. At least 7 specimens per plate were cut along the 0° direction, that is, along the warp. The specimens were then placed in a vacuum oven under the aforementioned conditions to keep them dry. The specimens were allowed to cool naturally to room temperature when required for testing. The total laminate thickness was 2.07mm for all laminates.

### 7.2.2 Flexural test procedure

All specimens were tested under 3-point bending according to ASTM D790-03 under a span-to-depth ratio of 40:1. The 3-point bending test was chosen above the 4-point test due to ease of procedure. In addition, it has been shown that the modulus can be overestimated with the 4-point test [1, 2], while strength values are similar [2]. Load and displacement values were used to calculate the flexural strength, modulus and strain to failure. After testing specimens were examined using optical microscopy or SEM.

### 7.2.3 Residual stress measurement

After consolidation the top and bottom configurations were transversely concave with the mesh layer on the inside. The presence of the mesh caused laminate distortion due to the large differences in CTE between the matrix-rich mesh layer and the rest of the laminate. Hence, thermal residual stresses resulted upon cooling from the process temperature. The maximum height at the middle of the

<table>
<thead>
<tr>
<th>Name</th>
<th>Tensile strength (MPa)</th>
<th>Compression strength (MPa)</th>
<th>Flexural strength (MPa)</th>
<th>CTE (µm/m·°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass/PEI</td>
<td>484.0</td>
<td>727.0</td>
<td>669</td>
<td>30.4</td>
</tr>
<tr>
<td>Glass/PPS</td>
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<td>566.0</td>
<td>511</td>
<td>28.6</td>
</tr>
<tr>
<td>Neat PEI</td>
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<td>152.0</td>
<td>138</td>
<td>55.8</td>
</tr>
<tr>
<td>Neat PPS</td>
<td>90.3</td>
<td>148.0</td>
<td>125</td>
<td>52.2</td>
</tr>
<tr>
<td>AISI304 mesh</td>
<td>819.0</td>
<td>-</td>
<td>-</td>
<td>17.3</td>
</tr>
</tbody>
</table>

Table 7.1 Mechanical properties (warp) and coefficient of thermal expansion values for test materials.

Figure 7.1 Laminate temperature-pressure cycle.
7.2 Experimental details

Figure 7.2 Schematic arrangement of laminate lay-up for PSL analysis (left) and possible deformation after separation (right).

Figure 7.3 Measurement set-up for PSL analysis (left) and close-up of laser and separated layer (right).

The laminate for the PEI and PPS specimens were around 1.0mm. It was then suspected that there may be considerable residual stresses with the mesh in the middle configuration. In order to measure the extent of the residual stresses, the process simulated laminate (PSL) technique was employed. This process is fully described in [3], but a short description is given here.

The PSL technique consists of building up a laminate with a certain number of plies separated by a release layer. After processing, the laminate can be separated at specified layers. Internal stresses due to, for example, CTE mismatch or temperature variations through the thickness of the laminate during processing will deform each separated layer. The deformation takes the shape of a curve. For this investigation, simply the measurement of the maximum height of the curve is taken as qualitative indication of the residual stress gradient.

A plain and a hybrid middle configuration laminate was produced for both PEI- and PPS-based laminates. The laminate lay-up is schematically shown in Figure 7.2. The same processing conditions as those for the flexural specimens were used for consolidation. After consolidation the lamintes were cut to 14cmx13cm with a diamond saw. The laminate was then carefully separated using a knife edge at each polyimide layer.

A laser coupled with a multimeter was used to measure the maximum height of each separated layer, as shown in Figure 7.3. Figure 7.2 also shows an image of the laser-point on a curved separation layer. A multimeter voltage reading was taken of a reference flat sheet before that of the curved layers and the difference between the two readings was taken to obtain a "deflection voltage". The voltage was then converted to a distance using a calibrated conversion factor.
Chapter 7. Flexural properties

7.3 Results

All laminates were subjected to 3-point bending and the test results are presented in this section. PEI and PPS laminates were named with the prefixes "PEI" and "PPS", followed by "p", "t", "m" or "b" indicating plain, hybrid top, middle and bottom configurations respectively.

7.3.1 Flexural properties - glass/PEI

The stress-strain curves for each configuration of PEI laminate are shown in Figure 7.4. Two distinct groups of curves can be seen, with the PEI_p and PEI_m specimens exhibiting almost linear behaviour until failure and the PEI_t and PEI_b laminates exhibiting clear non-linear behaviour. The non-linearity was attributed to the onset of matrix cracking in the matrix-rich mesh layer.

Table 7.2 gives an overview of the flexural ultimate stress, modulus and failure strain of the glass/PEI laminates. Compared to the plain PEI laminates, the PEI_m produced an improvement of 16% and 13% in the stress and strain respectively. These values are highlighted in the table. The other configurations resulted in similar stress values as the plain laminates, laying within one standard deviation of the average while the moduli decreased and strains increased considerably.

<table>
<thead>
<tr>
<th>Name</th>
<th>Flexural Stress</th>
<th>Flexural Modulus</th>
<th>Flexural Strain</th>
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<td>SD</td>
<td>Change (%)</td>
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<td>-</td>
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<td>683.2</td>
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<td>+16</td>
</tr>
<tr>
<td>PEI_b</td>
<td>576.6</td>
<td>9.4</td>
<td>-2</td>
</tr>
</tbody>
</table>

Table 7.2 Overview of flexural results, including standard deviation (SD) and percentage change of the hybrid laminate properties over those of the plain.
Specimens were examined using SEM before and after the flexural tests in order to gain insight into the differences in damage modes. The major failure mode of the PEI laminates were similar for the four different types of laminates, as tension dominated, as shown in Figure 7.5. Under three-point bending, the PEI_p specimens initially failed through first-ply buckling on the compression side followed by sudden tensile failure starting at the bottom-most ply, Figure 7.5(a). The buckling was often located under the loading nose and was therefore due to a stress concentration, but the failure was primarily tension-dominated. This was expected as the mechanical properties of glass/PEI are greater in compression than in tension.

The PEI_t ply experienced debonding between the mesh layer and the first glass layer although tension failure resulted, Figure 7.5(b). Upon closer inspection, extensive matrix cracks could be seen in the mesh layer, which ran towards, and then around, the metal fibres. The presence of the matrix cracks contributed to the non-linearity seen in the stress-strain curves. The extent of fibre/matrix debonding was small, as the cracks commonly ran a small distance around the metal fibres before continuing again into the matrix. Therefore the increase in failure strain found over the plain laminates was mainly due to matrix cracking and deformation.

Figure 7.5(c) shows that the PEI_m laminates had different behaviour compared to the other laminates. This was expected as under bending loads, shear stresses dominate in the centre of the laminate. Failure commenced by catastrophic tensile failure of the plies with delamination at the mesh layer. It is interesting to note that the delamination occurred directly at the mesh layer/glass layer interface rather than through matrix cracking that ran along the metal fibres. Clearly the fibre/matrix bonding was sufficient to resist the shear stresses and allow the flexural properties to improve.

Similar to the plain laminates, the PEI_b specimens exhibited tension-dominated failure, Figure 7.5(d). As PEI is weaker in tension, there was little resistance to failure in the matrix rich regions, which proceeded to the metal fibres. Once failure was initiated in the mesh layer, sudden tensile failure progressed through thickness of the laminate. On closer inspection, delamination was also seen along the mesh layer/glass layer interface, which may have been due to the sudden energy release associated with the tensile failure. Delamination progression progressed at this interface as a result of stiffness mismatch between these two layers.

### 7.3.2 Flexural properties - glass/PPS

The flexural stress-strain curves for the PPS laminates are shown in Figure 7.6. The PPS_p laminate exhibited linear behaviour up to a distinct first failure, indicated by the arrow in the figure, after which there was a non-linear increase until final failure. The hybrids all exhibited non-linear behaviour and the same distinct first-failure, indicated on the curves by a sudden drop in stress, with the PPS_m laminates producing the greatest drop at this point. The non-linearity of the top and bottom configurations were attributed to matrix cracking and early debonding between the metal fibres and matrix.

Table 7.3 gives an overview of the flexural properties of the glass/PPS laminates. In contrast to the PEI laminates, a marked decrease in the stress and modulus was found compared to those of the plain PPS. Interestingly, the PPS_m laminates did not mirror the behaviour of the PEI_m; while the flexural modulus did not greatly vary, the stress and strain decreased by 19% and 13% respectively. In fact, of the PPS laminates, the strain values were the only ones to decrease.

SEM images of the PPS laminates were taken after failure and are shown in Figure 7.7. Figure 7.7(a) shows the failure of the plain glass/PPS laminate. The PPS_p laminates failed in compression, which progressed though successive plies towards, and until, the mid-line (neutral axis). The failure then terminated in tension. It is likely that a stress concentration due to the loading nose may have been the initiator, and as the difference between the tension and compression values were less than
Chapter 7. Flexural properties

Figure 7.5  SEM images of the glass/PEI laminates and their hybrids (left) with closer inspection (right).

for PEI, compression failure was more easily induced. The effect of gradual ply-by-ply failure on the compression side was seen in the stress-strain curve, where there was a small drop in stress and then a decrease in gradient until sudden final failure.

A similar failure mode occurred with the PPS t laminates, Figure 7.7(b). In this case, the stress concentration underneath the loading nose simply deformed the PPS matrix and did not initiate the failure. Instead, compression failure occurred because the top few layers of the laminate were already in compression before testing. No matrix cracks were evident in the mesh layer during SEM inspection, but metal fibre/matrix debonding did occur which contributed to the lower flexural strength of the
7.3 Results

Figure 7.6 Typical flexural stress-strain curves for PPS-based laminates.

<table>
<thead>
<tr>
<th>Name</th>
<th>Flexural Stress (MPa)</th>
<th>Flexural Modulus (GPa)</th>
<th>Flexural Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PPS_p</td>
<td>493.0 12.7 -</td>
<td>25.4 0.6 -</td>
<td>0.023 0.000 -</td>
</tr>
<tr>
<td>PPS_t</td>
<td>430.2 15.0 -13</td>
<td>21.4 0.6 -16</td>
<td>0.027 0.001 +18</td>
</tr>
<tr>
<td>PPS_m</td>
<td>400.9 9.1 -19</td>
<td>24.3 0.9 -4</td>
<td>0.020 0.001 -13</td>
</tr>
<tr>
<td>PPS_b</td>
<td>385.2 23.5 -22</td>
<td>21.4 1.1 -16</td>
<td>0.027 0.001 +16</td>
</tr>
</tbody>
</table>

Table 7.3 Overview of flexural results, including standard deviation (SD) and percentage change of the hybrid laminate properties over those of the plain.

laminate. Cracks were initiated and propagated within the glass ply directly beneath the mesh layer and continued through the thickness until final tensile failure.

As with the PEI_m specimens, different behaviour was observed with the PPS_m laminates, Figure 7.7(c). Compression failure initiated and progressed to the mesh layer, where extensive fibre/matrix debonding resulted. This was indicated as a pronounced first drop in the stress-strain curve. After this drop, the residual flexural strength was lower, and the laminate deflected until the test limit was reached without any tensile failure.

Tensile failure were the primary mode for the PPS_b laminates, Figure 7.7(d). The poor metal fibre/matrix adhesion coupled with the low tensile strength of the PPS led to failure initiation at the mesh layer. The failure was gradual as the matrix was able to yield considerably, and this was seen by the moderate slope reduction at the first stress drop on the stress-strain curve. Eventually failure continued with compression failure at the top few plies.

7.3.3 Surface treated specimens

In an attempt to improve the flexural properties, and to investigate the importance of the metal fibre/matrix adhesion, the mesh was surface treated and tested. Subscripts “ano” and “gb” refer
Chapter 7. Flexural properties

![SEM images of the glass/PPS laminates and their hybrids](a) PPS_p

![SEM images of the glass/PPS laminates and their hybrids](b) PPS_t

![SEM images of the glass/PPS laminates and their hybrids](c) PPS_m

![SEM images of the glass/PPS laminates and their hybrids](d) PPS_b

**Figure 7.7** SEM images of the glass/PPS laminates and their hybrids (left) with closer inspection (right).

to anodised and grit-blasted specimens respectively. The middle and bottom configurations were tested as, particularly for the PPS laminates, metal fibre debonding was prevalent. Figure 7.8 shows the flexural stress-strain curves of the treated hybrids, with the original untreated hybrids and plain laminates as comparison. The stress-strain curves for the PEI laminates clearly show that all the curves of the treated hybrids follow the same trend as the untreated hybrids. The PPS laminates however revealed slightly different curves: the distinct first failure indicated by a drop in stress for the plain laminates and untreated hybrids disappeared. In addition, the non-linearity seen in the untreated hybrid curves became less pronounced. In both PEI/PPS cases, the grit-blasted hybrids decreased the
### 7.3 Results

<table>
<thead>
<tr>
<th>Name</th>
<th>Flexural Stress</th>
<th>Flexural Modulus</th>
<th>Flexural Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Value (MPa)</td>
<td>SD (MPa)</td>
<td>Change (%)</td>
</tr>
<tr>
<td>PEI\textsubscript{p}</td>
<td>589.2</td>
<td>10.5</td>
<td>-</td>
</tr>
<tr>
<td>PEI\textsubscript{m, ano}</td>
<td>695.0</td>
<td>16.3</td>
<td>+18</td>
</tr>
<tr>
<td>PEI\textsubscript{b, ano}</td>
<td>668.7</td>
<td>18.2</td>
<td>+13</td>
</tr>
<tr>
<td>PEI\textsubscript{b, gb}</td>
<td>545.0</td>
<td>21.8</td>
<td>-8</td>
</tr>
<tr>
<td>PPS\textsubscript{p}</td>
<td>493.0</td>
<td>12.7</td>
<td>-</td>
</tr>
<tr>
<td>PPS\textsubscript{m, ano}</td>
<td>573.5</td>
<td>13.5</td>
<td>+16</td>
</tr>
<tr>
<td>PPS\textsubscript{b, ano}</td>
<td>458.4</td>
<td>23.2</td>
<td>-7</td>
</tr>
<tr>
<td>PPS\textsubscript{b, gb}</td>
<td>303.2</td>
<td>26.0</td>
<td>-39</td>
</tr>
</tbody>
</table>

**Table 7.4** Overview of flexural results, including standard deviation (SD) and percentage change of the treated hybrid laminate properties over those of the plain.

The flexural performance and therefore were not used in the middle configuration.

Table 7.4 gives an overview of the flexural properties and compares the performance to the plain laminates. The flexural properties for the PEI laminates were different depending on the position of the mesh layer. Treatment of the mesh did not vastly improve the behaviour over the PEI\textsubscript{m} laminates, confirming that the natural adhesion of the PEI laminates to the stainless steel mesh was already sufficient in this case. However, in the bottom configurations, clear improvements in the stress and strain were achieved with anodising compared to the plain laminates, without compromising the modulus. This was obviously due to improved load transfer at the metal fibre/matrix interface. It was therefore expected that the same would be found with the grit-blasted specimens, but a significant reduction in stress and modulus resulted, and the reasons for this will be explained later.

Unlike the PEI laminates, improvements in flexural properties were found for both PPS configurations. Anodising the mesh for the middle configuration increased the flexural values even above that of the plain laminates. This highlighted the weakness of the fibre/matrix adhesion in the failure mode of the PPS\textsubscript{m} laminates, which can be concluded to have been the direct reason for the lower flexural values. In the bottom configuration, there was an improvement in flexural properties of the PPS\textsubscript{b, ano} over the PPS\textsubscript{b}, but were still lower in value than the plain laminate. While the adhesion improved the load transfer between the metal fibres and matrix, the low tensile strength of the PPS was the weak-point. As with the PEI\textsubscript{b, gb} laminates, the grit-blasted specimens produced a large decrease in stress and modulus.

In Chapter 4, it was seen that similar levels of adhesion was achieved with both grit-blasted and anodised stainless steel sheet specimens. Therefore it was expected that the same improvement in flexural properties would also be achieved with the treated meshed. The reason for this difference was due to two causes, the first procedure-related and the second due to a physical reason. The particles used for grit-blasting were between 150-210\(\mu\)m in size. Considering that the mesh diameter was only 140\(\mu\)m, surface roughening was limited. In addition, it was not guaranteed that the entire surface area of the mesh was reached. Figure 7.9 shows the SEM images of the untreated and treated meshes. It appeared that the treatments were successfully applied to the meshes with the entire surface roughened by grit-blasting or anodising. On closer inspection, the extent of roughening was clearly different. The grit-blasted mesh was flaky in nature, as indicated in Figure 7.9(b) and was not as aggressive a treatment as anodising. These flakes did not provide a physically stable surface for the matrix to which to adhere as the bending load was applied, thereby reducing the flexural performance.
The anodised mesh provided a far greater, and more physically stable, surface area for the matrix to mechanically lock, as shown in Figure 7.9(c).

It should be noted that there was an increase in modulus for laminates with the anodised mesh compared to those with the untreated mesh, and was the case for both the PEI and PPS laminates. The same increase was not seen with the grit-blasted specimens. The SEM images show that the surface morphology had been vastly altered through the anodising process and this may have been a contributing factor to the stiffness increase. However, it is also likely that these results were erroneous and therefore need further investigation. Hence the modulus values will not be further considered in the discussion.

### 7.3.4 Residual stresses

Residual stresses in the form of ply deformations were measured of the plain and middle configuration laminates and are shown in Figure 7.10. The deformations were measured at the maximum height of the curved ply layer, and the ply number refers to the position within the laminate as described in Section 7.2.3. The plain PEI and PPS laminates show a relatively smooth decrease in curvature to the midline. This is typical residual stress generation that arises through non-isothermal solidification of the laminate during the cooling stage of consolidation [3]. As a result, the outer layers 1 and 6 were under the greatest compression stress and the central layers 3 and 4 were in tension.

The hybrid laminates show very different behaviour in the central plies of the laminate, where there was a large increase in curvature. The PPS laminates were curved more than the PEI laminates and therefore had greater levels of residual stresses in the centre.

Although the CTE of PEI is higher than that of PPS, the residual stress arose from differences in matrix shrinkage. It is known from literature that the shrinkage of thermoplastic polymers depends on whether the polymer is amorphous or semi-crystalline [4]. Two types of shrinkage occurred in the case of semi-crystalline PPS; that due to the temperature drop, and that due to densification upon crystallisation. In the case of amorphous PEI, only the former applied. It has been shown that the total shrinkage for semi-crystalline matrices can be an order of magnitude higher than that for amorphous matrices [4], accounting for higher residual stresses present in PPS.

### 7.4 Discussion

Two different failure modes were found for the glass/PEI and glass/PPS plain laminates and the reasons were observed and accounted for in the previous section. Summarising, glass/PEI is far stronger in compression than in tension and therefore tension failure dominated. The difference in compression and tension properties for the glass/PPS is much smaller, and due to stress concentrations at the loading nose of the test fixture, compression failure was initiated.

The flexural behaviour of the hybrids, however, could not be fully explained in terms of failure inspection. The results have shown that the presence of the mesh has a pronounced influence, particularly on the flexural stress and strain, which will be discussed in this section. In addition, classical laminate theory (CLT) was employed to help understand the effect of the mesh layer through analytical considerations.

#### 7.4.1 Mechanical contribution of the mesh layer

It was not entirely clear from the experimental results the role played by the mesh layer and in particular, whether the mesh layer contributed mechanically to the flexural properties of the laminates.
In order to determine the contribution of the mesh layer, CLT was utilised. Changes in laminate flexural stiffness (rigidity), shift of neutral axis and calculation of the transverse shear was calculated.

Under 3-point bending, the deflection in the centre of the laminate is given by [5]:

\[
    w_c = \frac{PL^3}{48bd_{11}} \left( 1 + 12\frac{f_{55}}{d_{11}L^2} \right)
\]

where \(w_c\) is the deflection in the centre of the laminate, \(P\) is the applied load, \(b\) and \(L\) are the width and the span length of the laminate respectively, and \(f_{55}\) is the inverse of \(F_{55}\), a constituent of the ABD matrix according to [5], also known as the transverse shear stiffness. Lastly, \(d_{11}\) is the flexural stiffness in the warp direction and is a term in the abd matrix, defined as:

\[
    \begin{bmatrix}
        \epsilon \\
        \kappa
    \end{bmatrix} = 
    \begin{bmatrix}
        a & b \\
        b & d
    \end{bmatrix} 
    \begin{bmatrix}
        N \\
        M
    \end{bmatrix}
\]

Considering that a span of around 80mm was used and that \(d_{11} >> f_{55}\), as indicated by the elastic and shear moduli in Table 7.5, for both the PEI and PPS laminates, the term in the brackets in Equation 7.1 approximates to 1. At this point, it is already clear that the contribution of the transverse shear to the deflection is negligible. Rearranged for \(P/w_c\), the following is obtained:

\[
    \frac{P}{w_c} = \frac{48b}{d_{11}L^3}
\]

The values shown in Table 7.5 were used to calculate all the terms in the ABD matrix which was inverted to obtain the abd matrix. The thickness of each ply and of the mesh layer was taken as the total laminate thickness divided by the number of layers. Then \(P/w_c\) was calculated for the plain and hybrid laminates for both PEI and PPS and the results are shown in Table 7.6. For the top and bottom configurations, the stiffness values of the mesh/matrix layers were considerably lower than that of the glass/matrix layers, resulting in the drop in \(P/w_c\). Similar stiffness reductions in the experimental results was found for the hybrid laminates with the untreated meshes, at roughly -18% and -11% for the top and bottom configurations respectively for PEI and -16% for the top and bottom configurations for PPS. Therefore the drop in flexural stiffness of the top and bottom configurations was shown to be directly due the mesh layer.

It was already stated that the contribution of the transverse shear to the laminate deflection was negligible. Nevertheless the maximum shear stress due to bending, \(\tau_o\), was also calculated for each of the laminate configurations using:

<table>
<thead>
<tr>
<th>Material</th>
<th>(E_x) (GPa)</th>
<th>(E_y) (GPa)</th>
<th>(\nu_{xy})</th>
<th>(G_{xy}) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass/PEI</td>
<td>26.0</td>
<td>24.0</td>
<td>0.29</td>
<td>3.36</td>
</tr>
<tr>
<td>Glass/PPS</td>
<td>22.0</td>
<td>20.0</td>
<td>0.29</td>
<td>3.71</td>
</tr>
<tr>
<td>PEI/mesh</td>
<td>11.2</td>
<td>11.2</td>
<td>0.29</td>
<td>3.36</td>
</tr>
<tr>
<td>PPS/mesh</td>
<td>11.7</td>
<td>11.7</td>
<td>0.29</td>
<td>3.71</td>
</tr>
</tbody>
</table>

Thickness mesh layer = 0.23mm  
Thickness glass layer = 0.23mm  
Span length = 80mm

Table 7.5  Data used for CLT calculations.
Chapter 7. Flexural properties

(a) glass/PEI

(b) glass/PPS

Figure 7.8 Typical flexural stress-strain curves for PEI- and PPS-based laminates with treated mesh.

<table>
<thead>
<tr>
<th>Name</th>
<th>$P/w_c$ (N/mm)</th>
<th>% Change</th>
<th>Name</th>
<th>$P/w_c$ (N/mm)</th>
<th>% Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEI_p</td>
<td>23.42</td>
<td>-</td>
<td>PPS_p</td>
<td>19.82</td>
<td>-</td>
</tr>
<tr>
<td>PEI_t/b</td>
<td>19.66</td>
<td>-16.1</td>
<td>PPS_t/b</td>
<td>17.23</td>
<td>-13.1</td>
</tr>
<tr>
<td>PEI_m</td>
<td>23.40</td>
<td>-0.1</td>
<td>PPS_m</td>
<td>19.81</td>
<td>-0.1</td>
</tr>
</tbody>
</table>

Table 7.6 Calculated theoretical values of $P/w_c$. 
7.4 Discussion

\( \tau_o = -\frac{3P_o}{2bh} \)

Table 7.7 shows the maximum shear stress, experienced at the neutral line of the laminate, was very low in each case. It was shown in the results that each of the middle PEI and PPS configurations experienced delamination as a failure mode. According to these values, the maximum shear stress of the matrix should not have been reached and therefore another reason was responsible for this failure. In addition, because no direct stresses are experienced at the neutral axis and the calculated maximum shear stress was so low, theoretically the mesh layer in the middle cannot have made a significant mechanical contribution during bending.

**Figure 7.9** SEM images at two magnifications of the untreated and treated meshes.

**Figure 7.10** Maximum height measurements of plain and hybrid laminates.
Chapter 7. Flexural properties

**Table 7.7** Maximum shear stress values for each laminate configuration.

<table>
<thead>
<tr>
<th>Laminate</th>
<th>$\tau_o$ (MPa)</th>
<th>Laminate</th>
<th>$\tau_o$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEI_p</td>
<td>13.0</td>
<td>PPS_p</td>
<td>10.9</td>
</tr>
<tr>
<td>PEI_t</td>
<td>11.4</td>
<td>PPS_t</td>
<td>9.1</td>
</tr>
<tr>
<td>PEI_m</td>
<td>15.1</td>
<td>PPS_m</td>
<td>8.1</td>
</tr>
<tr>
<td>PEI_b</td>
<td>11.8</td>
<td>PPS_b</td>
<td>8.3</td>
</tr>
</tbody>
</table>

7.4.2 Effect of adhesion

Treatment of the mesh was necessary to improve the flexural strength over the untreated meshes, and for both the PPS and PEI laminates, the effect was clear for mesh placed in the bottom. This was expected as the mechanical properties of the mesh were able to fully contribute in resisting the direct tensile stresses generated during bending. However, it should be noted that the increase in flexural strength cannot simply have been due to the presence of the mesh layer. Compared to the matrix, the metal volume fraction was very low in the mesh layer, which could already be deduced from the mesh dimensions. Hence the mechanical contribution of the metal was also relatively low to the overall laminate.

While theoretically placement of the mesh in the middle did not significantly contribute during bending, it was shown in the case of the PPS laminates that adhesion was necessary to improve the flexural properties. The PEI laminates with treated meshes showed no improvement in flexural properties over the untreated meshes, as the natural adhesion of PEI to the mesh was sufficient. This indicated that a minimum level of adhesion was required to resist transverse shear.

7.4.3 Effect of thermal residual stresses

The mechanical properties of the mesh and the mesh/matrix adhesion have proved to play a part on the flexural properties of the hybrid laminates, but cannot account for the large increases seen in the tests, particularly in strength. It was also shown that another effect was present that contributed greatly to the flexural behaviour of the laminates, and this can be attributed to the thermal residual stresses.

It was noted that the laminates with the mesh placed on an outer layer were curved in shape after processing. The curvature arose due to thermal residual stresses as a result of CTE differences between the matrix-rich mesh layer and the adjacent glass, with the mesh layer being in tension and glass layers in compression. There was also significant residual stresses induced in the laminates with the mesh placed in the middle, and was shown to be the case with the PSL tests. In order to calculate the theoretical values of the thermal residual stresses, CLT was again employed.

If the laminate with the mesh in the middle is first considered, it can be assumed that after processing, the laminate is in equilibrium i.e. that the tensile stress in the mesh layer = the compression stress in the adjacent glass fibre layers. Figure 7.11 schematically shows this condition. Additional bending stresses do not need to be considered, as the laminates were flat after production. It is also assumed that the laminates cooled uniformly through-the-thickness.

From compatibility:

\[(Fa_{11} + \alpha \Delta T)_{meshlayer} = (Fa_{11} + \alpha \Delta T)_{glasslayer}\]

where $\alpha$ is the CTE, $\Delta T$ the change in temperature and $a_{11}$ a stiffness term obtained from the abd matrix. The positive left term indicates that the mesh layer pulls on the glass layer due to the higher
7.4 Discussion

Figure 7.11  Equilibrium conditions for the mesh in the middle (left) and in the top/bottom (right)

<table>
<thead>
<tr>
<th>Input</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Delta T$ (°C)</td>
<td>290</td>
</tr>
<tr>
<td>thickness mesh layer, t (cm)</td>
<td>0.23</td>
</tr>
<tr>
<td>thickness glass layer, t (cm)</td>
<td>0.23</td>
</tr>
<tr>
<td>$\alpha_{\text{meshlayer}}$ (µm/m-°C)</td>
<td>19.16</td>
</tr>
<tr>
<td>$\alpha_{\text{glasslayer}}$ (µm/m-°C)</td>
<td>10.06</td>
</tr>
<tr>
<td>x (mm)</td>
<td>0.9816</td>
</tr>
<tr>
<td>$z_{\text{interface}}$ (mm)</td>
<td>0.8666</td>
</tr>
</tbody>
</table>

Table 7.8  Input values for residual stress calculations on a glass/PEI hybrid laminate.

CTE, and conversely for the negative term on the right. Equation 7.5 can be rearranged to find the internal load, $F$, and consequently the stress acting on the mesh and glass layers. The stress is that induced after production and hence is of the thermal residual stress.

If the laminates with the mesh in the top or bottom layer is considered, an extra term must be included in Equation 7.5 that takes into account the curvature of the laminate. After processing, an extra strain was produced, due to the curvature, which resulted in an internal bending moment:

$$ (Fa_{11} + \alpha \Delta T)_{\text{meshlayer}} = (-F a_{11} + \alpha \Delta T - F x d_{11} z_{\text{interface}})_{\text{glasslayer}} $$

where $x$ is the distance of the midline of the mesh to the neutral axis and $z_{\text{interface}}$ is the distance of the neutral line to the mesh/glass layer interface. Figure 7.11 schematically shows this condition. In a similar way, the residual stress may be found in the mesh and glass layers.

The values used to calculate the residual stresses are found in Table 7.8. A 9-ply glass/PEI laminate was considered, and a glass layer was replaced with a mesh layer for the hybrid laminates. The full abd matrix and CTE calculations may be found in Appendix A. The results are shown in Table 7.9.

Table 7.9  Results of residual stress calculations on a glass/PEI hybrid laminate.

The distribution of the residual stress distribution through the thickness of the hybrid laminates is also shown in Table 7.9. It can be seen that there was a contribution to the flexural property reduction for the top hybrid configuration. After processing, the bottom glass plies were in tension, which helped to accelerate the initiation of tensile failure. The converse occurred for the bottom configuration.

Compared to the compression and tension strengths of glass/PEI, the resulting residual stresses appeared to be very small. Therefore to check the theoretical values, the curvatures of the hybrids
Chapter 7. Flexural properties

Table 7.9 Results of the residual stress calculations on a glass/PEI hybrid laminate.

<table>
<thead>
<tr>
<th>Mesh in the middle</th>
<th>Mesh in the top/bottom</th>
</tr>
</thead>
<tbody>
<tr>
<td>F = 6.465 N/mm</td>
<td>F = 5.726 N/mm</td>
</tr>
<tr>
<td>( \sigma_{\text{meshlayer}} = 28.11 \text{MPa} )</td>
<td>( \sigma_{\text{meshlayer}} = 24.90 \text{MPa} )</td>
</tr>
<tr>
<td>( \sigma_{\text{glasslayer}} = -3.51 \text{MPa} )</td>
<td>( \sigma_{\text{glasslayer}} = -3.11 \text{MPa} )</td>
</tr>
</tbody>
</table>

with a mesh in the top/bottom were considered. The resulting curvature was calculated and compared to that of the real test specimens. The radius of curvature, \( R \), was calculated as follows:

\[
R = \frac{1}{k_x} = \frac{1}{M_{\text{internal}}d_{11}}
\]

where \( k_x \) is the curvature, taken from the abd matrix and \( M_{\text{internal}} \) is the internal moment due to the internal residual load, \( F \). From the radius of curvature, the maximum height in the middle of the laminate may be calculated. For a laminate of length 100mm, the maximum height was calculated to be 0.44mm. Compared to the real specimen height of 1.0mm, this was very low. Hence in reality, the residual stress induced during the production process was greater than that calculated in Table 7.9, and the same can be assumed for the laminate with the mesh in the middle. There may have been a number of contributing factors for this discrepancy. One was due to the assumption that during production, cooling of the laminate was uniform through-the-thickness, which was not the case, and therefore a cooling-rate gradient was present. Another factor was due to the assumption that the hot-press plates, which were in contact with the laminate during production, did not have an affect in constraining the surface plies.

The consideration of residual stresses within the mesh ply is also important. While the overall mesh layer was in tension, the mesh itself, constraining the matrix, was in compression. The mesh was effectively solely contributing to the stiffness and the strength of the mesh layer and its state of compression obviously also had an impact with the middle and bottom hybrid configurations. Therefore there was a more complex arrangement of residual stresses within the hybrid laminates, which was not considered and which needs further investigation.

Nevertheless, the increase strength for the mesh in the middle configuration is most likely explained by the presence of compressive residual stresses. These stresses had to be overcome before
the laminate failed in tension, thereby resisting failure until a greater stress. These stresses were large at the mesh layer compared to the rest of the laminate, as indicated by the deflections measured in Section 7.3.4.

7.5 Conclusion

Glass fibre/PEI and PPS laminates were reinforced with a metal mesh layer and tested in flexure under 3-point bending. It was found that flexural strength and strain improved over the plain glass laminates when the mesh layer was placed in the middle of the ply-stack. In this configuration, improvements of up to 18% and 17% in the flexural strength resulted for the PEI and PPS hybrid laminates, respectively, with little compromise in the modulus. The following observations and conclusions were drawn:

- The flexural stiffness of the plain and hybrid laminates were calculated using classical laminate theory and were shown to be in good agreement with the experimental values. It was concluded that the stiffness reduction observed in the hybrids were directly due to the presence of the mesh layer.

- Thermal residual stresses were generated during processing as a result of CTE mismatch between the mesh layer and the glass layers, and contributed greatly to the behaviour of the laminates during flexure. The residual stresses were crudely calculated for the hybrid laminates and it was shown that, as a result, the mesh layer was highly in tension.

- For the mesh placed in the middle, the flexural properties improved due to the presence of significant thermal residual stresses, which placed the glass fibres in compression. As both the PEI and PPS laminates failed in tension, this was favourable for flexure.

- For the mesh placed in the bottom layer, adhesion of the metal to the matrix was critical for improvements in flexural strength, which were due to the mechanical contribution of the mesh and the favourable residual stress distribution within the glass layers. The strength increase was not as large as with the middle configuration, as the mesh layer was taking direct tensile load and thus failure was more readily initiated in the weaker matrix-rich regions.

- For the mesh placed in the top layer, only flexural strain increased. As the failure was tension-dominated there was very little mechanical contribution from the mesh layer. In addition, the residual stress distribution was not favourable for tensile failure.

Hence through this investigation it was shown that a combination of the mesh mechanical properties and thermal residual stresses affected the behaviour of the hybrid laminates in flexure. This has implications for low velocity impact, where the residual stresses would also have contributed to the response of the hybrid laminates.

Bibliography


Chapter 7. Flexural properties


Chapter 8

Heat Emitting Layers for enhancing NDE

8.1 Introduction

Composites are susceptible to damage but are difficult to inspect because defects may occur, and begin, beneath the surface undetectable to the naked eye. To meet the increase in composite usage, technologies must be developed for non-destructive evaluation (NDE) of the structures such that they may be performed relatively quickly, and efficiently, during routine inspection. Currently ultrasonic methods, such as c-scanning, are most commonly used for NDE and are extremely effective in detecting, and extracting information about, defects [1]. However, these are point methods, and scanning needs to be performed over the entire area for larger-scale detection to take place, which is both time consuming and expensive [2]. In addition, while c-scanning may be suitable for quality control during manufacturing, the suitability is lost when a method is needed for inspection during service life of the structure. A number of NDE techniques are currently being researched and developed for assessing damage in composite laminates, such as thermography [3, 4], optical methods [5, 6, 7], electrical resistance methods [8, 9, 10] and acoustic emission [11]. Of these, infrared thermography is revealing its potential as a suitable NDE tool because through-the-thickness inspection is enabled simply through analysing surface temperature changes.

Although infrared thermography is already proving to be a useful method, composites do not behave as the ideal material for such analysis. Composites, and particularly unidIRECTIONalS, are anisotropic materials and as a consequence there is much noise associated with thermal images that are produced with thermography. The noise must be filtered out before proper analysis or distortion of information will result. Therefore, a simpler method to clearly detect and visualise defects in laminates utilising the benefits of infrared thermography would obviously have a huge impact on the NDE procedure.

This chapter describes a new approach for thermography inspection that uses a fully integrated heat emitting layer (HEL) within the laminate. The idea was to show that a HEL adds a NDE capability function to the overall composite structure for fast and large area inspection. First finite element simulations were performed to help predict and understand the optimum parameters for the HEL. In the experimental stage, flat laminates were prepared and the results used to compare with those of the simulations. In addition, two more complex shapes, a wing leading edge and a pipe section, were considered as real case studies for the application.
8.2 Heat emitting layer for thermography

Infrared thermography NDE is a full-field, non-contact method that allows through-the-thickness inspection by simply analysing surface temperatures with an infrared detector. The technique is based on a thermal radiation interaction between the inspected body and the detector which depends on their temperatures, material properties and geometric configuration. The net heat flow between a body and the infrared detector is depicted in Figure 8.1. It is clear that only a fraction of the emitted energy by the body reaches the detector. This net heat flow, based on the Stefan-Boltzmann law, is described as follows:

\[ Q_{\text{net}} = A_1 F_{1-2} \sigma (T_1^4 - T_2^4) \]

Where, \( A_1 \) is the transversal area of the investigated body, \( \sigma \) is the Stefan-Boltzmann coefficient \((5.6704 \times 10^{-8} \text{W/m}^2 \cdot \text{K}^4)\) and \( T_1 \) and \( T_2 \) are the temperatures of the specimen and detector. \( F_{1-2} \) is the transfer factor and it depends on the surface emmisivity as well as the geometrical arrangement or shape factor of both bodies.

Two types of thermal imaging techniques exist that are used to examine subsurface features of structures [12]. The first is a passive approach in which the tested specimen has an initial temperature that is different to that of the ambient. The second is an active approach where an external dynamic stimulus is applied to the specimen surface to alter its temperature. In both cases, the presence of a defect alters the heat transfer through the material, and the result is a change in surface temperature distribution. In many situations, NDE of a composite test area takes place when the overall structure is not in operational use and hence a significant temperature difference does not exist. An active approach is necessary to analyse the test area for defects and is the focus of this study. Furthermore, if this technique is to be used while the structure is in service, there are many situations in which the underside is out of reach for inspection. The most useful techniques are those in which inspection of only one side is necessary to visualise through-the-thickness. Therefore, in this study all inspection is from one side only.

With previous thermography NDE techniques, it has been standard practice to use an external source to heat the sample composite material [13]. A number of drawbacks are associated with this method [12] such as difficulties in obtaining a fast and uniform temperature change over a large test
area, finding an appropriate position of the heat sources with respect to the test area, and noise
generated due to anisotropy and inhomogeneity. In this study, the concept of using an internal heat
source in what has been termed HELTHY (Heat Emitting Layer for THermographY), was investigated.
It was aimed at reducing drawbacks associated with thermography.

In the study, the concept utilises a HEL comprising of woven glass fabric with unidirectionally
interwoven electrically resistive fibres, particularly metallic ones, placed at a specified distance apart.
The same glass fabric can be used for the HEL as the rest of the laminate. Busbars are placed
perpendicularly across the resistive fibres at each end of the hybrid fabric allowing for an electric
current to be transferred to the resistive fibres, as shown in Figure 8.2. The HEL heats upon the
application of a current and a heat front progresses through the thickness of the laminate, shown in
Figure 8.3. In the case of a defect, such as a delamination above the HEL, the heat front is delayed
due to the insulative effect of the air gap. This delay can be monitored as a temperature distribution
on the surface using an infrared camera as it is depicted in Figure 8.4. The idea of heating from
within is that thicker, and geometrically more complex shaped, laminates may be investigated because
a uniform heat front can be created.

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{helthy.png}
\caption{Schematic of the hybrid HEL with electrical connections.}
\end{figure}

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{helthy_detection.png}
\caption{HELTHY detection process.}
\end{figure}
8.3 Simulation

With the aim of finding optimal conditions for HELTHY, several variables such as power input, horizontal separation of the wires $S$, vertical position of the HEL within the laminate $V$, heating time $t_h$ and delamination geometry $L$ were investigated using a thermal parametric finite element (FE) model based on the heat diffusion equation in two dimensions:

\[
\nabla \cdot (K \nabla T) + \dot{q} = \rho C_p \frac{\partial T}{\partial t}
\]

where $T$ is the temperature, $K$ is the thermal conductivity, $\rho$ is the density, $C_p$ is the specific heat capacity, and $t$ time. The term $\dot{q}$ is an internal volumetric heat generation source and this power is generated by the HEL.

The defect or delamination was simulated as an air pocket or a foreign insert within the composite. From the heat transfer point of view, the presence of defects within the composite acts as an additional thermal resistance to the heat flux. Figure 8.4 describes the main geometrical variables and typical FE results for HELTHY are also presented. The simulation of the heat transfer process through the laminate, described by Equation 8.2, was performed using Matlab coupled with Comsol Multiphysics, a commercial FE software.

In this investigation, a 6-ply glass fabric/PPS laminate with a thickness of 1.5mm was studied. The composite was assumed to be homogeneous and orthotropic. Stainless steel metal wires of 70$\mu$m diameter were used for the HEL and they had a wire spacing, $S$, of 5mm. A power input of 20W...
8.3 Simulation

<table>
<thead>
<tr>
<th>Material</th>
<th>$K$ (W/mK)</th>
<th>$\rho$ (kg/m$^3$)</th>
<th>$C_p$ (J/kgK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel</td>
<td>16.3</td>
<td>7930</td>
<td>475</td>
</tr>
<tr>
<td>GF/PPS</td>
<td>0.722</td>
<td>2066</td>
<td>937.5</td>
</tr>
<tr>
<td>PI</td>
<td>0.25</td>
<td>1400</td>
<td>1200</td>
</tr>
<tr>
<td>Air</td>
<td>0.0257</td>
<td>1.205</td>
<td>1005</td>
</tr>
</tbody>
</table>

Table 8.1 Thermal properties for the material used in the finite element model.

(0.17W/cm$^2$) activated for 10s, was applied on the metal wires to simulate similar conditions as those used in the experimental study. These values were chosen after rough experimental pretests were performed. The laminate width was defined as 5S or 25mm in this case. The defect was simulated as a polyimide (PI) insert of 5mm length with a thickness of 125µm. Free convection conditions were applied in the top and lower surfaces with a constant ambient temperature of 20°C. The lateral boundaries were thermally insulated, in order to simulate thermal symmetry. The material properties for the laminate were computed using the rule of mixtures with 60% wire volume fraction, and they are shown in Table 8.1.

![Figure 8.5](image)

Figure 8.5 Influence of HEL position on the temperature difference measured at the composite surface with a power input of 0.17W/cm$^2$ and 10s heating.

In order to analyse the data obtained from the simulations, two temperatures $T_d$ and $T_f$ were defined as the temperatures measured on the laminate surface above the defect and a metal wire respectively. The influence of the vertical HEL placement, $V$, on the difference in temperature ($\Delta T$) between $T_d$ and $T_f$ can be observed in Figure 8.5. Three positions for the HEL were analysed: top-ply, middle-ply and bottom-ply, and delaminations simulated at every layer, with the 1st at the bottom surface and the 5th closest to the upper surface. It is clear that $\Delta T$ is strongly related with the HEL and defect locations. The highest $\Delta T$ occurred when the HEL and defects were placed on the top ply.
of the laminate; then, this value decreased quickly for deeper defects. When the HEL was placed in the bottom-ply, an even \( \Delta T \) distribution was obtained for all defect locations. However, these values were smaller than 0.5\(^\circ\)C because a considerable part of the applied power was transferred through the bottom surface to its surroundings. As a result, a small heat flux was useful for detection. Finally, the placement of the HEL in the middle-ply produced a symmetric heat flux through the laminate and therefore higher \( \Delta T \) values were found in general for all defects.

At this point, it is important to note the relevance of \( \Delta T \). This value provides a link between the thermal resolution of the infrared detector and HEL/THY. Thus, the detection probability of a defect using a given hardware is proportional to \( \Delta T \), i.e. the smaller the \( \Delta T \), the lower the detection probability. The most obvious way to increase \( \Delta T \) is by increasing the input power and an example of the result of doubling the power is shown in Figure 8.6. The other way is by changing the heating time. Figure 8.7 shows the influence of \( t_h \) on \( \Delta T \) for defect positions 1 and 5, which are the most interesting from the temperature point of view. It is clear that for heating times larger than 10s, there is not an appreciable increase on \( \Delta T \), which means that there is a lengthy time-base available for inspection. In reality, the material properties are temperature dependent, thus \( \Delta T \) may decrease with time. However, the detection capability then is limited only by the thermal resolution of the infrared detector.

The last variable of interest was the spacing of the metal wires, where two cases were studied at the same power input: 2.5mm (Figure 8.8a) and 10mm (Figure 8.8b). It can be observed that \( \Delta T \) increased a little for the 10mm separation in comparison with the 2.5mm case. Therefore from the thermal resolution point of view, the increased number of metal wires do not significantly improve the detection capabilities of the technique for the case considered. However, the minimum defect size for detection is proportional to \( S \).

Based on the simulation results, it can be concluded that the placement of the HEL depends on
8.4 Experimental technique

8.4.1 Flat laminate preparation

Glass fabric (GF)/polyphenylene sulphide (PPS) prepregs were used in this investigation. PPS thermoplastic was chosen because it is a matrix used in high-level engineering applications such as primary aircraft structures. Fine copper mesh was used as the busbar material due to the high electrical conductivity of the metal. For the HEL, AISI316 type stainless steel, 70µm diameter, was used due to its excellent corrosion resistance and deformation (forming) properties. The HEL layer was made by hand weaving the stainless steel wires into satin 8H glass fabric at desired intervals. The presence of blue glass fibres prewoven into the fabric (at intervals of 10mm by 10mm) by the manufacturer allowed

![Figure 8.7 Influence of heating time $t_h$ on temperature difference between defects and HEL wires on the composite surface with a power input of 0.17W/cm².](image)
Chapter 8. Heat Emitting Layers for enhancing NDE

Figure 8.8 Influence of wire spacing on the temperature difference on the top surface with the same power input (0.17W/cm² for 10s). a) 2.5mm spacing and b) 10mm spacing.

Figure 8.9 A schematic showing the lay-up of the laminate including the hybrid layer.

straight lines of metal wires at equal distances to be stitched. Due to the obvious labour intensity of this production process, laminate dimensions were limited.

Teflon inserts have been routinely used by researchers to simulate delaminations [6, 14, 15]. However, the processing temperature of PPS is just below the melting point of Teflon. Therefore, it was necessary to use another material with a higher melting point, and chemical inertness over the required temperature range, as an insert. Rectangular pieces of 125µm PI foils were found to be more than acceptable. Six and twenty four ply laminates, corresponding to thicknesses of 1.6mm and 5mm respectively, were produced with the HEL placed within the stack. The copper busbar was placed at the ends of the HEL to provide the electrical connections for resistance heating. Figure 8.9 shows an expanded schematic example of the lay-up.

A picture frame mould of dimension 200mmx100mm was used to prepare the lay-up, which was then consolidated in a hot platen press, under the processing conditions described in Chapter 7.

Three variables were investigated in this study: the position of the HEL with respect to laminate depth, the metal wire spacing within the HEL and the laminate thickness. The laminate lay-ups and details are described in Figure 8.10 and Table 8.2.
8.4 Experimental technique

![Figure 8.10](image_url) Schematic lay-up of laminates with embedded inserts, where G - glass prepreg layer, H - HEL and PI - polyimide.

<table>
<thead>
<tr>
<th>Name</th>
<th>Variable</th>
<th>wire spacing (mm)</th>
<th>Insert size (mm)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Wire separation, S</td>
<td>2.5-10</td>
<td>100x20</td>
<td>1.6 (6 plies)</td>
</tr>
<tr>
<td>B</td>
<td>HEL position, V</td>
<td>5</td>
<td>50x20</td>
<td>1.6 (6 plies)</td>
</tr>
<tr>
<td>C</td>
<td>Laminate thickness, S</td>
<td>10</td>
<td>50x20</td>
<td>5.0 (24 plies)</td>
</tr>
<tr>
<td>D</td>
<td>Laminate thickness, S</td>
<td>5</td>
<td>50x20</td>
<td>5.0 (24 plies)</td>
</tr>
<tr>
<td>E</td>
<td>Insert size</td>
<td>10</td>
<td>50x5</td>
<td>5.0 (24 plies)</td>
</tr>
</tbody>
</table>

Table 8.2 Laminate details.

8.4.2 Case studies

A small-scale structure of an aircraft wing leading edge was prepared. Two aspects could then be assessed: the effect of the manufacturing technique on the reliability of the HEL/busbar configuration, and the effect of curvature on HELTHY. A four-ply laminate was prepared, in the same way as with the flat laminates, with a row of defects placed between each layer, the schematic of which is shown in Figure 8.11(a). The metal wires were stitched 5mm apart. The thermoplastic rubber forming technique [16, 17] was employed to produce the desired shape. It was thought that the copper mesh busbars would be damaged during the fast forming of the material, therefore they were replaced by nickel-coated carbon rovings, which were stitched through the metal wires as shown in Figure 8.11(b). Figure 8.11(c) shows the finished leading edge section.

![Figure 8.11](image_url) Leading edge details: (a) schematic of all the defects with dimensions, (b) positioning of a nickel-coated carbon fibre busbar, and (c) the thermoformed leading edge section.
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Figure 8.12 Cylindrical pipe details: (a) schematic of all the defects with depth position and dimensions, (b) positioning of the defects, and (c) the finished pipe section.

The second case study was a composite pipe. A 100mm internal diameter pipe was filament wound over a stainless steel mandrel. Glass fibre rovings were wet-wound with an epoxy matrix to create a pipe with an overall thickness of 9.0mm. Three layers of glass fibres were initially wound before the HEL complete with electrical connection was placed in position. A series of 200µm thick Teflon inserts with dimensions varying between 20x20mm and 5x5mm were placed at different depths over the part during the winding process, shown in Figure 8.12(a) and (b). Finally, the product was cured and post-cured according to the supplier’s specifications. Figure 8.12(c) shows the finished pipe section.

8.4.3 Method

A power supply with the range 0-3A and 0-30V, was used to provide power to the HEL. The specimens were suspended in air to allow free convection of heat from both front and back surfaces. To reduce thermal noise from the external environment, the samples were placed in a closed chamber with only one window open for inspection. The samples were connected at the busbar ends and the power adjusted
8.5 Results and discussion

8.5.1 Influence of wire spacing

Figure 8.14 shows the thermal images taken of Laminate A, where the wire spacing was varied. The laminate was examined from both sides to show the dependence of the sensitivity of inspection on the position of the HEL, which will also be discussed in the following section. The simulations had shown that there was not a significant increase in $\Delta T$ when smaller wire spacing were used, and hence that the inspection capability would not necessarily increase. The images obtained indeed verify that regardless of the wire spacing, the temperature difference between that of the metal wire and where the insert was placed was similar, indicated by similar variations in colour (i.e. temperature). However, it is also clear that smaller wire spacings produced a more defined view of the inserts because the contrast between the metal wires and the inserts was more obvious.

There was also a difference in sensitivity depending on where the HEL was placed in comparison with defects and the detector. In Figure 8.14(a) the HEL was on the 4th ply in contrast to Figure 8.14(b) where the HEL was on the 3rd ply (counting from the bottom-most ply). The simulation in Figure 8.5 predicts that the temperature difference on surface is not the same on both sides. This result underline the importance on the HEL placement within the structure. This aspect will be analysed further in the following section.

8.5.2 Influence of HEL position

Figure 8.15 shows the thermal images taken with the HEL closest to, in the middle of, and furthest from, the camera respectively. For this purpose, Laminates A and B were used because the inserts were placed in the same vertical position within the laminate. All images were taken at around 3s of input power, with the same power per unit area.

The simulations performed had shown in Figure 8.5 that when the HEL is in the top layer with the defect placed next to it, the greatest $\Delta T$ values are obtained. Then $\Delta T$ quickly reduces through-the-thickness and the defects placed furthest from this layer would therefore be the hardest to detect. As Laminate B showed, Figure 8.15(a), the furthermost defect was not detected. Placing the HEL for adequate heat-up of the samples without heating past the maximum service operating temperature of the matrix, defined as $T_g$-28°C [18], where $T_g$ is the polymer glass transition temperature. The infrared images were taken using a FLIR ThermoVision A40-M camera and the results are shown in the form of a temperature map of the surface of the specimen. The HELTHY setup, without the specimen chamber, is shown in Figure 9.8.
closest to the inspection surface resulted in the poorest overall detection, predicted by the simulations, for the heating time used.

Positioning the HEL deeper within the laminate allowed for easier, and clearer, defect detection, as was seen with the other two configurations, Figures 8.15(b) and (c). The simulations had shown that both the middle and bottom HEL positions produced very similar ∆T values but there was a difference for the time needed to reach the values. The thermal images obtained showed that for the heating time used, the clearest image was obtained when the HEL was at the bottom of the laminate, furthest away from the camera. The images taken of the middle configuration displayed all three defects but were less sharp.

In addition, the effect of the defect position in relation to the HEL was also evident. From all the images, defects placed above the HEL produced a different thermal signal to that placed beneath and with this information, positions of the defects in relation to the HEL could be deduced.

8.5.3 Influence of heating time

Figure 8.16 shows the thermal images taken of Laminate B at different heating times for the same input power settings. Two cases were considered; the HEL placed close to the inspection surface, Figure 8.16(a), and the HEL placed on the opposite side to inspection, Figure 8.16(b).

All three inserts could be seen after a heating time of 3s when the HEL was placed close to the
8.5 Results and discussion

8.5.4 Influence of increased laminate thickness

The results for the thin laminates had shown that clear and faster inspection was obtained with the HEL placed in the bottom layer of the laminate. Therefore for the thick laminates inspected, all measurements were taken with the HEL positioned away from the inspection side. Figure 8.17 depicts the thermal images obtained from the thicker laminates shown at two different times. The first image is when the inserts can initially be detected. The second is the time before which the image becomes blurred due to heat diffusion and the inserts cannot be further visualised. The time range therefore provides an "inspection window" for a given input power.

Figures 8.17(a) and (b) are the thermal images of Laminates C and D respectively and differ only in the metal wire spacing in the HEL. The difference between the images is very little, and the furthest insert detect from the inspection side was at the middle, placed at 2.9mm from the surface. Unlike with the thin laminates, the clarity of the inserts was not enhanced with a closer wire spacing. This suggests that there is a relationship between the minimum wire spacing and laminate thickness for acceptable defect contrast. The images show that for the powers used, the times to gain the same inspection images were very different. The time required to gain the first image with visible inserts was ten seconds less for Laminate D, where the wire spacing in the HEL was 5mm. A faster, more uniform heat distribution within the HEL was obtained with a closer wire spacing and a faster heat front through the thickness resulted. The effect was also seen in the time range, which was shorter for Laminate D by around seven seconds.

Inserts with a smaller width were used in Laminate E and the resulting images are shown in Figures 8.17(c). Similarly the furthest insert detectable was at the middle but there was a difference in clarity between the top-most and middle insert. The simulations had shown that when the HEL is placed in the bottom, the greatest $\Delta T$ is obtained when the insert is in the middle of the laminate. While the thinner laminates did not show this discrimination, the difference is clearly evident in all the thicker laminates, where the $\Delta T$ values are greater and hence more detectable. The time for first inspection was almost the same as that of Laminate C, which had the same wire spacing in the HEL layer. However, the top-most insert was detectable over a very short inspection time, as the thermal signal was weak due to the small width dimension.
8.5.5 Influence of input power

Laminates B and C were used to determine the influence of input power on the sensitivity of HELTHY and the thermal images are shown in Figure 8.18. The figure displays the images of Laminate B examined over the power range 0.02-0.19W/cm$^2$ and Laminate C over the range 0.13-0.42W/cm$^2$, which were the power ranges where at least one of the inserts were the most clearly visible. At the lowest input powers investigated, only two of the inserts could be found in Laminate B and one in Laminate C. As the power was increased, all three became visible in Laminate B and two in Laminate C, as the contrast between the metal wires and the inserts increased. Note that the surface temperatures were all kept far below the glass transition temperature of PPS. The images show that, as with heating time, for the two laminate thicknesses examined HELTHY is tolerant of the input power.

8.5.6 Case studies

Figure 8.19 shows the thermal images for the leading edge with the depth of the defects increasing from left to right. Two views are displayed to show the inserts as clearly as possible. The smallest inserts could not be found due to two reasons. The first was that the insert size was at the limit in comparison with the wire spacing. Secondly, and more importantly, it can be seen from the thermal images that the metal wires were wavy, which created noise in the detection process and reduced sensitivity. The larger inserts could be found at all depths despite the wire waviness, which are seen as darker patches on the thermal images. Despite the acute viewing angle, the largest inserts could still be seen in the top-view, Figure 8.19(b), indicating that this highly curved geometry does not prevent the detection capability.

Finally, the thermal images obtained of the pipe section are shown in Figure 8.20. The largest insert could be found at all depths up to 8.25mm from the inspection surface, and all inserts could clearly be seen up to 5.25mm depth. However, from the images, particularly Figure 8.20(c), there appears to be poor uniformity of the thermal signal measured by the infrared camera. The reason was due to the highly reflective surface of the epoxy pipe, which is a major source of noise during the inspection process.

8.6 Conclusion

A new concept using hybrid fibre reinforced polymers with infrared thermography was presented as a potential method for non-destructive evaluation (NDE) of composite structures. The main outcomes were as follows:

- The accordance of the simulations to the experimental results show that if material parameters are known, models can be created to accurately predetermine the necessary heating time and power, for each defects size expected.

- Smaller fibre spacings did not improve inspection capability as temperature differences between the fibres and the surroundings were increased. However, once a defect was found, a more defined view could be obtained.

- Positioning the HEL deeper within the laminate allowed for clearer defect detection due to a more even distribution of heat through the thickness of the laminate.

- The concept proved to be very tolerant of input power and inspection time giving early signs that the technique is relatively robust.
Furthermore, two case studies were prepared that showed the application of HELTHY to more complex geometries and displayed the benefits of a thermographic technique where heat is generated from within.

Bibliography


Figure 8.16  Thermal images of Laminate B using a power setting of 0.18W/cm².
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(a) Laminate C (P = 0.12W/cm$^2$)

\[ t = 30s \quad t = 47s \]

(b) Laminate D (P = 1.39W/cm$^2$)

\[ t = 20s \quad t = 30s \]

(c) Laminate E (P = 0.78W/cm$^2$)

\[ t = 28s \quad t = 60s \]

Figure 8.17 Thermal images of the 5mm thick laminates
Figure 8.18  Thermal images of Laminates B and C at different power settings
Figure 8.19  Positioning (above) and thermal images (below) of each leading edge section.
Figure 8.20  Thermal images of the pipe section with inserts
Chapter 9

HEALTHY applied to resistance welds

9.1 Introduction

Joining of thermoplastic composites can be a critical step because it can initiate a number of irregularities in the structure resulting in weakening of the properties. Traditional joining methods for metals and thermostets (mechanical fastening and adhesive bonding) are feasible, but not ideal for thermoplastic composites. Mechanical fastening has a number of disadvantages: introducing stress concentrations in the material, delamination during drilling, different thermal expansion of the fasteners relative to the composite, weight increase and extensive labour and time requirements. Adhesive bonding also presents some difficulties. It requires extensive surface preparation, is generally difficult to control in industrial environments, and adhesives used (typically epoxies) have long curing cycles.

Fusion bonding is a joining method that uses the property of thermoplastic polymers to diffuse when heated above their glass transition temperature $T_g$ (for amorphous polymers) or their crystalline melting point $T_m$ (for semi-crystalline polymers) and regain their mechanical properties after cooling below this temperature. Known also as welding, it can be generally described as joining of two parts by fusing their contact interfaces, followed by cooling (consolidating) under pressure that enables the bond to be made.

One fusion bonding technique, resistance welding, is considered to have great potential for future applications [5]. In fact, the process has already been established in the joining of ribs to the skin of the Airbus A340 and A380 wing leading edges (J-nose), Figure 9.1. In order to melt the thermoplastic matrix, resistance welding utilises an electrically resistive heating element placed at the weld line for local heating.

As yet, there is not a well-defined way to inspect the quality of the weld during service. An interesting solution may be to apply HEALTHY to resistance welds using the heating element itself as the heat emitting layer. This chapter describes HEALTHY applied to two aerospace grade thermoplastics with both carbon and glass fibre reinforcements. A comparison was made to a pre-existing thermographic technique and ultrasonic inspection used to provide reference images.

9.2 Background

9.2.1 Resistance Welding

Resistance welding is one of the most attractive welding techniques for thermoplastics today. It is a rather simple method that uses electrically resistive implant, known as the heating element, sandwiched between the bonding surfaces of the laminates to provide the necessary heat to the joint. The principle
Chapter 9. HELTHY applied to resistance welds

Figure 9.1  Welded thermoplastic composite J-nose for the A340 and A380 airliners. J-nose part(left) and welding assembly(right) [6].

(a) carbon fabric heating element (b) stainless steel mesh heating element

Figure 9.2  Lap shear strength (LSS) comparisons for different heating elements in carbon/PEI at a range of input energies [9].

of the resistance welding process is described in more detail elsewhere [5, 7, 8]. The welding pressure that has to be applied during the whole welding process enables intimate contact between the laminate surfaces and promotes molecular diffusion in the interface. As a result, the heating element remains trapped in the joint. During consolidation, it supplies the necessary welding energy to the joint and is one of the main contributors to the joint quality and controllability. Carbon fibre heating elements have been used almost exclusively in the literature. The attractiveness of the carbon fibre lies in its compatibility with the reinforcing material of the laminates in cases when carbon fibre reinforced materials are used. The fact that the heating element remains in the bond after the welding makes a carbon fibre heating element the obvious choice for the investigators. However, based on our tests [9] and from those of literature [10], stainless steel meshes are proving to be a more practical heating element, with larger processing windows, higher average strengths and less sensitivity to variations in process parameters. As an example of the comparison, Figure 9.2 shows the lap shear strength values obtained with a carbon fibre fabric and a stainless steel mesh used as heating elements in carbon/PEI welds. Stainless steel meshes were also used as heating element materials in the resistance welding of the Airbus A340 J-nose. However, there are drawbacks to resistance welding. At the manufacturing stage, there is the possibility of heating element movement in the laminates and of uneven heating due to edge effects [5].

During service life, the composite welds are subject to cyclic loads. Thus, the fatigue behavior of
9.2 Background

(a) Unidirectional weld  
(b) Quasi-isotropic weld

Figure 9.3  Cross section fatigue delamination in resistance welded joints [11].

these joints has been reported to be related to the composite lay-out [11]. Failure of the unidirectional specimens occurs at the weld interface while failure of the quasi-isotropic specimens occurs either at the weld interface or in the skin laminate as it is illustrated in Figure 9.3. In addition, there is the possibility of corrosion of the metal mesh [12], which would lead to disbonded regions. This situation could also arise when carbon fibre is used as reinforcement because electrocorrosion may occur in the joint. Therefore, there is a necessity to adequately assess the welded joint after the manufacturing process and during its service life.

9.2.2 NDI and Lockin Thermography

Ultrasonic methods, such as c-scanning, are most commonly used for NDI and are extremely effective in detecting, and extracting information about, defects during manufacturing [13]. However, the inspection of complex welded shapes or assembled structures is a much more difficult task due to the two sided access required by the c-scan technique. Realistically if this technique is to be used while the structure is in service, there are few situations in which access to both sides are available for inspection. Therefore, in this study all inspection is performed from one side only, in the reflection mode.

Infrared thermography is a full-field, non-contact method that allows through-the-thickness inspection by analysing surface temperatures with an infrared detector. A special type of thermography is lockin thermography (LT), where lockin refers to the time dependence of an output signal to an input reference signal. With this method, the object receives a modulated external excitation, typically, for example, using a sinusoidal heat input from a high power lamp, so-called photothermal LT. Thermal waves are generated beneath the surface of the specimen, which propagates through the thickness of the object due to thermal conductive processes. Where heat propagation parameters change, i.e. at inhomogeneities, part of the thermal wave is reflected and interferes with the surface waves. This interference can be detected using an infrared camera, after which a post-processing module compares the input modulated signal to the output signal measured on the surface of the specimen. Figure 9.4 schematically describes this process. The results are displayed in terms of amplitude or phase differences and a thermal map of the specimen surface can be made in which defects through-the-thickness may be visualised.

There are a number of advantages of LT compared to other thermography techniques [14, 15], and therefore has been chosen for this investigation. However, it has been shown [16] that the inspection capabilities of LT depends on a number of parameters such as surface properties, material properties and geometrical configuration.
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9.2.3 Heat Emitting Layers for Thermography

The most efficient way to perform a thermographic inspection is to "place" the input signal inside the specimen. The technique for NDI of resistance welds proposed in this study is based on HELTHY where the heating element was used as a heat emitting layer, and the technique will henceforth also be referred to as HELTHY. Two variations of this technique are presented here.

Upon the application of a current, the metal heating element heats due to the Joule effect and produces a thermal front which progresses through the thickness of the laminate. In the case of a defect such as a delamination, the heat front is delayed due to the insulative effect of the trapped air. Using the electrical resistance analogy, the behavior of a unperturbed and perturbed heat fluxes, $HF_1$ and $HF_2$ respectively, are illustrated in Figure 9.5. From the heat transfer point of view, the presence of defects within the composite ($k_m$) acts as an additional thermal resistance, $k_d$, to the heat transfer. Thus, the difference between the heat fluxes can be monitored as a temperature distribution on the surface using an infrared camera as depicted in Figure 9.6(a). It is interesting to mention that there could be special situations where defects should appear as hot spots. For example, when the thermal properties of the defect are higher than those of the composite, as in the case of a misplaced metal strip, or when there is a localised erosion zone. The first HELTHY technique is based on simply mapping the intensity of the thermal signal over the surface of the weld area. The benefit of this method is that real-time processing may be performed, as defects can be detected during the transient heating of the weld.

Whereas LT typically utilises high-power lamps to generate thermal waves within the specimen, the resistive heating properties of the heating element is utilised instead to generate thermal waves at the weld zone, Figure 9.6(b), and forms the basis of the second HELTHY technique. The benefit of this method compared to photothermal LT is that many of the problems associated with photothermal LT are reduced, resulting in a more efficient inspection process. Also, since defects occur at the weld line, it is rational that producing heat within this region would provide clearer images after lockin post-processing.
9.3 Experimental

9.3.1 Materials

Glass fibre and carbon fibre woven reinforced PEI and PPS laminates were used in this investigation. The heating element was a 0.2mm diameter plain weave stainless steel mesh with an opening of 0.86mm. Pieces of 60µm thick polyimide (PI) foils were placed within the weld zone to prevent full consolidation in these regions. Figure 9.7 describes the lap joint geometry and insert dimensions and placement. The defects were placed on top of the welding mesh before adding the top laminate. After consolidation the laminates were painted with matt-black paint to reduce thermal noise due to external surface reflection.

Different thicknesses of laminates were used to investigate the sensitivity and limits of inspection to thickness effects. A thickness range from 1.5mm to 3.9mm was used. The Airbus A340 J-nose, has a skin thickness of 3.0mm and a rib thickness of 1.5mm and therefore, these values can be regarded as realistic for aerospace structures. Table 9.1 gives an overview of the laminates used for resistance welding.
9.3.2 Resistance Welding

A pressure block with integrated electrical connectors for the mesh heating elements was developed in-house to provide simultaneous heating and pressure during welding. A power of 8-9W/cm² was placed on the mesh for 50-90 seconds. A homogeneous pressure of 0.8MPa was applied on the weld zone throughout welding and consolidation through pneumatic cylinders acting on wooden pressure blocks as shown in Figure 9.8. All heating elements were pre-impregnated with either PPS or PEI before being placed in the weld. A thermally conductive coating was applied to the mesh to prevent short-circuiting of the carbon welds.

Table 9.1  Laminate thicknesses used in the production of resistance welds.

<table>
<thead>
<tr>
<th>Laminate</th>
<th>GLASS Thickness (mm)</th>
<th>CARBON Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PPS</td>
<td>1.5</td>
<td>1.9</td>
</tr>
<tr>
<td>PEI</td>
<td>1.9</td>
<td>2.0</td>
</tr>
<tr>
<td>PPS</td>
<td>3.5</td>
<td>3.9</td>
</tr>
<tr>
<td>PEI</td>
<td>3.5</td>
<td>3.5</td>
</tr>
</tbody>
</table>

Figure 9.8  Experimental setup for resistance welding (left) and welded laminates (right).
9.3.3 NDE

In this study, three NDE methods were used: HELTHY, HELTHY with LT and ultrasonic c-scan. HELTHY was performed using a power supply with the range 0-3A and 0-30V, to provide power to the heating element. The heating elements were connected at the exposed ends of the welds and the power adjusted for adequate heat-up of the samples without heating past the maximum service operating temperature of the matrix. In order to measure the surface temperature response of the weld zones, a Cedip Titanium 530L series long-wave infrared camera was used, and the entire sequence of heat-up was recorded. The camera was placed perpendicularly and 0.5m away from the specimen surface such that the whole weld line could be observed.

In order to perform HELTHY with lockin, the same power supply was connected to a lockin module. In this case, the power supply was controlled using a reference step pulse input signal which was then used for lockin post-processing using Altair-LI software from Cedip. The same infrared camera was used to monitor the surface temperature response.

Ultrasonic inspections were performed on the specimens with the aim of obtaining a baseline for further analysis. This technique is especially suited for detecting air pockets and distributed air inclusions, such as delaminations and porosity in laminates. An automated c-scanner (Midas c-scan system 3mx1.5m) was employed to obtain a systematic and full inspection of the flat panels. The system uses a Midas narrowband (10MHz) transducer and dynamic range of 81dB, 0.5mm grid in length direction and 1.0mm index in width direction.

9.4 Results and Discussion

Baseline welds were first prepared to determine the ultrasonic and thermographic images that would be obtained of a clean weld without defects. An example of one of the baseline welds, a 7mm total thickness glass/PPS joint was inspected with c-scan and HELTHY and the resulting images are shown in Figure 9.9. In both images, there is a clear region with the same intensity value running from right to left. This is the weld area and indicates the heating element position. Above and below the heating element, there is a thin strip of a darker shade which indicates the limits of the overlap, where voids have been squeezed out of the weld zone. In this zone, there is little to no matrix material and it is a small air gap of the thickness of the heating element. Few imperfections can be seen in both pictures, the major being the areas towards the extreme right and left of the welds, which are darker shades compare to the rest of the weld. These are caused by edge effects of the heating element, which is a known problem in resistance welding [5]. The edge effect results from poor convection of heat away from the weld zone to the air causing higher temperatures at the weld ends. More squeeze flow of the matrix out of the weld zone occurs, and a poorer bonded region results.

![Figure 9.9](image)

**Figure 9.9** Glass/PPS control weld c-scan (above) and HELTHY (below) images.

After inspection, three images were obtained; c-scan, a thermal map of the weld surface, and
a plot of the temperature profile. In this case, the x-axis of the temperature profile plot was defined along the weld line using thermal pixel coordinates px. The temperature was measured at the mid-line of the weld. The advantage of this type of data display is that an alternative way of obtaining information from the weld is offered, that may not be visible from the original thermal image, and changes in temperature due to the inserts can be more clearly identified. In general, the position of the inserts relative to the heating element and inspection surface affects the change in temperature, namely those that are placed between will result in cold spots and those placed beneath the heating element will result in hotspots.

The HELTHY images shown in the following two sections were recorded after tuning the power settings to a value that showed a large enough thermal contrast without damaging the weld.

9.4.1 Glass Composite Welds

Glass welds with inserts placed to simulate defects were first investigated. The c-scan displays all five of the inserts for both the thin PPS and PEI laminates, as shown in Figure 9.10. The smallest defect on the PPS laminate was not clearly detected, and this was because the delaminated surfaces at the defect were not separated enough to produce a large enough attenuation of the ultrasonic signal for detection. This is known as a ‘kissing bond’, and is a well-known problem for many NDE techniques [21].

The HELTHY thermal image mirrors the c-scans, and clear information about the position of all the defects was obtained. The smallest defect towards the right end of the PEI weld is the least clear because it is masked by the imperfection due to voids resulting from the welding process. The extent of the imperfection is also seen in the c-scan, which begins at the insert placed towards the right of the image. However, the temperature profile clearly shows the position of the inserts, seen as sharp drops in temperature, or valleys on the plot.

The temperature profiles show that, ignoring the defect areas, there is a small variation in temperature across the weld. Particularly clear in the case of the PEI weld, this is typical of the fact that the heating element does not heat the weld exactly uniformly, and the edges of the heating element heats faster than in the middle. Despite this, the defects can still be distinguished.

Figure 9.11 displays the images obtained from the thicker glass laminates. The c-scan reference images clearly define the positions and relative dimensions of the inserts for the PPS. The defects placed in the PEI laminate were masked by the high void density across the weld, which attenuated the signal to the same extent. The void content associated with resistance welding of PEI is already well-known [22].

All defects could be found in both laminates with HELTHY, to varying degrees of clarity. In order to obtain better contrast for visualising the inserts, the applied power was increased with respect to the thinner laminates of the same material. The inserts placed in the PPS laminates could be seen more clearly than with PEI. The PEI weld was not as uniformly consolidated as the PPS weld and was also seen with the thinner laminate. This variance arose from the high viscosity of the PEI matrix at the welding temperature, which restricts polymer diffusion during the application of pressure thereby preventing the movement of some of the voids out of the weld zone. However, the vast majority of the voids are not within the weld, but a few layers within the adherent laminates, as shown in Figure 9.12. The highest density of voids was towards the right of the weld, as shown in the c-scan. The voids did not affect the detection of the defects, as shown in the thermal image and temperature profile, although the peaks, indicating the defect position, were smaller in regions of high void density. Ignoring the defect areas, the temperature across the weld decreases in the same way as the thin laminate. This trend indicates a high void density within the laminate.

An interesting feature, clear in the HELTHY images, is the wavy form of the weld of the PEI
laminate, caused by the heating element which had moved during the consolidation process. The detection of this type of feature can be used as an in-situ quality check after joining to determine any movement of the heating element and further to determine whether it is acceptable.

### 9.4.2 Carbon Composite Welds

Figure 9.13 displays the images obtained from the thin carbon laminates. The c-scan for the PEI weld shows that the consolidation was not uniform and revealed a large area of voids compared to the PPS weld.

All five of the inserts could be found within the PPS weld with HELTHY, along with the waviness of the heating element. Towards the right of the PPS weld, a dense area of voids slightly masks the fourth insert. The voids are also detectable on the temperature profile. The voids were likely created by the movement of the heating element during consolidation, and is seen as a decay in temperature at a px value of around 270, where the void density was high enough to absorb a considerable amount of heat. Despite this, the temperature across the weld in that region remained fairly constant and Figure 9.14(a) shows that the voids in this case were within the heating element.

In the case of the PEI weld, four of the five inserts could be found on the thermal image within the joint, despite the heavy presence of voids, which again was attributed to the masking effect. The temperature profile shows a very small temperature drop at the centre of the weld, for the middle defect. As with the thick glass-PEI weld, the temperature profile generally decreases from 31.3°C to 28.5°C. Again, because the temperature profile was affected in this way, it could be deduced that there was a very high void density at the laminate, as is shown in Figure 9.14(b).

![C-scan and Thermal image](image)

**Figure 9.10** NDE images of 1.5mm glass-PPS (left) and 1.9mm glass-PEI (right) laminates.
A higher input power was required for the thin carbon samples than the thin glass samples. In general, the carbon laminates required significantly higher power than the glass laminates. Unlike glass, carbon fibres have a significantly higher thermal conductivity value in the longitudinal direction along the axis of the fibre than in the perpendicular direction [23]. Hence when the weld was heated from within, carbon conducts the heat away from the laminate at a much faster rate, and using too little power resulted in a weak heat flux across the joint. This inconvenience is solved by increasing the inspection power. Nonetheless, the maximum power used for inspection, 3.8W/cm$^2$ was well below the power used for resistance welding 8-9W/cm$^2$ and thus in none of the cases had the weld integrity been affected by HELTHY.

Figure 9.15 displays the images obtained from the thick carbon laminates. The c-scan was able to detect all five inserts in the PPS laminate. Four inserts were found in the PEI laminate, as well as the high presence of voids across the entire weld area. The second insert was not found and it is likely that it was a kissing bond.

All inserts could be seen in the thermal image for the PPS laminate, and are indicated as peaks on the temperature profile plot. The c-scan shows that there is a thin strip of voids running along the mid-line of the weld and a micrograph taken of the cross-section within this region shows that the voids are present mostly in the weld, Figure 9.16(a). A variation due to the presence of these voids is not visible on the selected temperature profile. However, the voids can be visualized with a transversal temperature profile.

Although the c-scan image did not detect all inserts, the presence of voids in the PEI laminate, as shown in Figure 9.16(b), did not affect the detection capability of HELTHY. All five of the inserts could be found on the thermal image and are indicated as valleys on the temperature profile.
9.4 Results and Discussion

Figure 9.12  Micrograph of 3.5mm glass-PEI weld showing void distribution close to the heating element. Cross-section was taken from the center of the weld at defect position 4.

9.4.3 Influence of Heating Time and Input Power

The 1.9mm carbon/PPS laminate was chosen as an example to show the influence of transient heating time and input power on inspection. Two input powers were used, 0.95W/cm$^2$ and 1.73W/cm$^2$ shown in Figures 9.17(a) and (b) respectively, with the thermal signal captured at different heating times.

After one second, the temperature profiles do not show an appreciable difference between the area of defects and the rest of the weld in both cases. Despite this, the inserts could still be detected when a higher power was used. There is a minimum energy required to provide a detectable temperature difference between the inserts and the surrounding area for a given time, as it was illustrated in Figure 9.5.

As the heating time increased, the temperature difference increased for both power settings. It is therefore clear that HELTHY is tolerant to heating time and power. It is important to note that the limiting factor is the technique is the sensitivity of the infrared detector used for inspection.

9.4.4 HELTHY with LT

In an attempt to derive more information from the weld, HELTHY was coupled with a lockin module to produce a phase map of the weld surface. Figure 9.18 shows the results of the images and phase plots obtained for the 1.5mm glass/PPS weld at different inspection frequencies. The inserts in the joint are seen here as peaks on the phase plots in contrast to the valleys in previous temperature profiles. The difference in shape is due to the position of the inserts with respect to the heating element and inspection surface as it was mentioned before. This approach has been chosen intentionally in order to present both analysis possibilities.

As with the thermal images obtained with HELTHY, the phase images for the thin glass weld clearly show the positions of all the inserts. In addition, the dimension of each insert can clearly be seen, information that was not available with the temperature profile plot. The inserts can be seen on the phase plot, with less noise evident compared with the temperature profile. Frequency did not influence the quality of inspection, and this was most likely because even the highest frequency used was able to penetrate the entire 1.5mm thickness of the weld.

9.4.5 Further Comments

It has been stated in Section 9.2.2 that single-sided inspection would be advantageous for inspection of resistance welded structures that are in service. Considering HELTHY, the technique is not strictly one-sided because access to the meshes is required for resistance heating. This can, of course, be
9.5 Conclusion

HELTHY was extended to non-destructive inspection of resistance welded composite joints, where the embedded heating element was used as the heat source. Flat glass and carbon thermoplastic composite welds were manufactured and studied. Ultrasonic inspection was used as a reference to evaluate the technique, also for comparison and verification of the position of inserts placed at the weld. The following observations were made:

- The reinforcement fibre type influenced the inspection capability more than the matrix type, as carbon has anisotropic thermal behaviour, which is not present with glass.

- The difference in input power between PPS and PEI polymers was attributed to the highly dense presence of voids, which also affected the defect detection capability of the c-scan. The voids
9.5 Conclusion

Figure 9.14  Micrographs of the welds showing void distribution. Cross-sections were taken at the higher void density zones indicated by the c-scans.

(a) 1.9mm carbon-PPS  (b) 2.0mm carbon-PEI

Figure 9.15  NDE images of 3.9mm carbon-PPS (left) and 3.5mm carbon-PEI (right) laminates.

acted as insulators, absorbing thermal energy requiring more input power to transmit the heat. Nevertheless, the presence of voids within the laminate can be detected using the temperature profile plot in most of the cases.

- As in Chapter 8, HELTHY proved to be tolerant of input power and heating time.

As a final note, this inspection method can be extended to any fusion bonding welding process where different thermal activation methods can be used to melt the joint interface.
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(a) 3.9mm carbon-PPS  (b) 3.5mm carbon-PEI

Figure 9.16  Micrographs of the welds showing void distribution. Cross-sections were taken at the higher void density zones indicated by the c-scans.

Bibliography


Figure 9.17  Influence of heat time and power on inspection capability of the 1.9mm carbon/PPS laminate.


Figure 9.18 HELTHY images taken with the lockin module for the 1.5mm Glass/PPS welds (P=0.32W/cm²).


Chapter 10

Integrated ice-protection for composite aircraft structures

10.1 Introduction

Throughout aviation history, icy conditions have been one of the major weather hazards encountered by aviators. In the right conditions, ice accretion on aircraft structures is an unavoidable occurrence both on the ground and in flight. Usually, the ice forms on engine inlets and aerodynamic leading edge surfaces. At the lowest level of interference with the aircraft, ice causes lift and drag penalties thereby reducing aerodynamic efficiency [1]. At the highest level, severe ice accretion can lead to a change in flight dynamics, loss of control and ultimately to the aircraft crashing [2, 3]. A review of the icing phenomena has been presented by Kind et al. [4]. To limit or protect flying machines against ice accretion, anti-icing (the prevention of ice accretion) and deicing (the removal of accumulated ice) methods have been developed for in-flight and ground purposes. Four types of ice protection systems (IPS) have been used with varying degrees of application and success. These methods are chemical, mechanical, thermal and hybrid technologies and are described in Table 10.1.

The most common IPS are pneumatic, bleed-air and electro-thermal techniques. Pneumatic is a mechanical IPS and uses several inflatable bladders or pneumatic boots bonded onto leading edge surfaces. The system is activated periodically breaking-up the ice. The pneumatic boots are energy efficient but the ice debris can damage engine or aircraft components. In addition, ice can form behind the leading edges where the boots are ineffective [5] and the presence of an ice layer before the activation of the system imposes an aerodynamic performance penalty [6].

The most well-developed IPS technique in airliners relies on the use of hot bleed air from engine compressor stages. The hot air is siphoned off and led through piping to vulnerable areas and expelled through small holes towards the inner surface of the aircraft skin [7]. The outer skin surface is then heated by thermal conduction. However, bleed air extraction decreases engine performance as net thrust is lowered, and the extensive piping network adds weight and maintenance problems. Since the area of heating is limited, there is a risk of ice runback and refreeze behind protected areas, which ultimately affect aerodynamic performance. Most importantly, with improvements in engine design and efficiency, there is less available high-pressure air to be used by conventional bleed air IPS.

Electrothermal IPS have been used in areas that bleed air ducts cannot reach such as nose cones. These heater pads are simply bonded or incorporated to the aircraft surface where required [8]. As current passes through the pads, they heat through resistance heating, warming the surface and melting the ice. Figure 10.1 shows a typical lay-up of an electrothermal heater [5, 8]. The major drawback of
this system is the power consumption necessary for adequate heating.

With changing aerodynamic, engine designs, and operation conditions, systems that have worked in the past, such as bleed air and pneumatic boots, do not fit with new highly efficient, highly streamlined aircraft structures as those depicted in Figure 10.2. In addition, as the challenge of aircraft 'lightness' results in the tendency towards newer alloys and composites, aircraft systems must also be streamlined to the changes in design and materials. Thus, the pipeless electrothermal heaters are proving to have the most potential in the future of IPS, and they are being chosen for the new aircraft generation.

In the quest for more affordable aircraft, fuel-efficient engines, light-weight structures, all-weather
operations and more optimal aircraft systems implies there is a clear necessity to look for suitable technology alternatives for IPS. The impetus behind this work is to investigate the potential of a fully integrated multifunctional anti/deicing structural material based on electrothermal heating. Glass fibre reinforced polymer composites were used as the base material with embedded metal wires providing the source of heating. The hybrid layer was placed within a ply stack and fully consolidated into the laminate. In this study, the electrical properties of stainless steel wires were utilised to provide internal heat energy through resistive heating. The concept of this hybrid arrangement is described followed by an experimental investigation into the anti/deicing performance of the hybrid layer. Next, the effect of thermal cycling on the hybrid layer is described. Finally, the production method to create a demonstrator is described.

10.2 Materials and Experimental Method

10.2.1 Heat emitting layer

The IPS concept utilised a heat emitting layer (HEL) comprising of woven glass fabric with unidirectional interwoven electrically resistive metallic fine wires, in this case stainless steel, placed at a specified distance apart. The same glass fabric could be used for the HEL as the rest of the laminate. Busbars were placed perpendicularly across the resistive wires at each end of the hybrid fabric allowing for an electric current to be transferred to the resistive wires, in the same way as that described in Chapter 8.

10.2.2 Sample Preparation

Glass fibre/polyphenylene sulphide (PPS) prepregs, 8-harness satin weave glass fabric and PPS foil were all used as the base materials. For the HEL, AISI 316 type stainless steel, 70µm diameter, was used due to its excellent corrosion resistance and deformation (forming) properties. The HEL layer was made by hand weaving the stainless steel wires (SSF) into glass fabric at intervals of 5mm. The presence of blue glass fibres prewoven into the fabric (at intervals of 10mm by 10mm) by the manufacturer allowed straight lines of metal wires to be stitched at equal distances.

The material selection for the busbars was defined considering the forces that the product will suffer under forming. Thus the materials chosen were nickel-coated carbon fibres, copper wire (270µm diameter) and copper mesh (56µm diameter fibres finely plain woven).

Four-ply laminates were produced with the hybrid layer placed in the 2nd layer. A frame mould was used to prepare the lay-up, which was then consolidated in a hot platen press. The lay-up was heated under a pressure of 1 bar until a temperature of 320°C, when the pressure was increased to 10 bars and held at the same temperature for 20 minutes. The laminate was then cooled to room temperature. For the icing experiments, several panels of 10cm x 20cm were produced.

10.2.3 Thermal analysis

Thermocouples were placed at various positions on the surface of the laminates and used to log the temperature profile during the anti/deicing experiments. In order to analyse the surface temperature distribution on the composite panels and the viability of the busbars, an infrared camera FLIR ThermaCAM SC2000 system was also used. The camera has an uncooled long wave focal plane array (320*240 pixels). The thermal activation of the HEL system was performed using a Delta Elektronika power supply connected to the busbars.
10.2.4 Icing experiments

Static icing conditions have been commonly used to provide initial proof-of-concepts for IPS [15, 16], and was also used in this investigation. A small climate chamber cooled with liquid nitrogen was used for the icing experiments. The system is shown in Figure 10.3a. Nitrogen is injected into the chamber (2) by a computer-regulated machine (1). At several points on the panel, thermocouples were placed on its surface to measure the temperature during the process. The data were acquired with an interface (3) and sent to the computer (4). The HEL was activated by an external power supply unit (5).

Figures 10.3(b) and (c) shows the arrangement of the laminate inside the climate chamber. Four thermocouples were placed at various positions on the laminate, labelled T1-T4 in the figure. The laminate was clamped in a free-standing fixture such that the cooled air could flow above and below. Water was dropped onto the laminate using a pipette through an opening at the top of the chamber either before chamber cooling (for anti-icing) or after (deicing). A barrier was placed around the edge of the laminate to contain the water, which was set at 80ml volume corresponding to roughly 5mm of ice thickness. The chamber was then cooled to -50°C either after activation of the HEL (anti-icing) or preceding activation (deicing).

10.2.5 Thermal cycling experiments

The CTE value of the PPS is significantly greater than that of the stainless steel wire, at 50µm/m·°C and 17µm/m·°C respectively. Therefore when the wire heats, the volumetric expansion/shrinkage of the surrounding matrix would be significantly greater. Repeated expansion and contraction below the $T_g$
could potentially result in two types of defects occurring: debonding of the wire/matrix interface and
matrix cracking [17]. If the concept IPS is to be installed in all-weather aircraft, a rough estimation of
the number of times the IPS would be activated could be assumed to be around 10000 (once a day over
27 years). In addition in reality, relative humidities of up to 100% would also be encountered. At the
microscopic level, the water absorption may also affect the fibre/matrix interface initiating debond. A
crude experiment was devised to provide an idea of the effect of thermal cycling and humidity on the
fibre/matrix interface and the matrix itself.

Five sets of three 16mm length metal wires were stitched into glass fabric at a spacing of 5mm
within the sets and 20mm between each set. Copper mesh busbars were placed at the wires’ ends. The
glass layer was then placed in the second layer of a four-ply stack, as with the laminates described in
Section 10.2.2. The laminate was placed in a climate chamber, and connected to a computer-controlled
power supply. The computer also recorded the temperature of the laminates through thermocouples
placed on top of the metal fibres. Figure 10.4(a) shows the schematic arrangement of the experiment.
At the start and then during the one month period, pieces of the laminate were cut using a diamond
saw, as each of Cycles 1-4 were reached, defined in Table 10.2. The pieces were examined using scanning
electron microscopy (SEM) and were examined for signs of damage such as wire/matrix debonding
and matrix cracks. Figure 10.4(b) shows the laminate at the start of Cycle 3 where the position and
nature of the cut-outs can be seen. Two pieces were cut per cycle, and each piece contained three
metal wires.

Keeping the chamber temperature at a constant 22°C and 100% humidity, power was applied to
the HEL until a maximum temperature was reached and then turned off to reduce the temperature
to ambient. The peak cycle temperature was chosen to be 60°C so as not to heat past the maximum
operating temperature of the matrix. The minimum temperature was set at 30°C to reduce the overall
cycle time. Cooling the chamber to more realistic temperatures required much energy and time, and by
using a higher temperature, the entire 10000 cycles could be performed over a period of one month. For
the tests to be valid, the CTE values should be constant between the minimum temperature used for
the icing tests and the maximum temperature of the thermal cycling experiments, i.e. -50°C-50°C. It
is already known that the CTE of stainless steel and PPS [?] is roughly constant over this temperature
range, which rendered the test valid. Table 10.2 summarises the conditions used in the thermal cycling
experiment.

In order to keep the same electrical conditions, the power was monitored and adjusted after each
cut-out such that the current would remain constant. Table 10.3 gives an overview of the electrical
settings used during each of the cycle periods.

<table>
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<td>Relative humidity (%)</td>
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<td>Cooling period (s)</td>
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<tr>
<td>Cycle 4</td>
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Table 10.2  Thermal cycling conditions
10.3 Results and Discussion

10.3.1 Busbar analysis

Three GF/PPS flat panels with dimensions of 10cm x 20cm and four plies were manufactured. The HEL was placed in the 2nd layer, and all laminates were activated at room temperature with the same input power. The busbar configurations for the nickel-coated carbon fibre bundle, copper wire and copper mesh and their infrared images are shown in Figure 10.5. The images show that all busbar materials provide good connection to the HEL, resulting in equal heating of the metal wires. However, Figure 10.5(a) shows that there is evidence of considerable heat loss from the nickel-coated carbon, at the points of connection between the metal wires and the busbars. The copper wire and mesh showed negligible heat losses and good heat distribution of the metal wires, as shown in Figures 10.5(b) and (c).

The difference in heat loss between the various materials arose through contact issues between the metal wires and the busbars. Figure 10.6 shows the two main heat sources that are possible at the busbar. The first is through resistance heating of the busbar itself and as nickel has a slightly lower electrical conductivity than copper, this is a contributing factor. The second, contact resistance heating, arises at the point of contact between the busbar/wire junction. Considering the nickel-coated carbon, the high processing pressures used during consolidation forced the busbar bundle to spread, resulting in a very high number of contact points, and therefore resistances. Evidence of contact resistance heating can be seen in Figure 10.5(a) at the busbar/wire junctions, shown as "hotspots".

The manufacture of a more complex shape than that of a flat panel defines the requirements of a busbar. Aside from the obvious requirement of electrical conductivity, the busbar should be reliable and flexible such that it can be deformed along with the rest of the HEL into the desired shape without loss of contact, breakage or damage to the surrounding material. Considering a singly-curved shape,
10.3 Results and Discussion

(a) nickel-coated carbon fibres  (b) copper wire  (c) copper mesh

Figure 10.5  Schematic arrangement of busbars (above) and infra-red images after activation of the HEL (below.)

(a) resistance heating  (b) contact resistance heating

Figure 10.6  Heating mechanisms at the busbar/metal wire junction

either wire or mesh material are feasible. Highly doubly-curved shapes would suit wires, as meshes would wrinkle and create local protuberances in structure. A leading edge profile has single curvature, and for reliability a mesh was chosen as this increased the chances that contact with the metal wires would remain even after a fast, high-pressure process such as rubber forming.

10.3.2 Heat distribution

The next consideration of the IPS is to determine a way to adequately distribute the heat of the metal wires over the entire surface area of the structure. Three methods were chosen and tested, based on placing a thermally conductive layer on top of the HEL. These methods were: a carbon fabric layer, carbon and glass fabric layers, and a copper mesh layer. Figure 10.7 shows the infra-red images obtained from the three different laminates.

While carbon fabric is a relatively good thermal conductor, the electrical conductivity of the material resulted in the short-circuiting of the HEL. The current from the metal wires was directed to the carbon fabric, and heating was reduced, as shown in Figure 10.7(a). As a simple solution, glass fabric was introduced in between the HEL and the carbon. Although the problem of short-circuiting was eliminated, heat distribution was poor, as the individual lines of the metal wires could clearly be
seen, as shown in Figure 10.7(b). The glass fabric layer insulated the heat between the HEL and the carbon layer, resulting in a loss of thermal efficiency. In addition, heat that was conducted through the glass layer was then effectively conducted to the surface, due to the excellent thermal conductivity of carbon, resulting in the defined lines of heat. The use of an electrically insulated copper mesh appeared to have been a very good solution, as heat was well distributed over the surface, Figure 10.7(c).

The performance of these surface layers also differed in terms of the time taken to produce the same temperature rise, for the same input power. Whereas the laminate with a carbon fabric surface layer did not heat further regardless of the time, the metal mesh layer heated to the same temperature faster than the carbon layer with a glass insulating layer. Therefore, the coated copper mesh was used as heat distributing thermal layer for the concept IPS.

It should be noted that the ideal solution would be to coat the metal wires themselves with an electrically insulating material such as that used for the copper mesh. In this case, carbon could be used for the HEL and the insulating glass layer removed, producing a much more efficient system. This was attempted, however the delicate process of stitching resulted in the coating being removed.

10.3.3 Icing experiments

The final configuration of the concept IPS was defined for testing and consisted of the lay-up shown in Figure 10.8. A number of anti- and deicing experiments were performed in order to determine the behaviour of the IPS concept under static icing conditions and to define power (per unit area) and time settings. Successful anti/deicing was considered to occur when the surface temperature of the laminate reached 2°C. Of the experiments performed, the four most important test results are shown and discussed in this section.
Deicing

Figure 10.9 shows the results of two deicing tests performed on the laminate. An equilibrium power setting was defined as the power necessary to keep the surface temperature at a constant 2°C, the equilibrium temperature. Figure 10.9(a) shows one of the tests performed to find the minimum equilibrium power setting. Initially the power was set at 0.25W/cm² and four distinct temperature profiles resulted, which depended on the location of the thermocouples on the laminate. The two greatest temperature rises were found in the centre of the laminate, T2 and T4, on both the upper and lower surfaces, where the insulating effect of the ice allowed for a greater temperature rise on the top of the laminate. Outside of the mesh area, on the metal wire, T1, a lower temperature profile was found. Most likely, due to the difficulty of exact placement of the thermocouple on a metal wire, it was placed in the space between two wires resulting in a significantly lower temperature rise.

As expected, the smallest variation was at the busbar albeit with a temperature rise of 20°C from the starting temperature. This indicated that there was an increase in thermal losses at the connections between the busbar and metal wires, compared to that found in Section 10.3.1. The heat loss arose from the increased power required to heat the laminate at the chamber temperature of -50°C. Since the applied current was kept constant, increasing the power led to an increase in resistance of the busbar mesh, from $P = I^2R$, and thus to greater heat loss.

At the set input power, the temperature in the middle of the laminate rose close to 0°C and settled at this value: an equilibrium was reached between the heat produced by the wires and that conducted to the ice. Due to the latent heat of fusion, extra thermal energy was required to melt the ice between the surfaces of the laminate and the ice. This threshold was overcome by boosting the input power until the temperature rose to the equilibrium temperature and then correcting to find the minimum power to maintain this value. Finally, the minimum equilibrium power after the power boost was 0.27W/cm². At the minimum power setting, the time taken to reach a surface temperature of 2°C was 3000s. In order to reduce this time, more tests were performed at higher input powers. Figure 10.9(b) shows one of the tests performed to find the maximum equilibrium power setting achievable with the power supply used, at 0.73W/cm². In this case, rather than placing the thermocouple, T4, on the underside of the laminate, it was instead placed at the limit of the mesh area to determine the temperature variation across this region.

The two greatest temperature rises were those at the mesh, T2 and T4, and the differences between the profiles was small at around 5°C after the equilibrium temperature was reached. The temperature at T4 was due to its position at the edge of the mesh, where the uniformity of the heat distribution was lower than in the middle. The temperature at a metal wire T1, was found to be slightly lower and this was again due to the positioning of the thermocouple.

The lowest temperature profile was again found at the busbar, although the temperature rise was significantly greater than that found at the minimum equilibrium setting, at around 45°C. As before, this was because of the higher input power, which resulted in greater busbar resistance, and therefore temperature rise.

At the maximum power setting, the time taken to reach a surface temperature of 2°C was 170s. During the tests, only the surface temperature was known, which may have differed greatly from the temperature at an embedded metal wire. It was therefore necessary to check for damage of the matrix surrounding the metal wires. Shortly after the equilibrium temperature was reached at the maximum power setting, the power was terminated and the laminate inspected for damage. A cross-section of the laminate was taken at the mid-line of the laminate, perpendicular to the direction of the metal wires, and examined using SEM. Figure 10.10. There appeared to be no evidence of damage, such as degradation or cracking of the matrix, surrounding the wire as shown in Figure 10.10(a). Closer
inspection of the wire/matrix interface, Figure 10.10(b), also showed no evidence of disbonding or cracks.

Anti-icing

Figure 10.11 shows the results of two anti-icing tests performed on the laminate. In these tests, the heating speed of the laminate is less important since the system can be activated before ice accretion. Instead, keeping the equilibrium power to a minimum is more critical.

The first test was performed using the same minimum equilibrium power found during the deicing
10.3 Results and Discussion

Figure 10.10 SEM image of a metal wire after the maximum power setting was applied.

experiments, and shown in Figure 10.11(a). As before there were four distinct temperature profiles resulting from the four thermocouples. The two greatest temperature rises were found at the metal wire and in the middle of the laminate, at T1 and T2 respectively. However, the equilibrium temperature was only reached by the thermocouple placed above the metal wire. This indicated that a higher input power was needed for anti-icing than for deicing, which was due to the absence of the insulating effect of the ice. As the power was applied, the heat from the surface was effectively conducted to the surrounding air meaning that an increase in power compared to the deicing case was required to maintain a higher temperature at the surface.

If the profile of T2 is compared to T4, it is clear that there was a greater temperature difference across the mesh. At this power setting, the temperature at the edge of the laminate settled at -10°C. The difference was attributed to two reasons. The first was due to the absence of insulating ice, which allowed the heat to distribute more evenly over the surface area of the laminate. The second was due to the heat dissipation to the surrounding air at the edges of the laminate.

After a series of further tests, a minimum power setting of 0.41W/cm² was found that allowed the edge of the laminate to reach the equilibrium temperature, and the profiles are shown in Figure 10.11(b). The temperature difference across the laminate had almost doubled with the increase in power, and the surface temperature at the middle of the mesh had reached 20°C. This indicates that for this dimension of laminate, anti-icing is inefficient, as a greater input power is necessary to counteract the heat losses at the edges of the laminate. In real-life, this would be overcome by using a larger HEL area than the region over which ice accretion is expected to occur.

10.3.4 Thermal cycling experiments

The thermal cycling experiment was performed over a period of one month and at various intervals pieces of the laminate were cut and examined using SEM. Over the month, the temperature remained consistently within the 30-60°C temperature range. The power was reduced after each cut to provide the remaining metal wires with the same current, and there were no fluctuations that indicated degradation at the busbar junctions.

The greatest input power used for the icing tests was 0.73W/cm² with an amperage per wire of 0.37A, which was far higher than the 0.16A used for the thermal cycling experiments. The icing tests were performed at -50°C hence greater input power was required to heat the laminate. As the starting temperature of the thermal cycling experiments was about 22°C, the input power was reduced to produce a slower, and therefore more measurable and controllable, temperature rise.

Figure 10.12 shows typical cross-sections of the metal wires taken before and after each cycle.
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(a) at equilibrium power settings for deicing

(b) at an increased power setting

Figure 10.11  Temperature- and power-time curves for anti-icing experiments.

period. Figure 10.12(a) shows a reference wire taken from the laminate before thermal cycling. The metal wire was well-embedded in the PPS matrix and the glass layers can be seen above and below. Fracture of the glass fibres can be seen above the metal wires, which was due to the stitching procedure. Two distinct regions can be seen in the surrounding matrix, indicated by different shades, from the boundary between the PPS foil used to impregnate the HEL and the PPS semi-preg layers.

Figures 10.12(b)-(d) show that there was no change to the wire/matrix interface, nor the surrounding matrix. After Cycle 4 however, cracks in the matrix were found surrounding 2 of the 6 wires inspected. An example is shown in Figure 10.12(e), which shows matrix cracks running radially away from the wire. It is important to note that in neither of the cut samples were cracks seen to have been
10.3 Results and Discussion

10.3.5 Manufacture of a full-scale IPS

Thermoforming has great potential as a manufacturing process for general aviation movable components. The process offers low manufacturing costs, but imposes higher loads into the material, such as maximum pressure and temperature loads that the embedded elements must tolerate, during the...
process. If the IPS is able to survive such a thermoforming process as rubber forming, it will be also
suitable for the same integration capabilities using other manufacturing techniques such as resin trans-
fer moulding or vacuum infusion. Thus as an application case a section of a general aviation rudder
has been manufactured with the integrated IPS concept.

The rudder leading edge of the Enear Eaglet 2-seater recreational aircraft was chosen to be
integrated with the IPS developed in this study. This structure was chosen because the dimensions
and geometry allowed the rudder to be manufactured in-house; the rudder is 1.5m in length and about
0.1m flat width. Figure 10.13 shows the aircraft and the rudder structure. As the rudder is hinged
onto the vertical tail, the leading edge is not continuous and hence two separate heater pads needed
to be integrated.

After dimensioning the heater pads and preparing two HEL layers with wires placed 5mm apart,
the leading edge was prepared with the same lay-up and materials as the laminates used in the icing
experiments. Figure 10.14 shows the overview of the laminate preparation. First a 1.5mm steel mould
was prepared with an opening of 1.1m x 0.5m. Within the mould, two coated meshes were placed on
top of a layer of PPS foil, Figure 10.14(a). Two more PPS foil layers and the HEL with busbars were
placed on top of the meshes. Polyimide-coated copper wire was used as electrical connectors to the
busbars, Figure 10.14(b). A glass layer followed by two carbon layers were placed on top of the HEL
and a small cut made for the connectors to pass through. Finally, as extra insurance against short-
circuiting, small patches of glass fabric was placed around the connector outlets, Figure 10.14(c). The
lay-up was consolidated in a hot platen press under the same conditions as used previously, with the
exception that the peak pressure was reduced to 6 bars to reduce loading of the electrical components
within the layers. Due to dimension limitations of the press, consolidation took place in three stages
along the length of the mould. Figure 10.14(d) shows the consolidated laminate.

A stainless steel negative mould with the correct dimensions of the leading edge had already been
manufactured in-house. The laminate was placed in a blank holder for rubber forming and heated to
320°C under infrared panels, Figure 10.15(a). The laminate was then formed using a rubber die into
a metal mould under a pressure of 100 bars for 20 seconds and simultaneously cooled to 180°C. The
rubber die was removed and the leading edge allowed to cool in the mould to room temperature. Figure
10.15(b) shows the untrimmed product, from the carbon-side, released from the mould and shows the
connectors still intact. Finally, 10.15(c) shows the final trimmed leading edge.

The last stage was to check the activated IPS with an infrared camera, Figure 10.16. At a power
setting of 0.4W/cm² (200W), taken from the anti-ice tests, the time taken to reach 40°C over the
surface of both heater pads took just over one and a half minutes from room temperature. The rather
long period was due to the time needed to conduct the heat evenly over the entire mesh area. Also,
10.3 Results and Discussion

(a) laminate heating (c) trimmed leading edge

(b) untrimmed leading edge

Figure 10.15 Production of the leading edge.

There may have been small losses from the electrical connections within the IPS.

10.3.6 Additional comments

The idea behind the IPS system developed in this investigation used embedded metal wires as a heat source from within the structural laminate. While the outcomes were clearly positive, there were a number of additions that were necessary to make the system work. The IPS was applied to a carbon fibre structure, and to prevent short-circuiting with the metal wires a glass-fibre HEL was prepared and an additional insulating glass layer was used, which ultimately increased weight. Ideally, the metal wires would be coated with a very thin electrically insulative but thermally conductive layer, such as polyurethane, thereby eliminating the glass layers and further reducing the probability of corrosion of the wires. A top layer of mesh was employed to distribute the heat evenly. While the idea of using a mesh layer is not weight-saving, it does potentially offer additional benefits such as lightning-strike and erosion protection, the latter of which is investigated in the following Chapter.

The question also arises of how the IPS developed in this investigation compares to an existing IPS. One of the most efficient electrical heater IPS currently exists of expanded graphite foil that is used in between an electrically insulating glass layer and an thermally conductive outer layer [22]. The system is able to heat an iced surface, during flight, from 1°C to 34°C in 90 seconds at around 0.85W/cm², and claims to require one third of the power of other commercially available heater pads. As an indication, from the crude deicing tests performed in the experiments, a power of 0.73W/cm²
heated the surface from -50°C to 2°C. However, since the conditions of these experiments were static, a direct comparison cannot be made.

As a general note, it is important to ensure that all electric circuit paths for the busbars and wires remain closed throughout the lifetime of the structure. A critical limitation of embedded electrical conductors into composites is the mismatch in fatigue life. Metals typically exhibit significant lower fatigue life than fibre-reinforced polymer matrix composites [23]. For example, for copper, if the stress is kept below 35% of its ultimate tensile stress, the copper will remain adequately conductive for twenty thousand cycles [23].

This investigation has explored an extra application of hybridising metal wires with fibre-reinforced composites; to add a heating functionality to the laminate. While this functionality is aimed as an ice-protection system for the extreme case of aircraft structures, there is potential for other applications such as wind-turbine blades or automotive structural panels. In addition, the IPS system is also well suited to those areas of the aircraft that are subjected to run-back and refreeze.

10.4 Conclusions

A hybrid concept for anti/deicing in composite aircrafts has been investigated, offering a structurally integrated ice protection system. The key was an embedded heat emitting layer (HEL) was utilised to input thermal energy from within the composite structure. The concept was experimentally investigated to develop electrical connections and thermal conductivity and further tested in an ice chamber. Finally a demonstrator was manufactured to show how the system may be integrated. The following observations and conclusions were drawn:

• Copper mesh proved to be very reliable and electrically efficient busbar material. Further, the HEL layer alone was not effective enough to distribute the heat evenly over the surface of the laminate. Hence a metal mesh was used as a surface layer to eliminate this problem.

• Laminates with the embedded HEL were placed in a climate chamber cooled to -50°C. The IPS was tested in anti and deicing conditions. It was found that the IPS concept was more effective as a deicing system, as less power and time was required for the surface of the laminate to reach temperatures above freezing point.

• Positive results were found for the resistance of the IPS to thermal cycling. While few instances of matrix cracks were found towards the end of the 10000 cycles, this was due to ageing of the
polymer rather than the large difference in the coefficient of thermal expansion between the metal wires and PPS matrix.

• The lay-up integrity of the integrated IPS was tested through the thermoforming technique of rubber forming. The system proved to be robust enough to cope with the large forming pressures and all electrical connections remained intact.

Bibliography


Chapter 11

Improving erosion resistance of polymer reinforced composites

11.1 Introduction

This investigation concentrates on the inherent problem of erosion. Erosion tests are detailed with the aim of improving erosion resistance of the anti/de-icing concept described in the previous chapter using a metal mesh protective layer.

11.1.1 Erosion

Erosion has been defined as the effect of solid particles impinging a target surface causing local damage combined with material removal [1]. In aircraft, the most susceptible areas to be eroded are leading and trailing edges, radomes, landing gear doors, aerodynamic covers and fairings. This situation is particularly more critical during takeoff and landing due to sand erosion. From the materials point of view, it is important to note that fibre-reinforced polymer composites exhibit poor erosion resistance compared to metallic materials [2] and neat polymers. Nevertheless, the wear behavior of composites has received much less attention than that of conventional materials [3]. Interesting reviews of the phenomenon have been presented by Teware et al. [4] and Arjula [5].

Figure 11.1 shows a summary of erosion behaviour given in the form of weight-loss versus impingement, or impact, angle of the erodent particles. At lower erodent impingement angles, ductile materials are susceptible to micro-cutting and ploughing mechanisms, resulting in greater material removal from erosive wear, usually reaching a peak at 30° [6]. Brittle materials are able to resist this effect due to the harder surface. At higher impingement angles, however, repeated impact by erodent particles can cause micro-fractures to occur on the surface, ultimately leading to material removal. In the case of ductile materials, much of the energy of the erodent particles is dissipated in deforming the material, thereby limiting the amount of material removed [6].

Generally neat thermoplastic polymers exhibit good erosion resistance compared to those reinforced with brittle fibres. The ductile nature of most thermoplastics results in very good erosion resistance particularly at higher impingement angles. When brittle fibres are added to the polymer matrix, the erosion resistance decreases further at lower impact angles because the matrix is removed first, exposing the reinforcement that can be easily damaged through micro-bending and fracture mechanisms [7]. Previous studies have shown that using more ductile fibres reduce this effect [7, 8].

The ice-protection system described in the previous chapter used a mesh layer to dissipate the heat evenly over the surface. While lighter alternatives also exist, such as improving thermal
conductivity through nanoparticle additions, the mesh may also prove to be beneficial for erosion. The mesh, composed of fine wires woven into an open plain weave, is essentially a resin-rich layer. Regarding erosion, it is possible to take advantage of the ductility of the polymer matrix at high impingement angles while improving erosion properties of the composite laminate at low impingement angles.

## 11.2 Experimental Technique

### 11.2.1 Material preparation

In aircraft wings, hydraulic actuators are usually close to leading edges. Thus, the thermoplastic polymer polyphenylene sulfide (PPS) was chosen as the matrix material thanks to its chemical resistance to hydraulic fluids such as Skydrol. Woven 8HS glass and 5HS carbon fibre reinforced PPS prepregs and PPS foils, supplied by Ten Cate Advanced Composites in The Netherlands, were used to manufacture the composites. Sample laminates were prepared using two top (impact side) layers of glass/PPS and two layers of carbon/PPS. This was done to help visualize the eroded area by providing a contrasting background for the white glass/PPS. On top of the glass/PPS ply, various metal meshes were embedded as the erosion protection layer and their details are presented in Table 11.1. In addition, 1mm thick ALCOA AL6013T4 aluminium alloy sheets were also analysed and used for comparison of erosion behaviour with an aircraft-grade metal.

Four types of specimens were prepared for the study: i) A regular laminate designated **Plain**, (ii) a laminate with a fine mesh (**Fine**), (iii) a laminate with a rough mesh (**Rough**), and (iv) a laminate using a rough mesh impregnated with rubber blended PPS (**Blend**). Rubber blended PPS was extruded using 80% PPS Ticona Fortron 0214C1 granules and 20% Kraton GX 1657 styrene ethylene butylene styrene (SBS) co-polymer. The extrusion was chopped into fine granules and dried at 80°C for at least 24 hours to remove moisture. To produce foils, the granules were dispersed evenly over a stainless steel plate and placed in a hot plate press for consolidation. The foils were used to impregnate the rough mesh to create a toughened layer. Meshes were first pre-cleaned using a degreasing procedure and then placed on top of the laminate stack, as shown in Figure 11.2(a). For the rough mesh, extra
11.2 Experimental Technique

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Table 11.1 Metal mesh configurations.

Figure 11.2 Material preparation. (a) Laminate stack showing plain and mesh layers, and (b) Example of an impregnated mesh surface.

PPS foils were placed on top of it to ensure sufficient impregnation within the laminate, i.e. the wires were completely surrounded by polymer. The desired laminate stack was placed in a hot plate press for consolidation using the temperature and pressure conditions as specified by the material supplier. Due to the low viscosity of the PPS at the processing temperature, complete impregnation of the mesh was often not possible, and some wires were slightly exposed as shown in Figure 11.2(b). Finally, the laminates were cut to the desired dimensions using a diamond saw, cleaned with high-pressure air, and weighed.

11.2.2 Erosion tests

Sand was selected as the erodent in this investigation because it is a very real problem for outdoor composite structures and has been most commonly used in previous literature [3, 7] on erosion of composites. Two different mass ranges of Australian (garnet) sand were used as the eroding particles; 150-300µm (fine) and 200-600µm (rough). The erosion tests were performed in a standard sandblast chamber as shown in Figure 11.3. The specimen holder was fixed while the nozzle holder could be adjusted to vary the impact angle and height. The angles were adjusted in the range between 15-90°. The distance between the nozzle and the sample was kept at a constant 45mm, apart from those tests performed at 90° angle which, due to apparatus limitations, was adjusted to 25mm. The sand velocity was 70m/s, measured using a rotating disc technique as the one described by Ruff and Ives [9], and the impact time was fixed at 45s. At this velocity, the mass flow rate for the rough and the fine sand corresponded to 6.6g/s and 11.2g/s respectively.

After erosion testing, the laminate and Aluminium plate specimens were air blasted to remove excess particles before being weighed to determine the extent of material loss. It is clear that since ma-
Chapter 11. Improving erosion resistance

Figure 11.3  Erosion test equipment. (a) Sandblasting chamber, and (b) Nozzle and specimen arrangement.

<table>
<thead>
<tr>
<th>Name</th>
<th>Volume resin content</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rough</td>
<td>0.84</td>
<td>2.36</td>
</tr>
<tr>
<td>Fine</td>
<td>0.74</td>
<td>2.97</td>
</tr>
<tr>
<td>Copper</td>
<td>0.65</td>
<td>3.55</td>
</tr>
<tr>
<td>Alu rough</td>
<td>0.67</td>
<td>1.73</td>
</tr>
<tr>
<td>Alu fine</td>
<td>0.80</td>
<td>1.59</td>
</tr>
<tr>
<td>Aluminium (6013)</td>
<td>-</td>
<td>2.71</td>
</tr>
<tr>
<td>Glass/PPS</td>
<td>0.48</td>
<td>1.92</td>
</tr>
</tbody>
</table>

Table 11.2  Composite volume fraction and densities of the mesh and glass/PPS layers.

Materials of different densities were investigated, a direct comparison of the erosion rate in the traditional sense, i.e. the mass loss per unit mass of erodent particles causing the loss, was insufficient. Therefore a second erosion rate was defined as the volume loss per unit mass loss of erodent particles and was also used as a comparison. The weight- and volume-based erosion rates are distinguished by the subscripts \( w \) and \( v \) respectively. In order to simplify the volume loss calculations, it was assumed only the surface mesh-polymer layer was eroded and that the glass layers underneath remained untouched. It will be shown later that this assumption was valid. The densities used to calculate the erosion rate of the various materials investigated are presented in Table 11.2. Finally, the specimen surfaces and cross-sections were examined under an optical microscope to visualize the extent of damage.

11.3 Results

11.3.1 Rough sand

Figure 11.4 displays the influence of the rough sand impact angle on the erosion rates \( \text{ER}_w \) and \( \text{ER}_v \) of the Aluminium and composite laminate specimens. At all impact angles tested, the Plain glass/PPS samples lost considerably more weight than the Aluminium plates and corresponding to an \( \text{ER}_w \) of nearly 11 times more at 90°. When the reinforcing meshes were added to the laminate, a dramatic reduction in \( \text{ER}_w \) was found. In many cases similar, and even lower, \( \text{ER}_w \) were obtained compared
to that of the Aluminium plates. It is also clear from the results that the rough mesh resulted in consistently lower \( \text{ER}_{w} \) than the fine mesh. However, when the volume loss was considered, the \( \text{ER}_{v} \) shows virtually no difference between the performance of both the rough and the fine meshes compared to the aluminium.

The Plain laminate showed peak erosion rates at 45°, indicating that the damage was semi-ductile, i.e. neither completely ductile nor brittle, in nature and is in keeping with previous results [10]. It was expected that the presence of the higher volume of matrix at the surface of the meshed laminates would result in erosion peaks at a lower angle, due to the greater ductility of the PPS and metal wires. The peak values were found at 45°, meaning that the ductility of the matrix rich layer was not obvious in the erosion behaviour. It is widely accepted [7] that the erosion behaviour of a material is dependent on a number of parameters and does not necessarily reflect its failure mode.

At 30° sand impacting angle, the Rough and Fine laminates exhibited a slight dip in erosion rate. This phenomenon occurred only on the tested mesh laminates but it was not seen on the aluminium sheet nor the Plain laminates. The surface of the meshed laminates were not relatively smooth compared to the other materials due to the presence of the mesh underneath as shown in Figure 11.2, resulting in an undulated surface. It is likely that at 30° impingement angle, a large proportion of sand particles impacted locally at normal, or close to normal, angles due to the undulations, resulting in a decrease of material removal. Figure 11.5 shows an exaggerated view illustrating this effect.

Figure 11.6 shows the visual and microscopic images of the specimens that were impacted at impingement angles of 90° and 30° with rough sand. A ruler was placed at the bottom of each image to aid comparison of the damage area of each of the specimens. The microscopic images are cross-sections taken across the centre of the damaged area, cut at the line AA shown in Figure 11.6(a-ii). In the case of the laminates impacted at 30°, the cross-section was taken parallel to the particle flow, which flowed from left to right, indicated by the arrow in Figure 11.6(a-ii). Compared to the Aluminium plates of Figure 11.6(a), the poorer erosion resistance of the Plain glass/PPS laminate is clear as depicted in Figure 11.6(b). The Aluminium sheets had comparatively higher erosion resistance and the sand particles removed material through roughening of the uppermost surface regions at both angles. In contrast, the erosion damage at 90°, and to a lesser extent at 30°, exposed the carbon layers of the Plain laminate, placed two glass layers below the surface of the Plain laminates. The sand particles did not spread upon impact, as with the Aluminium sheets, but were concentrated over a smaller area and therefore with more energy to create more damage in the depth, as shown in the cross-section images.

Interestingly, the laminates reinforced with both the rough and the fine meshes behaved very differently from the Plain glass/PPS laminate and each other, as shown in Figures 11.6(c) and (d). This aspect is clearly observed in their cross-section images. At higher impingement angles, the damage area was similar to that of the plain laminate but it did not reach the same depth, and the glass layers were not exposed. The matrix rich mesh layer restricted the depth of penetration of the sand, and improved the erosion resistance, compared to the Plain laminate. The erosion resistance of the plain laminates was low due to the brittle nature of the glass fibres. Increasing the volume fraction of the tougher matrix through the presence of the mesh layer served to increase the resistance, even at low impingement angles. In fact, the mesh layer acted as an interleave, which toughened the laminate and made it more impact resistant [11]. The difference between the rough and the fine meshes stems from the same reasoning; that the rough mesh held a larger volume fraction, and also a thicker layer, of matrix. The fine mesh was completely eroded away at the site of sand impact at 90°, whereas the rough mesh became roughened and more exposed. The result was a similar volume of material loss shown by the \( \text{ER}_{v} \) in Figure 11.4(b).

At low impingement angles, the mesh also served to restrict the damage area and depth of penetration of the sand compared to the Plain laminates, as can be seen in the cross-section images of
Figure 11.4 Effect of sand impact angle on the erosion rate using rough sand particles.

Figure 11.5 Schematic of sand particles impinging on an undulated surface.
11.3 Results

Figure 11.6(ii). The reason for this can be attributed to two mechanisms. In the first case, the wires of the metal mesh acted as an extra ductile reinforcement to the matrix. Due to the circular cross-section of the wires, locally the sand impacts normal to its surface even at globally low impingement angles. The deformation of the wires due to the impact force can be seen in the images. As mentioned previously, ductile materials perform well at high impingement angles, and hence the impact energy of the particles was maximising the benefits of the metal performance with that of the polymer matrix. The second mechanism was due to a “barrier” effect of the mesh, which served to restrict the damage area of the matrix rich areas that surround the metal wires, and is highlighted in the microscope image in Figures 11.6(c-ii) and (d-ii).

11.3.2 Fine sand

The influence of the fine sand impact angle on the erosion rates $ER_w$ and $ER_v$ of the Aluminium and composite laminate specimens are shown in Figure 11.7. Experiments were not continued with the fine mesh due to the extent of damage compared to the rough mesh with the rough sand, and it can be assumed that a similar trend would occur. Instead, a PPS-rubber blend was produced and used to impregnate the rough mesh layer.

The erosion effect of the fine particles on all tested specimens was found to be considerably less than with the rough particles. This was expected since for the same erosion time and speed, the kinetic energy was less for the fine particles. The erosion rates of the Plain laminate compared to the Aluminium plate were similar with that of the rough sand, at around 11 times at 90°, although the erosion rates at the lower impingement angles were notably lower. In this case, the kinetic energy of the erodent particles may not have been great enough to create a considerable amount of material removal as with the rough sand.

The erosion rates $ER_w$ and $ER_v$ of the Aluminium plate and the meshed laminates were comparable as the densities of all the materials were very similar. As with the rough sand, peak erosion rates occurred at 45° for all but the Blend laminates, hence reducing the erodent particle size did not alter the semi-ductile nature of damage of the materials. A reduction in the erosion rates occurred with the inclusion of meshes, and in all cases were similar to, or slightly higher than, that found with the Aluminium plate. The peak erosion rate was shifted to 30° for the Blend laminates, indicating a change to more ductile damage, even though material removal was similar to the laminates reinforced with the rough mesh.

Figure 11.8 shows the visual and microscopy images of the specimens that were impacted at impingement angles of 90° and 30° with fine sand. As with the rough sand, the cross-section of the laminate images impacted at 30° was taken parallel to the particle flow, which flew from left to right. The Aluminium plates showed similar damage to those impinged with rough sand, and the Plain laminates showed a smaller area of damage but were also eroded until the first carbon layer.

The microscopy images of Figure 11.8(c) and (d) show that the Rough and Blend laminates presented similar damage. At 90° the PPS layer eroded to the extent that the meshes were exposed and greatly deformed by the sand particles. At 30°, the oblique impingement angle resulted in the mesh and matrix being shaved away, rather than deformed as was with the rough sand, and the surface had a smoother appearance. The slightly greater erosion rates found at some angles compared to the Aluminium sheet were therefore due to the material removal that included more of the metal mesh.

The Blend laminates were more resistant to erosion when impacted at 90° as can be seen in the extent of the damage area in Figure 11.8(d). The softer, more ductile matrix blend better withstood the impact. At an angle of 30°, the restricted effect of the blend became more obvious as the matrix was more readily eroded, together with the metal mesh. It may have been that the PPS-blend specimen was
too soft for impact and is obvious there is a limit to how soft the material can be for erosion resistance. A lower volume fraction of the rubber may have produced better erosion resistance properties particularly at lower impingement angles.

11.3.3 Effect of varying mesh material

In order to determine the importance of the metal mesh itself for erosion resistance, three additional meshes were examined. A copper, a rough aluminium, and a fine aluminium mesh were evaluated under the same conditions as those specimens eroded with rough sand particles. Rough sand was chosen because it is more aggressive and therefore more discriminative for comparison of the erosion resistance. Figure 11.9 displays the results of the erosion tests, with the rough stainless steel mesh (Rough) also displayed for comparison.

Once again, peak erosion rates occurred at $45^\circ$ indicating that the damage mode was not influenced by the use of different metals. The variance of the $ER_w$ between each of the meshes tested shows that there is clearly great influence of the mesh material on the erosion resistance of the laminate. The copper mesh had the highest $ER_w$, followed by the fine and the rough aluminium meshes respectively.

There is a clearer difference between the meshes when the $ER_v$ is considered. The $ER_v$ of the Copper laminates show a decrease in erosion rate compared to the other laminates. The Alu Fine and Rough laminates lost the greatest and least volume of material respectively on average across the range of impingement angles. It should be noted that the dimensions of the meshes varied between each other and the erosion behaviour may have stemmed from geometrical, i.e. matrix versus metal volume fraction, or material reasons, or a combination of the two. A closer inspection of the sample cross-section images is shown in Figure 11.10. The most obvious is the damage of the copper mesh layer which was completely eroded away at an impingement angle of $90^\circ$ and heavily eroded at $30^\circ$, Figure 11.10(a). Two reasons contributed to the damage. First, the copper was too soft to provide adequate resistance to the eroding particles which can be particularly seen at $30^\circ$, where the wires are very deformed compared to images taken of other meshed laminates. Second, the smaller volume fraction of the PPS in the surface layer due to the small mesh opening also contributed in lowering the erosion resistance. For the time tested however, the erosion was limited to the mesh layer and the glass layers appeared undamaged. This layer was thinner than that of the other meshes, accounting for the lower $ER_v$ compared to the $ER_w$.

In the case of the Aluminium meshes, they were eroded more extensively, as shown in Figures 11.10(b) and (c), than the rough stainless steel mesh. If the volume fractions are considered, the Copper and Alu Rough laminates, and the Rough and Alu fine, have similar matrix content of around 0.84 and 0.80 respectively (Table 11.2). If the matrix was considered as the most important factor governing the erosion behaviour, as was claimed by Roy et al. [2], it would be expected that the erosion rates of those specimens with similar volume fractions would be the same. However, this was found not to be the case, which provides evidence that the metal wires are also contributing to the erosion behaviour. The hardness of aluminium is greater than that of stainless steel and so at high impingement angles, the Alu fine mesh is able to offer sufficient erosion resistance. Aluminium is harder than copper but the mesh diameter used in this case was larger, again allowing greater erosion resistance compared to the copper mesh. The microscope images show that the aluminium wires were also deformed due to
the erodent particles at both impingement angles. At lower impingement angles the same mechanism mentioned earlier also contributed; the "barrier" effect of the Aluminium rough mesh can be seen more clearly in its microscope image shown in Figure 11.10(c), where the erosion of the matrix is confined causing wave-like forms on the surface.

11.4 Discussion

It has been frequently found in literature that the erosion rates of fibre-reinforced polymers are an order of magnitude higher than that of metals [3]. With the testing conditions used in this investigation, the difference in erosion rates between the woven glass/PPS plain laminate and the aluminium sheet was found to be lower than one order of magnitude, excluding those at 90°. This was especially true at lower impingement angles and with larger sand particles used as the erodent. The energy required to locally bend and thus fracture the reinforcing fibres was higher compared to unidirectional laminates because resistance to bending was provided by the fibre bundles running parallel, to the erodent particle flow [3], thereby reducing the erosion rate. This effect also served to restrict the damage area, which was less than half that of the aluminium sheet, but not the damage depth.

Erosion penetration was found to be dependent on the toughness of the surface layers which, in turn, was increased with the addition of a resin-rich metal mesh layer. The high volume fraction of matrix that could be held within the mesh increased the local ductility of the surface layer contributing to its toughness. The presence of the metal wires also served to restrict the damage area due to the barrier effect. As the wires were ductile, the problem of fibre fracture experienced with the plain glass/PPS laminate was eliminated and extra energy was expended instead in deforming the metal, reducing the material loss. These mechanisms combined were found to be so effective, that the erosion rates were reduced to values close to that of the aluminium.

It is clear that there must be a trade-off between ductility of the wires, for absorbing impact energy of the erodent particles, and hardness to restrict the amount of material removal of the wires. In order to visualise the importance of this trade-off, microscope images were taken of the surface of the Plain, Rough, Alu Rough and Copper mesh specimen before and after erosion at 30°impingement angle and shown in Figure 11.11. The extent of material removal of the Plain laminate is obvious in Figure 11.11(a), where the next ply, the carbon fibre layer, can be seen as dark patches within the image. It should be noted that the thickness of each glass ply layer was 0.25mm meaning that the sand particles eroded to a minimum depth of 0.5mm, more than the total thickness of any of the mesh layers. The inclusion of a relatively matrix rich mesh layer, in comparison to the Plain surface layers, clearly restricts damage and material removal to this layer. After erosion, the surface of the Rough laminate was still mainly intact with the surface of the stainless steel wires exposed and roughened by the sand. The barrier effect is evident, with the left side of the vertical fibres more exposed than the right side, due to the sand that impacted from the left. Reducing the hardness of the wires, such as in the case of the Alu Rough and Copper laminates, resulted in the type of damage shown in Figures 11.11(c) and (d). At this angle, the sand particles damaged the wires with a shearing-type deformation typical of ductile failure, which absorbed a large amount of energy and restricted the volume of material lost. However, the extent of damage of the wires themselves was greater than that observed for the stainless steel. Therefore it is more beneficial to have a harder mesh material, which deforms less, offers greater erosion resistance and increases the erosion lifetime of the laminate.

The investigation has shown that the erosion performance of the fibre-reinforced polymer structure can be enhanced through the addition of a metal mesh layer. It was also found that using a metal mesh would conveniently provide multifunctional uses for the original anti/deicing concept.
The use of a mesh embedded on top of the laminate in comparison with a bonded metallic strip is a weight-saving solution for erosion as the density is reduced. Next, metal meshes are currently being used to provide lightning-strike protection for aircraft and wind-turbine blades [12]. As the mesh layer is placed on the surface, this would then provide an inherent lightning-strike protection for the system and local aircraft structure. Finally, rapid and efficient heat distribution over the surface of the structure has been shown to be extremely important for an effective IPS.

11.5 Conclusions

The investigation has shown that the erosion performance of polymer reinforced composite structure can be enhanced through the addition of a metal mesh layer. Erosion tests were performed on plain and metal-meshed glass/PPS laminates and compared to an aircraft-grade aluminium sheet. The effects of impingement angle, particle size and metal mesh material were studied.

- The behaviour of all the materials did not differ considerably with the two ranges of erodent particle size used, apart from the extent of material removal, and was mainly dependent on the impingement angle. The plain glass/PPS laminates exhibited erosion rates that were up to 11 times higher than that of the aluminium sheet, but with a smaller damage area. The restriction of damage area was due to the weave of the glass fibres, which reduced the lateral erosion.

- Erosion penetration was found to be dependent on the toughness of the surface layer, which was increased with the addition of a metal mesh. The mesh layer acted as an interleave, such that a large volume fraction of the matrix was present on the surface of the laminate. The matrix held within the mesh increased the local ductility of the surface layer also contributing to its toughness.

- The presence of the metal wires also served to restrict the damage area due to what was termed as the barrier effect. As the wires were ductile, extra energy was expended instead in deforming the metal, reducing the material loss. These mechanisms combined were found to be so effective, that the erosion rates of glass/PPS laminates were reduced to values similar to that of the aluminium sheet.

- Of the mesh metals investigated, stainless steel was found to have the lowest erosion rates. This was due to a trade-off between having the highest volume fraction of matrix within the mesh layer and a high relative hardness, which reduced the amount of material removal during erosion.

Finally, it was also found that using a metal mesh would conveniently provide and/or enhance multiple functions for polymer reinforced composites as was shown with the integrated anti/deicing protection system case.

Bibliography


Chapter 11. Improving erosion resistance

Figure 11.6  Eroded specimens impacted with rough sand at 90° and 30° angles. The images show the top eroded surfaces (left) and their cross-sections (right) for both impacted angles.
Figure 11.7  Effect of sand impact angle on the erosion rate using fine sand particles.
Figure 11.8  Eroded specimens impacted with fine sand at 90° and 30° angles. The images show the top eroded surfaces (left) and their cross-sections (right) for both impacted angles.
Figure 11.9  Effect of rough sand particles on the erosion rate of specimens with different mesh material.
Figure 11.10  Micrographs of various metallic meshes after impact at 90° (above) and 30° (below) angles with rough sand.
Figure 11.11 Microscopy surface images of various laminates with metallic meshes displaying the difference in damage.
Chapter 12

Conclusions and Recommendations

The main aim of this thesis was to examine the potential of metal fibres, wires and meshes for use in fibre-reinforced polymer composites. This Chapter summarises the outcomes from the thesis and outlines areas for future work.

12.1 Conclusions

12.1.1 Background

Metal inclusions were first introduced (Chapter 1) and subdivided into three groups of fibre bundles, fine wires and meshes. For convenience, the generic name for all types of inclusions was defined for this thesis as "fibres". An overview of previous work was presented performed on metal fibre hybridised with polymer composites (Chapter 2). It was shown that although metal fibres have been, and still are, readily available, the potential of these materials have not yet been fully explored, particularly for high-level engineering applications such as aircraft structures. Stainless steel was singled out as the metal fibre type in this research due to its availability and excellent mechanical properties.

12.1.2 Mechanical properties

Metal fibre bundles, wires and meshes were tensile tested to provide an idea of the properties that could be expected from these materials when added as reinforcement to polymers (Chapter 3). It was found that the three different types of fibre behaved differently according to their form. Fibre bundles became weaker, less stiff and more brittle with decreasing diameter of the individual fibres, due to the increasing influence of surface flaws on mechanical properties. The wires did not reflect this behaviour but revealed that properties may differ from wires obtained from different sources. The meshes had consistently lower tensile properties than the equivalent sheet metal, due to weakening crimp effects. It was clear that the metal fibres could be exploited for their ductility and stiffness.

Many material properties depend on the level of fibre/matrix adhesion. Attempts were made to improve the adhesion of stainless steel to two types of thermoplastics, PEI and PPS, focusing on the metal rather than the polymer (Chapter 4). It was found that the most effective surface treatments were those that roughened the surface of the metal at the grain-level, such as annodising. While there was no problem applying these techniques to metal meshes (Chapter 7), treating fine wires and bundles in this way would not be practical since procedures such as annodising are not continuous. Hence no feasible solution to improve adhesion was found during this research. Metal fibres were then embedded in various types of glass-fibre reinforced laminates and tests were performed in which stiffness and...
ductility contributions would be made clear, namely low velocity impact (Chapters 5 and 6) and flexure (Chapter 7). Impact tests revealed the importance of matrix compatibility, the importance of a minimum level of adhesion and the metal/matrix synergy (Chapter 6). The presence of the metal fibres alone does not improve impact properties, but is very effective as a combination of metal and matrix. The embedded metal reinforces the matrix against failure creating a tough layer. The matrix must be ductile enough to allow the embedded metal to deform, which at high impact energies absorbs energy, and the difference was shown with the impact behaviour of relatively brittle PPS laminates compared to tougher epoxy and PEI laminates with embedded meshes. At impact energies that are far below the perforation energy, the effect of metal fibres are minimal as the deformations experienced by the laminate are small (Chapters 5 and 6).

Adding metal fibres to a fibre-reinforced thermoplastic creates thermal residual stress issues due to thermal expansion (and shrinkage) differences. If the fibres are placed in specific places within the laminate, rather than uniformly dispersed, these stresses can have a remarkable effect on the entire laminate (Chapter 7). Metal meshes were placed in various positions within the laminate stack and placed under flexure. Due partly to the presence of the mesh and partly to the beneficial residual stress distribution, flexural properties were shown to improve. The exact contributions of the mesh layer and the residual stresses could not be determined. Finally the flexural properties were very dependent on the level of adhesion between the metal and the matrix.

### 12.1.3 Adding functionality

The electrical properties of the metal were used to add heating functionality to the laminate to create two new applications, and the concept of the heat emitting layer was introduced. Metal wires were stitched in parallel into glass fabric and embedded into the laminate. When electrically activated, the metal wires generate heat. The first application was to use this heat as an aid for thermographic non-destructive evaluation (Chapters 8 and 9). Such a heat emitting layer allows even heat-up of flat and curved panels, increasing inspection capability. The spacing of the wires and the thickness of the laminate determine the dimensions of the defect that can be detected, but proved to be a promising technique.

The second application uses the heat emitting layer for an integrated ice-protection system (Chapter 10). An additional metal mesh surface layer was required to adequately dissipate the heat generated by the wires. While in many cases this may not be desirable due to the weight gain, the mesh layer was shown to have benefits regarding erosion resistance (Chapter 11). Static icing tests were performed and the input power required for anti/deicing were shown to be relatively low. However, although the concept of using a heat emitting layer as an ice-protection system is certainly feasible, much work is needed to improve thermal and electrical efficiency as well as reliability.

### 12.1.4 Further comments

Considering mechanical properties alone, metal fibres are desirable, in theory, for applications where particularly ductility or toughness is required. However, other families of organic and inorganic fibres are currently available that are more competitive in terms of properties, price and weight.

Other properties, such as the electrical properties exploited in this research, make metal fibre inclusions far more interesting. Low volume fractions of metal fibres were used in the two application cases, with only a small weight penalty. Higher volume fractions could open the design envelope to include lightning strike protection and electromagnetic shielding. For metal fibres to have potential in future high-level engineering applications, the structural benefits of hybridising with other fibres as
well as taking advantage of properties unique to metals should be combined.

12.2 Recommendations

As with any thesis, the more that is researched, the more questions that are created. A number of recommendations are put forward in the first step to answering some of these questions.

Metal fibre bundles were not explored fully as they were not freely available during the period of this research, but are still interesting for their properties. The advantage is that due to their rough surface, adhesion is less of an issue than with the fine wires or meshes, and therefore provides a good starting base for the investigation of hybridising metal fibres with other reinforcement fibres.

The metal fibres have only been added as a "layer" reinforcement, and placed in specific positions within the laminate stack, such that there was a clear distinction between the metal layer and other reinforcement layers. The next phase would be to fully integrate the metal fibres to eliminate this distinction by hybridising through commingling or co-laying with other reinforcement fibres. Hybrid woven or non-crimp fabrics may offer benefits for impact properties by offering ply-by-ply toughness as well as integrated electromagnetic and thermal properties. In addition, the thermal expansion properties of the laminate can be tailored through hybridisation. This would have particular benefits for structural applications exposed to large temperature variations, where composite and metal parts, for example, need to be fastened together, and thermal residual stresses need to be reduced.

Stainless steel was chosen for this research as its all-round properties are very good, but titanium could offer a comparable, lighter alternative. In addition, the use of shape memory alloys could offer interesting properties for impact due to their deformation capability. Hence, other metals still need to be investigated and unlike the approach taken with this thesis, the most suitable metal for the particular application should be chosen.

In the case where metal fibres are added as a hybrid component in thermoplastic composites, the issue of thermal residual stresses are an issue. It was shown that the residual stress can be beneficial to the mechanical properties of the laminate. However, the full effect of the presence of these stresses is not yet fully understood and should be investigated.

While numerical modelling was not described in this thesis, a finite element investigation to model the mechanical behaviour of the hybrid laminates was performed. However, this was abandoned as it became clear through experimental testing that the hybrids behaved in a more complex manner than expected, particularly for the thermoplastic laminates where residual stresses were also an issue. More time and effort needs to be invested in developing accurate models for these hybrid composites, so that the effect of variables such as metal fibre volume fraction, placement and type, can be determined.

Finally, metal fibres were researched with high-end engineering applications, namely aircraft structures, in mind. There may also be applications in other areas, such as structural automotive parts, where metal fibres can find a place as reinforcements for less demanding structures, opening further the potential areas of investigation.
Chapter 12. Conclusions and Recommendations
Appendix A

Calculations for Chapter 7

This appendix outlines in more detail the calculations to obtained values used in Chapter 7. As the calculations are the same for the PPS and PEI laminates, those for the latter only are shown.

A.1 Calculation of coefficient of thermal expansion of the mesh and glass layers

The calculation of the coefficient of thermal expansion (CTE), starts with the equation for strain:

\[ \epsilon = \alpha \Delta T + \frac{F}{AE} \]  

(A.1)

Considering that the mesh and matrix are constrained, Figure A.1, compatibility yields:

\[ \left[ \alpha \Delta T + \frac{F}{\nu E} \right]_{\text{metal}} = \left[ \alpha \Delta T - \frac{F}{\nu E} \right]_{\text{matrix}} \]  

(A.2)

where \( \alpha \) is the coefficient of thermal expansion, \( \Delta T \) is the temperature rise, \( P \) is the force, \( \nu \) is the volume fraction and \( E \) is the Young’s modulus. It should be noted that the term on the right-hand side is negative as, due to the greater CTE value, the mesh will push on the matrix. For a 1° temperature difference, and using the values shown in Table A.1, the equation above can be solved for \( F \). This value is then inputted into Equation A.1 to give \( \alpha_{\text{mesh layer}} = 19.16 \mu m/m{°}C \). Similarly, the calculation can be repeated for the glass/PEI layer to give \( \alpha_{\text{glass layer}} = 10.06 \mu m/m{°}C \).

![Figure A.1 Schematic representation of mesh and matrix constrained.](image)

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For the mesh/PEI layer
\[ \alpha_{\text{stainless steel}} = 17 \times 10^{-6} \mu m/m \cdot ^\circ C \]
\[ \alpha_{\text{PEI}} = 56 \times 10^{-6} \mu m/m \cdot ^\circ C \]
Volume fraction \(\text{stainless steel} = 0.21\) (Based on mesh 40)
Volume fraction \(\text{PEI} = 0.79\)
Young’s Modulus \(\text{stainless steel} = 210 \text{GPa}\)
Young’s Modulus \(\text{PEI} = 3.28 \text{GPa}\)

For the glass/PEI ply
\[ \alpha_{\text{glass}} = 8 \times 10^{-6} \mu m/m \cdot ^\circ C \]
Volume fraction \(\text{glass} = 0.5\)
Volume fraction \(\text{PEI} = 0.5\)
Young’s Modulus \(\text{glass} = 73 \text{GPa}\)

<table>
<thead>
<tr>
<th>Material</th>
<th>(E_x) (GPa)</th>
<th>(E_y) (GPa)</th>
<th>(\nu_{xy})</th>
<th>(G_{xy}) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass/PEI</td>
<td>26.0</td>
<td>24.0</td>
<td>0.29</td>
<td>3.36</td>
</tr>
<tr>
<td>PEI/mesh</td>
<td>11.2</td>
<td>11.2</td>
<td>0.29</td>
<td>3.36</td>
</tr>
</tbody>
</table>

Thickness mesh layer = 0.23mm
Thickness glass layer = 0.23mm

Table A.1 Inputs for the calculation of CTE for a mesh/PEI and glass/PEI layers.

Table A.2 Data used for the abd matrix.

A.2 Calculation of the modulus of the mesh/PEI layer

From rules of mixtures:

\[ E_{\text{mesh layer}} = (\nu E)_{\text{mesh}} + (\nu E)_{\text{PEI}} \]

with \(\nu\) as the volume fraction value. Only half the value of the volume fraction of the mesh is used, as only the longitudinal fibres are considered to take the load. Hence, with \(\nu_{\text{mesh}} = 0.105\), the Young’s modulus of the mesh layer is calculated to be 11.23GPa.

A.3 Outputs of the abd matrix

The abd matrix was calculated using the inputs from Table A.2. The stiffness values for the mesh layers were calculated as described in this appendix, and for the glass layers were taken from the manufacturer. The outputs of the abd matrix are shown in Figure A.2.
A.3 Outputs of the abd matrix

### Outputs for the abd matrix

#### Figure A.2 Outputs for the abd matrix

<table>
<thead>
<tr>
<th></th>
<th>e_x</th>
<th>e_y</th>
<th>c_x</th>
<th>c_y</th>
<th>c_z</th>
<th>e_xy</th>
<th>e_yz</th>
<th>c_xz</th>
<th>c_yz</th>
<th>c_zz</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) Laminate with the mesh on the top/bottom</td>
<td>2.01E-05</td>
<td>-5.62E-06</td>
<td>0.00E+00</td>
<td>3.64E-06</td>
<td>-1.11E-06</td>
<td>0.00E+00</td>
<td>Nx</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-5.62E-06</td>
<td>2.46E-05</td>
<td>0.00E+00</td>
<td>-1.11E-06</td>
<td>3.63E-06</td>
<td>0.00E+00</td>
<td>Ny</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>1.44E-04</td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>-4.50E-20</td>
<td>Nxy</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.84E-06</td>
<td>-1.11E-06</td>
<td>0.00E+00</td>
<td>6.20E-05</td>
<td>-1.80E-05</td>
<td>0.00E+00</td>
<td>Mx</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-1.11E-06</td>
<td>3.83E-06</td>
<td>0.00E+00</td>
<td>-1.80E-05</td>
<td>6.63E-05</td>
<td>0.00E+00</td>
<td>My</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>-4.80E-20</td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>4.02E-04</td>
<td>Mxy</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### (b) Laminate with the mesh in the middle

<table>
<thead>
<tr>
<th></th>
<th>e_x</th>
<th>e_y</th>
<th>c_x</th>
<th>c_y</th>
<th>c_z</th>
<th>e_xy</th>
<th>e_yz</th>
<th>c_xz</th>
<th>c_yz</th>
<th>c_zz</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.90E-05</td>
<td>-5.75E-06</td>
<td>0.00E+00</td>
<td>-3.53E-21</td>
<td>1.17E-21</td>
<td>0.00E+00</td>
<td>Nx</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-5.75E-06</td>
<td>2.44E-05</td>
<td>0.00E+00</td>
<td>1.17E-21</td>
<td>-4.12E-21</td>
<td>0.00E+00</td>
<td>Ny</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>1.44E-04</td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>-4.50E-20</td>
<td>Nxy</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-3.53E-21</td>
<td>1.17E-21</td>
<td>0.00E+00</td>
<td>5.21E-05</td>
<td>-1.51E-05</td>
<td>0.00E+00</td>
<td>Mx</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.17E-21</td>
<td>-4.12E-21</td>
<td>0.00E+00</td>
<td>-1.51E-05</td>
<td>5.64E-05</td>
<td>0.00E+00</td>
<td>My</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>-4.80E-20</td>
<td>0.00E+00</td>
<td>0.00E+00</td>
<td>4.02E-04</td>
<td>Mxy</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Appendix B

Pretests performed on hybrid laminates reinforced with metal mesh layers.

B.1 Introduction

A number of variables are associated with incorporating a mesh layer to a composite laminate. Of these, the most important are mesh diameter and opening, mesh positions and the number of layers. As the aim of the research was to determine the effect of the mesh layers on the impact behaviour of the laminate, it was important for these variables to be reduced through simple pretests. This appendix describes those pretests performed on hybrid laminates.

B.2 Experimental details

Preparation of the meshes and laminate production was as described in Chapter 6. In the case of mesh 54, a maximum of two layers were investigated, as the thickness of a glass ply would be overly exceeded. Two additional meshes were also investigated with the dimensions listed in Table B.1. All meshes were placed at the bottom of the laminate.

B.3 Results

B.3.1 Effect of number of mesh layers

Figure B.1 shows the influence of the number of mesh layers on the energy profile of the PPS and epoxy laminates. The most obvious change in impact behaviour was to the perforation energy, which increased by 20% and 23% for the PPS and epoxy laminates, respectively.

<table>
<thead>
<tr>
<th>Mesh number</th>
<th>Material</th>
<th>Wire diameter ($\mu$m)</th>
<th>Spacing ($\mu$m)</th>
<th>Number of layers</th>
</tr>
</thead>
<tbody>
<tr>
<td>54</td>
<td>AISI304</td>
<td>112</td>
<td>358</td>
<td>2</td>
</tr>
<tr>
<td>70</td>
<td>AISI304</td>
<td>112</td>
<td>250</td>
<td>2</td>
</tr>
<tr>
<td>100</td>
<td>AISI316</td>
<td>30</td>
<td>226</td>
<td>8</td>
</tr>
</tbody>
</table>

Table B.1 Mesh details.
B.3.2 Effect of mesh dimension

Figure B.2 shows the influence of mesh dimension on the energy profile of the epoxy laminates. It is clear that the mesh dimensions has an affect on perforation energy. Mesh 100, having the finest wire diameter, resulted in the lowest perforation energy. In was shown in Chapter 3, that the finer wires were lower in strength and had lower strain to failures. This accounted for the lower perforation energy, as the wires were more easily perforated compared to the others tested. Meshes 54 and 70 perforated at roughly the same impact energy and also behaved similarly at lower impact energies.
Figure B.1  Energy profiles of PPS and Epoxy laminates with the mesh placed in the bottom.
Figure B.2  Energy profiles of laminates reinforced with different meshes.
Appendix C

Material properties

This appendix gives a summary of relevant material properties of those used in the thesis. The thermoplastic materials and glass 8 harness satin weaves were obtained from Ten Cate Advanced Composites, and the epoxy resin was obtained from Hexion. Further material data can be obtained directly from these companies. Table C.1 gives the data for the thermoplastic neat resins and PEI prepreg and PPS semipreg. Table C.2 gives the property data for the epoxy resin.
Table C.1 Material data for neat thermoplastic resins and pre/semipregs. All values are obtained at 23°C and 50% humidity.

<table>
<thead>
<tr>
<th>Property</th>
<th>PEI</th>
<th>PPS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm²)</td>
<td>1.27</td>
<td>1.35</td>
</tr>
<tr>
<td>$T_g$ (°C)</td>
<td>215</td>
<td>90</td>
</tr>
<tr>
<td>$T_m$ (°C)</td>
<td>300</td>
<td>280</td>
</tr>
<tr>
<td>CTE (ppm/°C)</td>
<td>55.8</td>
<td>52.2</td>
</tr>
<tr>
<td>7781/PEI</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7781/PPS</td>
<td>warp</td>
<td>weft</td>
</tr>
</tbody>
</table>

| Volume resin (%)     | 50    | 47.5  |
| Tensile strength (MPa) | 484   | 445   | 340  | 383  |
| Tensile modulus (GPa) | 26    | 24    | 22   | 20   |
| Compression strength (MPa) | 727   | 676   | 425  | 295  |
| Compression modulus (GPa) | 29    | 27    | 26   | 24   |
| Flexural strength (MPa) | 669   | 585   | 511  | 390  |
| Flexural Modulus (GPa) | 28    | 25    | 23   | 20   |

Table C.2 Material property data for epoxy resin.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy Epikote 4908 resin and hardener</td>
<td></td>
</tr>
<tr>
<td>Density</td>
<td>1.15g/cm²</td>
</tr>
<tr>
<td>$T_g$</td>
<td>82°C</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>74MPa</td>
</tr>
<tr>
<td>Tensile modulus</td>
<td>2.9GPa</td>
</tr>
</tbody>
</table>
Nomenclature

Abbreviations

AISI American Iron and Steel Institute
AISI 304/316 Two forms of stainless steel
ASTM American Society for Testing and Materials
BVID barely visible impact damage
CAI compression-after-impact
CFRPC continuous fibre reinforced polymer composites
CLT classical laminate theory
CP cross-ply
CTE coefficient of thermal expansion
EMI electromagnetic interference
EMSE electromagnetic shielding effectiveness
ER erosion rate
GF glass fabric
GPS γ-glycidoxypropyltrimethoxysilane
HEL heat emitting layer
HELTHY heat emitting layer for thermography
ISO International Standards Organisation
IPS ice protection system
LT lockin thermography
NDE non-destructive evaluation
PEI polyetherimide
PI polyimide
PPS polyphenylene sulphide
PSL process simulated laminate
RFI radio frequency interference
SD standard deviation
SEM scanning electron microscope
SFRPC short fibre reinforced polymer composites
SS stainless steel
TGA thermogravimetric analysis
tolyl bis-[m-(2-triethoxysilyl-ethyl)toly]polysulphide
UD unidirectional
Symbols

\( a_{11} \)  a constituent of the abd matrix
\( b \)  specimen width (mm)
\( C_p \)  specific heat capacity (J/kgK)
\( d_{11} \)  a constituent of the abd matrix
\( \epsilon \)  strain (-)
\( \epsilon_{max} \)  strain at failure (-)
\( E \)  Young’s modulus (GPa)
\( f_{55} \)  a constituent of the abd matrix
\( F \)  internal load (N)
\( G \)  shear modulus (GPa)
\( I \)  electrical current (amps)
\( K \)  thermal conductivity (W/mK)
\( L \)  span length (mm)
\( \kappa \)  curvature
\( \nu \)  Poisson’s ratio
\( P \)  applied load (N), electrical power (W)
\( \rho \)  density (g/cm\(^3\))
\( R \)  radius of curvature (mm), electrical resistance (\( \Omega \))
\( \sigma \)  tensile stress (MPa)
\( \sigma_{uts} \)  ultimate tensile stress (MPa)
\( \tau_o \)  maximum shear stress due to bending (MPa)
\( T \)  temperature (°C)
\( w_c \)  central deflection (mm)
Curriculum Vitae

Tahira Ahmed was born on 28th November 1980 in Ascot, UK. At the age of four she was enrolled at, and fourteen years graduated from, Rougemont School, South Wales. During these years, she developed a strong interest in the sciences, and was accepted to follow Aeronautical Engineering at Imperial College, London. During this time, she interned at the Nordam group to work on the reverse engineering of aircraft pylon panels. Here she was introduced to new manufacturing techniques that led her interests towards the structural side of aeronautics. In the final year, she decided to take a project at the Delft University of Technology within the Design and Production of Composite Structures group (DPCS). The project was focused on the adhesion aspects of a new prosthetic shoulder design. After graduating, she took a post as a researcher at DPCS before taking a PhD position with the Materials innovation institute (M2i), in 2004. Her research, aimed at exploring the potential of metal fibres in polymer composites, led to those results found in this thesis. After finishing her PhD, she remained with DPCS to continue work on various projects including recycling of composites and actuators for smart structures.
Publications

This thesis


Others


Conferences


Schaumburg, Il. USA, 2008.


**Patents**


Acknowledgements

When I decided I'd had enough of big city life and wanted to get away for a little while, I never expected it to lead to a PhD and 5 years away from the UK! For this, I "blame" my very persuasive promotors, Adriaan Beukers, thank you for offering me such a great opportunity. This also goes for my supervisor, Harald Bersee. Your patience with me and the great chats we have make up for the occasional "allez les bleus!" comments coupled with the distinct lack of welsh rugby shirts in your wardrobe.

This work was funded by M2i, formally the Netherlands Institute for Metal Research and I gratefully acknowledge the support that they have offered during the research. To Angela and Bruno of Bekaert N.V., Belgium; thanks for taking notice.

The group when I first arrived was called "Production Technology". While the name wasn’t one of the reasons I decided to stay in Delft, the people who were to become my colleagues certainly were. Mark, my first officemate, thanks for the Composites 101 lessons and bringing me up to speed with pretty much everything. To the rest of the "old-school" Patricia (miss those hugs), Kjelt, Darko, Giovanni, Simon, Ton, and Dylan whether it was gymming, lunching, or plain old nattering, it was always a pleasure. Witchy, as quickly as you arrived, you left - I miss the pitter-patter of your footsteps in the hall!

Under the new, and more stirring title of "Design and Production of Composite Structures", new blood appeared in the form of Didier and Shafqat (two of the nicest office-mates I could ask for), Julie, Nassira, Irene, Nat, Natcha, Teun, Jordy, Carlos and last but not least, Ab. If you’re looking for cultural diversity, this is where you’ll find it - it’s a great group of people. Slightly further afield, Valeria, Sotiris (and now and then Pyrrhos) and Otto, thanks for the chats and general merriment-making! A special thanks to Lisette Vollmer, a big heart in a small package.

To Melchior, Eric, Wim, Floris, Tom, Minghun, Jean-Francois, Sander, Clemence, Annechien, Philippe, Martin, Olivier and Putu, who were part of the team; merci, bedankt, hokkien and makasih, I wish the best for your futures.

I can’t express my my gratitude enough to the people who physically helped in my research. This goes to Sebastiaan, Fred, Michel, Bob, Hans, Serge, Frans and Niels. The variety of work involved in the research was only possible through you.

Outside of the faculty I have met some very special people. It began with Enri and a little while after, Aura. Girls, thanks for being my paranymphs and my kitchen soul-mates. Calvin, Aurèlè and Julien, thanks for the movies, wiki-lessons and the full bellies. Marnix, you said that I helped you find and love your dribble, but anytime you need that saliva wiping off, I’ll be there. To the lot of you, those dinners, parties, jungle-speed nights and so much more than I can remember kept me busy and very happy. Thank you all.

François, I have a feeling I have a lot more wrinkles coming my way and they will all be because of you. Thanks for those that I have, and those that will come!

Phil, you kept me sane and slightly right-of-the-middle, I certainly wouldn’t have been here if it wasn’t for you. Thanks for being so patient with me.
I am very lucky to have had such loving family to take care of me; Auntie, Uncle, Kashif, Rabeel and Suleman - shukriya. Ay, you being here has brought sunshine.

Finally, this is really down to the two people who deserve everything because ang hindi marunong lumingon sa nakaraan, ay hindi makakarating sa patutunguhan. Mum and Dad, this was for you.