Matrix-to-composite toughness transfer in the mode I and mode II interlaminar fracture of glass-fibre/vinyl ester composites

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Statement of Originality

The Ph.D. research program has been conducted under the supervision of Dr. P.-Y.B. Jar, with Professor K. Takahashi (Kyushu University, Japan), Dr. P.J. Burchill (DSTO, Australia) and Dr. A.E. Lowe (ANU, Australia) acting as supervisors.

The research presented in this thesis is my own original work and due reference has been made to published work consulted during the course of the program.

The following papers have been produced:

Journal papers


Conference papers


Contributions have been made to the following papers during the course of the Ph.D. program, although they are not directly related to the research covered in this thesis:


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Abstract

Glass-fibre reinforced composites are increasingly used for structural applications. However, like high performance carbon-fibre composites, they are susceptible to delamination damage, which is often caused by low-velocity impact. This thesis has investigated the potential of increased matrix toughness to enhance impact-induced delamination resistance in glass-fibre marine composites. The mode I and mode II interlaminar fracture toughness ($G_{IC}$ and $G_{IIc}$ respectively) of unidirectionally reinforced glass-fibre composites, with brittle and rubber-toughened vinyl ester matrices, was characterised under static and dynamic loading conditions. Preliminary studies were made on the influence of fibre lay-up and fibre volume fraction on interlaminar fracture toughness characterisation. These studies established important design and fabrication parameters for specimens used in the following studies, which investigated matrix-to-composite toughness transfer and the influence of loading rate.

Mode I tests were conducted on standard double cantilever beam (DCB) specimens at test rates of 2 and 1000 mm/min, and on composite single-edge-notch bend (SENB) specimens at test rates ranging from 1 mm/min to 1 m/s. A quantitative assessment of toughness transfer was made through comparison of composite $G_{IC}$ with matrix $G_{IC}$ for same loading rate. For crack initiation in the DCB tests, $G_{IC}$ is higher in toughened composites, although matrix-to-composite toughness transfer is only partial due to presence of fibres within energy absorbing matrix deformation zone. During crack propagation, the contribution to fracture energy from fibre bridging mechanisms was greater in the toughened composites than their brittle-matrix counterparts. It was found that a larger matrix deformation zone in the toughened composites encouraged greater fibre bridging, which increased $G_{IC}$ for crack propagation. The high rate SENB tests only yielded values for crack initiation. The trend in results shows no effect of loading rate on matrix or composite $G_{IC}$. As in the DCB tests, the toughened composites provided higher $G_{IC}$ values, but toughness transfer was again only partial due to presence of fibres. It is concluded that the use of rubber-toughened vinyl ester will increase resistance to impact-induced mode I delamination.

Mode II tests were conducted using end-notch-flexure (ENF) specimens at test rates ranging from 1 mm/min to 3 m/s. The $G_{IIc}$ results were compared to the order of matrix $G_{IC}$. There was no significant effect of loading rate or matrix toughness on $G_{IIc}$. The absence of a loading rate effect is consistent with the bulk of the experimental data in the literature, but absence of matrix effect is not. It is concluded that failure is interface controlled, whereby unstable fracture is initiated after a similarly short period of crack growth in each composite, and before an increase in $G_{IIc}$ due to increased matrix toughness becomes apparent. The $G_{IIc}$ results indicate that the use of rubber-toughened vinyl ester matrices in glass-fibre composites will not improve resistance to impact-induced mode II delamination. However, through-thickness impact damage in composite structures is likely to result from mixed-mode (I/II) loading. Therefore, suggestions for future work include in-
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Chapter 1

Introduction

The principal advantages of fibre-reinforced polymer-matrix composites, when compared to traditional engineering materials, are superior strength and stiffness, low density, corrosion resistance, and aesthetic qualities. Consequently, their use for aerospace applications is increasingly evident. Fibre composites are a laminated structure consisting of layers of fibre impregnated with the polymer matrix. The main advantages of the laminated structure is that it allows critical load-bearing path failure to be controlled. However, the major weakness of laminated composite structure is the lack of significant through-thickness reinforcement which renders them prone to delamination failure [1].

Delamination can initiate from flaws and voids introduced during fabrication. However, one of the major causes of delamination damage is low-velocity transverse impact loading [2, 3]. Such impacts can introduce complex loading conditions [4]. Initial damage is
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Introduction

The principal advantages of fibre-reinforced polymer-matrix composites, when compared to traditional engineering materials, are superior strength and stiffness-to-weight ratios, corrosion resistance and aesthetic qualities. Consequently, their use for structural applications is increasingly evident. Fibre composites are a laminated structure consisting of layers of fibre impregnated with the polymer matrix. The main advantage of this laminated structure is that it allows critical load-bearing paths to be selectively reinforced. However, the major weakness of laminated composite structures is the lack of significant through-thickness reinforcement which renders them prone to delamination failure [1]. Delamination can initiate from flaws and voids introduced during fabrication. However, one of the major causes of delamination damage is low-velocity transverse impact loading [2, 3]. Such impacts can introduce complex loading conditions [4]. Initial damage is
usually in the form of matrix cracking, which then initiates delamination. Low-velocity and low-energy impacts are particularly problematic as the delamination damage is often internal or close to the back-face of the structure, making visual detection difficult. This kind of damage is commonly termed barely visible impact damage (BVID).

The presence of delamination cracks within a composite can influence structural integrity. Significant adverse affects have been reported for in-plane mechanical properties such as strength and stiffness, with residual compressive strength and fatigue performance also seriously affected [2, 5, 6]. The potential loss of structural integrity can have catastrophic effects and composites are often only used in secondary loading applications. Improved resistance to delamination is therefore necessary to expand the number of possible applications for composites, and provide designers with more confidence to use composites in primary load-bearing applications.

In response to the problem of delamination, extensive research has been conducted to standardise procedures for delamination testing of composites under static loading. The result has been the recent development of mode I (opening), mode II (shearing) and mixed-mode (I/II) test standards and protocols by the European Structural Integrity Society (ESIS), the American Society for Testing and Materials (ASTM) and the Japanese Industrial Standards (JIS) group [7–9]. The delamination resistance of a composite is characterised by its interlaminar fracture toughness and measured in terms of critical strain energy release rate ($G_c$). The $G_c$ is obtained from testing composite specimens containing artificial pre-cracks. The double cantilever beam (DCB), end notch flexure (ENF) and asymmetrical
DCB (ADCB) specimens are the geometries recommended by the protocols and standards for, respectively, mode I, mode II and mixed-mode (I/II) testing.

Extensive research has also been undertaken with a view to improving composite interlaminar fracture toughness [10]. One of the most effective ways is to alter fibre architecture. Stitched and knitted fibre architectures, which will provide some through-thickness reinforcement, can improve mode I and mode II interlaminar fracture toughness under static loading [11–13], and improve resistance to impact-induced delamination [14, 15]. The use of through thickness reinforcement does however have drawbacks. The introduction of stitching during fabrication can cause localised damage. Furthermore, the overall fibre architecture produces resin-rich pockets, reduced volume fraction and high stress concentrations, all of which can reduce structural performance [16].

The other most effective way to enhance composite $G_c$ is to use tougher matrices, either in the interlayer region between fibre-dense intralaminar regions or, as is more common, for the composite as a whole [10]. However, as noted by Hunston et al. [17], while tougher matrices increase fracture resistance, they often have inferior tensile properties compared to brittle matrices. In order to optimise the mechanical properties of a composite, it is important therefore to determine whether the increased bulk matrix toughness is transferred to the composite. The assessment of mode I toughness transfer is assisted by standardised procedures for evaluating mode I fracture toughness ($G_{Jc}$) of the bulk matrix [18, 19], which can then be compared with composite mode I interlaminar fracture toughness ($G_{Ic}$) [17, 20]. Standardised procedures for bulk matrix fracture tough-
ness under the other failure modes do not currently exist. Nevertheless, increased matrix toughness (measured in terms of $G_{1c}$) can result in increased composite mode II and mixed-mode interlaminar fracture toughness ($G_{IIc}$ and $G_{I/IIc}$ respectively) [10, 20, 21]. Tougher matrices can also increase composite $G_{1c}$, $G_{IIc}$ and $G_{I/IIc}$ at increased loading rates [22–29], thus highlighting the potential of toughened composites to enhance resistance to impact-induced delamination. Furthermore, the rate at which delamination damage is introduced to a composite structure is unpredictable. It can be caused by a tool dropped from a relatively short distance, or from explosive shock or blast loading. Therefore, the effect of loading rate on composite $G_c$ is also important. The trend in the published results varies [30], although this may be due to the response of the polymer matrix to the increase in rate. Frassine et al. [31, 32] and Friedrich et al. [22] have shown that the response of composite $G_{1c}$ can be directly related to the response of bulk matrix $G_{1c}$ with increased loading rate.

The first extensive application of fibre reinforced composites was in defence and aerospace industries which used high performance, and high cost, carbon-fibre reinforced composites. Therefore, most of the aforementioned research into interlaminar fracture toughness characterisation, standard test procedure development, toughness transfer and rate effects has been conducted using carbon-fibre reinforced composites. However, lower cost glass-fibre reinforced composites are now being used for military and civil structural applications. As discussed by Davies [33], one of the greatest areas of potential for application of glass-fibre composites is in a marine environment (for examples see references [34–36]).
Applications include civil and military surface vessels, offshore structures and underwater structures. However, Davies [33] also highlighted a number of problems, namely ageing in corrosive marine environments, fire resistance, and delamination due to wave impact or shock loading.

Glass-fibre marine composites usually have thermoset polymer matrices. These matrix-resins, in liquid form, facilitate easier fabrication of large structures by contact moulding (hand lay-up) or resin transfer moulding [33]. The matrices are usually polyester, vinyl ester or epoxy resins. Of these, vinyl ester has shown greater resistance to hydrolysis [37] and greater retention of mechanical properties after accelerated ageing tests [38] compared to more commonly used polyesters, and it is cheaper than epoxy. The inherent low fracture toughness of neat thermoset resins is a drawback, but for marine composites (and glass-fibre composites in general) it is encouraging to note that the toughness of commercially available vinyl esters can be significantly enhanced by the addition of relatively small amounts of liquid rubber [39, 40]. Pham and Burchill [39] showed that the $G_{ic}$ of a vinyl ester resin, Dow Derakane 8084, can be increased threefold by the addition of 5% (by weight) of liquid rubber (BF Goodrich VTBN X33). The increase in energy absorption was achieved mainly through cavitation of the rubber particles, which were approximately $2\mu m$ in diameter.

A potential marine application for rubber-toughened vinyl ester matrices is in composite joints in military vessels, where the tougher matrices may enhance resistance to impact-induced delamination [41]. Consequently, the desire to assess the delamination-resisting
potential of rubber-toughened vinyl esters matrices, particularly delamination-dominated BVID, has provided the motivation for the materials engineering research covered in this thesis. Interlaminar fracture toughness characterisation of glass-fibre reinforced composites with brittle and rubber-modified vinyl ester matrices has been conducted, with the emphasis on toughness transfer and loading rate effects. Furthermore, the focus has been placed on mode I and mode II failure. Mode I failure absorbs least energy during fracture and is therefore the most dangerous failure mode, while impact-induced delamination is usually mode II dominated [2,42]. Specifically, the major aims were:

1. to investigate the influence of loading rate on bulk matrix mode I fracture toughness ($G_{Ic}$),

2. to quantify the transfer of matrix $G_{Ic}$ to composite mode I interlaminar fracture toughness $G_{Ic}$ with increased loading rate,

3. to investigate the influence of matrix toughness and loading rate on composite mode II interlaminar fracture toughness ($G_{IIc}$).

The materials, fabrication methods and standard mechanical test procedures used throughout the thesis are detailed in chapter 2. Then, the main body of the thesis will comprise of chapters which address discrete aspects of the research, a structure which reflects the approach taken to achieve the aims. Each main chapter will have its own introduction to provide further background information about the subject covered. This is followed by a brief experimental section, results, discussion and conclusion. Preliminary studies on
the influence of fibre lay-up and fibre volume fraction are presented in chapters 3 and 4 respectively. These studies establish the appropriate specimen architecture and fabrication procedures for the following toughness transfer work. In chapter 5, mode I toughness transfer is studied using the standard DCB composite specimen. Then, in chapter 6, high rate mode I toughness transfer is investigated using composite SENB specimens. For both mode I studies, toughness transfer is assessed through direct comparison with bulk matrix \( G_{1c} \) evaluated for the same loading rate. The influence of matrix toughness and loading rate on mode II interlaminar fracture toughness is investigated in chapter 7, with composite \( G_{IIc} \) compared to the order of matrix \( G_{1c} \). Finally, conclusions and suggestions for future work are presented in chapter 8.
Chapter 2

Experimental

2.1 Introduction

This chapter describes the materials, fabrication methods and standardised test procedures used to conduct the research presented in this thesis. However, not all the materials and test procedures described here are used in every study presented in chapters 3-7. Therefore, these chapters will also contain a brief experimental section to identify the materials and test procedures used to achieve the aims of the particular study to which the chapter refers. Any variation to the standard test procedures will also be described in the relevant chapter.
2.2 Materials and fabrication

2.2.1 Fibre reinforcement

Unidirectional and woven roving E-glass fibres with silane sizing were used as reinforcements. Their specifications are summarised in Table 2.1.

<table>
<thead>
<tr>
<th>Fibre lay-up</th>
<th>Areal weight (g/m²)</th>
<th>Manufacturer</th>
<th>Product reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unidirectional</td>
<td>300</td>
<td>Vetrotex</td>
<td>P177</td>
</tr>
<tr>
<td>Unidirectional</td>
<td>300</td>
<td>Owens Corning</td>
<td>R25H</td>
</tr>
<tr>
<td>Woven roving</td>
<td>638*</td>
<td>Colan</td>
<td>AR106</td>
</tr>
</tbody>
</table>

*Warp fibres 354 g/m²; weft 284 g/m².

2.2.2 Matrices

In total, five vinyl ester resins were used as matrices. Two were commercial vinyl esters (Dow Chemical Co.): Derakane 411-45 a bisphenol A epoxy based vinyl ester resin, and Derakane 8084 a toughened version of 411-45 modified with a reactive acrylonitrile-butadiene copolymer during manufacture. The three other matrices were rubber-modified. Derakane 8084 and Hetron 922 (Huntsman Chemical Co.), which is equivalent to Derakane 411-45, were used as the base resins. Hetron 922 was mechanically blended with a core-shell rubber additive (EXL2602, Rohm and Haas) at a resin:additive weight ratio of 100:5 (17 parts styrene were also added to reduce viscosity). The 8084 resin
was mechanically blended with BF Goodrich liquid rubber additives: a vinyl terminated acrylonitrile-butadiene copolymer (VTBN X33) and a carboxy terminated acrylonitrile-butadiene copolymer prereacted with an aliphatic oligomeric epoxide (CTBN S22) [43]. The resin:additive weight ratio in these systems was 100:7.5. Each matrix-resin was formulated by weight ratio as follows: 100 parts resin or resin+additive, 0.2 parts promoter, Cobalt Naphthenate (CoNap); 0.3 parts gel time retarder, 2,4 Pentanedione (2,4-P); and 1.5 parts catalyst, Methyl Ethyl Ketone Peroxide (MEKP). The final additive wt% in the toughened matrices was 4.68% for 922+EXL2602 (9EXL), and 6.85% for both 8084+VTBNx33 (8X33) and 8084+CTBNS22 (8S22). The rubber-modified matrices are summarised in Table 2.2.

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Base resin</th>
<th>Additive type</th>
<th>code</th>
<th>supplier</th>
<th>wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>9EXL</td>
<td>Hetron 922</td>
<td>core-shell rubber</td>
<td>EXL2602</td>
<td>Rohm and Haas</td>
<td>4.68</td>
</tr>
<tr>
<td>8X33</td>
<td>Derakane 8084</td>
<td>liquid rubber</td>
<td>VTBNX33</td>
<td>BF Goodrich</td>
<td>6.85</td>
</tr>
<tr>
<td>8S22</td>
<td>Derakane 8084</td>
<td>liquid rubber</td>
<td>CTBNS22</td>
<td>BF Goodrich</td>
<td>6.85</td>
</tr>
</tbody>
</table>

2.2.3 Fabrication methods

Composite laminates were made by hand lay-up in an open mould, then evacuated using the vacuum bag technique. The mould design is illustrated in Figure 2.1. The fibre plies were layed-up within a resin dam on a flat mould plate. (The number of plies used for laminate fabrication will be noted in the relevant chapter). For each ply, the fibre:resin...
mass ratio was 1:1. A 15µm thick aluminium film, inserted at mid-thickness, acted as an artificial starter crack in the test specimens subsequently cut from the laminate. The film was coated as with a water based PTFE release agent (Safelease #30, Airtech Inc.) prior to insertion. When the lay-up procedure was complete, a caul plate was placed on top of the laminate to ensure uniform thickness, then the mould was evacuated through a port attached to a vacuum bag. During evacuation, the vacuum pressure was maintained at -60 kPa and bleeder cloth absorbed the excess resin which flowed while the vacuum was held. Bulk matrix test specimens were obtained from 10mm thick plates which were cast in aluminium moulds. The laminates and matrix plates were allowed to cure at room temperature for at least 24 hours, then given one of the following post-cure treatments:

1. oven post-cure: 70-75°C for 3 hours, or

2. autoclave post-cure: 90°C for 4 hours

Recent work has shown that the second treatment is the optimum time and temperature for maximum toughness of the Derakane 8084 resin [44]. After post-curing, the laminates and bulk matrix plates were sectioned using a water-cooled diamond saw in preparation for mechanical testing.
2.2.4 Fibre volume fraction

Figure 2.1: Mould design for hand lay-up fabrication.

2.3 Mechanical Testing

2.3.1 Matrix Specimens

The procedure in ASTM standard D638M-91a [16] was followed. 

Figure 2.2: Matrix specimens for mechanical testing (a) type III tensile specimen and (b) single-edge-notch bend (SENB) mode I fracture toughness specimen.
2.2.4 Fibre volume fraction

Composite fibre volume fraction was estimated using the following equation [7]:

\[ \%V_f = \frac{FAW \times N \times 100}{FD \times 2h} \]  

(2.1)

where \( FAW \) is fibre areal weight, \( N \) number of plies, \( FD \) fibre density (2.56 g/cm\(^3\) [45]) and \( 2h \) average thickness of at least 5 specimens cut from the laminate.

2.3 Matrix mechanical testing

2.3.1 Tensile

The procedure in ASTM standard D638M-91a [46] was followed to determine bulk matrix tensile modulus (\( E \)), yield strength (\( \sigma \)) and yield strain (\( \epsilon \)). Type M-III test specimens were used as shown in Figure 2.2(a). The gauge length (\( l \)) was 15 mm, width (\( W \)) 3 mm and thickness (\( t \)) 4 mm. A minimum of five specimens were tested in a screw-driven Instron 4505 universal testing machine (UTM) at a crosshead displacement rate of 1 mm/min. An extensometer was attached to each specimen to measure elongation. Instron series IX materials testing software (version 5.25) was used to calculate the tensile properties.
2.3.2 Mode I fracture toughness

The procedure in ASTM standard D5045 [18] was followed to determine the mode I critical strain energy release rate \( (G_{Ic}) \) and critical-stress-intensity-factor \( (K_{Ic}) \) of the bulk matrix. Single edge notched bend (SENB) test specimens were used, as shown in Figure 2.2(b). The specimens were prepared by milling and nominal dimensions were width \((W)\) 16 mm and thickness \((t)\) 8 mm. The depth of the machined U-notch was 6 mm. A natural crack was introduced to the bulk matrix specimen by tapping a new razor blade placed in the U-notch, such that the final crack length \((a)\) was in the range \(0.45 < a/W < 0.55\). Specimens were placed on a three point bend fixture with a span length \((2L)\) of 64 mm. A minimum of 3 specimens were tested at a crosshead displacement rate of 10 mm/min on either the Instron UTM or a servo-hydraulic Shimadzu UTM (model EHF-FD1). Load-displacement plots were recorded from the Instron UTM using a Philips PM8272 chart recorder, and from the Shimadzu UTM using an NEC Omniace RT3600 digital data recorder.

Total absorbed energy \((U_Q)\) was obtained by integrating the load-displacement plot from each test up to the maximum load point \((P_{\text{max}})\), or the load point corresponding to a 5% increase in initial compliance \((P_Q)\), where \(P_{\text{max}}/P_Q < 1.1\). The definition of the \(P_{\text{max}}\) and \(P_Q\) points is illustrated in Figure 2.3. True energy for fracture \((U)\) was obtained by subtracting the energy absorbed by system compliance, loading pin penetration and sample compression \((U_i)\) from \(U_Q\), following ASTM D5045. It should be noted that the crosshead speed for calibration was adjusted so that the time to reach \(P_{\text{max}}\) or \(P_Q\) was the
same as for the notched specimens.

Figure 2.3: Definition of (a) \( P_{\text{max}} \) and (b) \( P_Q \) points on the load-displacement plot from a mode I fracture toughness test using an SENB specimen.

The matrix \( G_{IC} \) and \( K_{IC} \) values were calculated as follows:

\[
G_{IC} = \frac{U}{(Wt^4)} \quad (2.2)
\]
\[ K_{Ic} = \frac{P_{\text{max}}}{W_{t}^{1/2}} \phi f \] (2.3)

where \( \phi \) and \( f \) are calibration factors given in the ASTM standard. With values for \( K_{Ic} \) and \( \sigma \), the mode I deformation zone size \( (r_D) \) in the matrix SENB specimens can be estimated using the fracture mechanics expression [47]:

\[ r_D = \frac{K_{Ic}^2}{2\pi \sigma^2} \] (2.4)

### 2.4 Composite interlaminar fracture toughness

The procedures outlined in the European Structural Integrity Society (ESIS) Protocols for Interlaminar Fracture Testing of Composites [7] were used for mode I double cantilever beam (DCB) and mode II end notch flexure (ENF) tests. A minimum of 5 specimens were tested in each mode. The specimens were not pre-cracked.

#### 2.4.1 Mode I

The DCB specimens were prepared as shown in Figure 2.4(a). The nominal specimen width \( (W) \) was 20 mm and initial crack length \( (a_0) \) 50 mm. The specimen length was sufficient for at least 80 mm of crack growth. The load was introduced through aluminium...
blocks attached to the end containing the artificial starter crack. From the tip of the starter crack on one side of the specimen, increment marks were placed every 1 mm for the first 5 mm and then at 5 mm intervals up to 80 mm. The side of the specimen was not covered with white paint, to assist crack identification, as suggested by the ESIS protocol. Instead, the translucency of the glass-fibre/vinyl ester specimens enabled the crack to be observed more easily with a transmitted light source. Specimens were tested at a crosshead displacement rate of 2 mm/min on the Instron UTM. Load-displacement plots from the chart recorder provided raw data for each crack length increment.

Expressions for the calculation of mode I critical strain energy release rate, $G_{Ic}$, are derived from the Irwin and Kies expression for fracture energy [48]:

$$G_c = \frac{P^2}{2W} \frac{dC}{da}$$  (2.5)

where $P$ is load, $C$ compliance and $a$ crack length. This expression accounts for the change in compliance with crack length. In this thesis, $G_{Ic}$ has been evaluated using the corrected beam theory method, which was developed by Hashemi et al. [49] and shown to provide results consistent with other commonly used evaluation methods such as Berry's experimental compliance calibration [50] and the areas method [51]. Hashemi et al. used the following compliance expression:
\[ C = \frac{\delta}{P} = \frac{8(a + |\Delta|)^3}{E_f Wh^3} \]  \hspace{1cm} (2.6)

where \( \Delta \) is a crack length correction factor, \( E_f \) flexural modulus and \( h \) half specimen thickness, to determine the expression:

\[ G_{I_c} = \frac{3P\delta}{2W(a + |\Delta|) N} F \]  \hspace{1cm} (2.7)

where \( \delta \) is crosshead displacement. The value of \( \Delta \), determined empirically, is the x-axis intercept on a plot of \( C^{1/3} \) versus \( a \). The values \( F \) and \( N \) are corrections for large displacement and end block effects. These effects can lead to overestimation of \( G_{I_c} \) [52] and should be applied when \( \delta/a > 0.4 \) [7]. Evaluation of \( F \) and \( N \) are dependent on end block dimensions and the evaluation method is given in the ESIS protocol [7]. The \( F \) and \( N \) corrections were applied to all \( G_{I_c} \) results.

Values of \( G_{I_c} \) were plotted as a function of crack length to produce a resistance (R) curve. Where possible, results were obtained for crack initiation \( (G_{I_c-init}) \), defined as the first non-linear point on the load-displacement plot, a 5\% offset in initial compliance \( (G_{I_c-5\%}) \) and steady state crack propagation \( (G_{I_c-prop}) \), defined by a plateau on the R curve. The flexural modulus \( E_f \) was evaluated from equation 2.6.
2.4.2 Mode II

The ENF specimen geometry is shown in Figure 2.4(b). The nominal specimen width (W) was 10 mm, and total specimen length 130 mm. For testing, a constant cross-head speed of 1 mm/min was maintained throughout the test. A small artificial starter crack was introduced at the polycrystalline edge. A linear variable differential transducer (LVDT) was used to measure the crack growth between the tip of the starter crack and the central loading area. The crack growth rate was calculated from the LVDT measurement and digital time and distance measurement. A maximum of 25 mm was obtained for crack growth between the tip of the starter crack and the central loading area. Specimen geometries are based upon Equation 2.3. In this case, G_{IIc} was calculated using the ENF compliance expression given by Equation [36] which uses the ENF compliance expression given by Equation [36].

\[
G_{IIc} = \frac{2L^3 - 3a^3}{8P(W-H)}
\]

where \( L \) is the half-span length, \( a \) crack length, \( E \) Young’s modulus, \( W \) width, \( H \) thickness. From equations 2.3 and 2.4, with \( a \) as the variable, the test was:

---

Figure 2.4: Composite interlaminar fracture toughness test specimen geometries (a) DCB (mode I) and (b) ENF (mode II).
2.4.2 Mode II

The ENF specimen geometry is shown in Figure 2.4(b). The nominal specimen width \( W \) was 20 mm, initial crack length \( a_0 \) 25 mm and total specimen length 120 mm. For testing, the specimen was placed in a three-point bending fixture with the half-span length, \( L \), set at 50 mm and the ratio \( a_0 / L \) maintained at 0.5. To monitor crack growth, one edge of the specimen was polished and increment marks applied every 1 mm with a pencil. A light source directed at the polished edge during the test enhanced crack identification and measurement. Since \( a_0 / L \) was set at 0.5, a maximum of 25 mm was available for crack growth between the tip of the starter crack and the central loading point. Specimens were tested at a crosshead displacement rate of 1 mm/min on the Instron UTM or the Shimadzu UTM. Load-displacement plots from the chart recorder provided raw data for each crack length increment.

Expressions for the calculation of mode II critical strain energy release rate, \( G_{IIc} \), are also based upon equation 2.5. In this study, \( G_{IIc} \) was calculated using the direct beam theory method [53], which uses the ENF compliance expression given by Russell and Street [54]:

\[
C = \frac{2L^3 + 3a^3}{8E_f W h^3}
\]  

(2.8)

where \( L \) is the half-span length, \( a \) crack length, \( E_f \) flexural modulus and \( h \) half the specimen thickness. From equations 2.5 and 2.8, with \( a \) as the sole variable for \( C \):
\[ G_{IIC} = \frac{9a^2P^2}{16EW^2h^3} \]  
\[ (2.9) \]

where \( P \) is load. For beams under small deflection, an expression for \( E \) can be obtained from equation 2.8 which substitutes into equation 2.9, yielding:

\[ G_{IIC} = \frac{9a^2P\delta}{2W(2L^3 + 3a^3)} \]  
\[ (2.10) \]

where \( \delta \) is displacement.

Values of \( G_{IIC} \) for crack initiation \( (G_{IIC-init}) \) and the maximum load point \( (G_{IIC-max}) \) were calculated. For \( G_{IIC-init} \), the crack length used in equation 2.10 was the original crack length, \( a_o \), of 25mm. Flexural modulus, \( E_f \), was evaluated using equation 2.8.

It is noted that large displacement effects can also affect \( G_{IIC} \) results obtained from ENF specimens [52], and corrections are required when \( \delta/L > 0.2 \) [7]. However, \( \delta/L \) was less than 0.2 in all the mode II ENF studies, therefore corrections were not applied.

### 2.5 Microscopy

Electron microscopy was conducted on a Cambridge S360 scanning electron microscope (SEM). Secondary electron images were obtained from specimens coated with a thin layer of gold. Backscattered electron images were obtained from specimens coated with car-
bon. A Wild Heerbrugg Photomakroscop M400 (chapter 4) and a Zeiss Axioskop I (chapters 5-7) were used to obtain optical micrographs.

Chapter 3

The influence of fibre lay-up

1. Introduction

As discussed in chapter 1, much of the interlaminar fracture research used to develop the standard test procedures has been conducted on carbon fibre composites, but these procedures are also suitable for marine composites [35]. The standards also recommend testing of specimens reinforced with unidirectional fibre as they are believed to provide conservative estimates of $G_c$, particularly for crack initiation. However, marine composites are used in various applications, including marine applications [36], and fabricate with woven fabrics to obtain a certain degree of isotropy in mechanical properties. Therefore, there have been interesting attempts to characterize interlaminar fracture toughness in composites with woven fabric lay-ups [36-37].
Chapter 3

The influence of fibre lay-up

3.1 Introduction

As discussed in chapter 1, much of the interlaminar fracture research used to develop the standard test procedures has been conducted on carbon-fibre composites, but these procedures are also suitable for marine composites [55]. The standards also recommend testing of specimens reinforced with unidirectional fibres as they are believed to provide conservative estimates of $G_c$, particularly for crack initiation. However, composites for many structural applications, including marine applications [33], are fabricated with woven fabrics to obtain a certain degree of isotropy in mechanical properties. Therefore, there are increasing attempts to characterise interlaminar fracture toughness in composites with woven fabric lay-ups [56, 57].
This thesis is concerned primarily with a comparison of composite materials with different matrices. Nevertheless, a preliminary study is presented in this chapter on the influence of fibre lay-up on mode I and mode II interlaminar fracture toughness. This study will determine if further toughness transfer studies should be conducted on composites with the recommended unidirectional lay-up, or with a woven fibre lay-up which is of greater interest to industry.

3.2 Experimental

3.2.1 Materials and laminate fabrication

The fibre reinforcement was woven roving E-glass (Colan AR106), and Derakane 411-45 and 8084 were used as matrices. Woven roving (WR) and quasi-unidirectional (UD) laminates were made. The WR laminates contained 10 fibre plies, oriented so that crack growth would be parallel to the warp fibre direction. The quasi-UD laminates contained 4-ply WR regions on either side of a central, 4-ply, UD region. To ensure no variation in results due to fibre type, the UD plies were obtained from the WR plies by removing all weft yarns (except for two end yarns which acted as binders). Each laminate was post-cured at 70-75°C for 3 hours. Composite thickness ($2h$) and fibre volume fraction ($V_f$) are given in Table 3.1.

While WR and UD lay-ups were chosen to minimise any difference in thickness or flex-
Table 3.1: Composite $2h$ and $V_f$ (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>$2h$ (mm)</th>
<th>$V_f$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UD/411-45</td>
<td>5.75 (0.11)</td>
<td>45</td>
</tr>
<tr>
<td>WR/411-45</td>
<td>5.03 (0.07)</td>
<td>50</td>
</tr>
<tr>
<td>UD/8084</td>
<td>5.85 (0.16)</td>
<td>44</td>
</tr>
<tr>
<td>WR/8084</td>
<td>5.96 (0.06)</td>
<td>42</td>
</tr>
</tbody>
</table>

ural modulus between UD and WR specimens, they did introduce anti-symmetry about the mid-plane in the cracked regions of UD specimens. The anti-symmetry may introduce stress concentrations at the interface of the WR and UD plies, thus deviating crack growth from the mid-plane of the specimens. However, during testing delamination crack growth occurred in the specimen mid-plane between the plies of interest and not at the interface of the WR and UD plies. Therefore, any effect of anti-symmetry was regarded as negligible.

3.2.2 Interlaminar fracture toughness tests

Mode I DCB

The standard mode I DCB test was conducted as described in chapter 2 but with raw data from the load-displacement plot noted for every 1 mm of crack growth. $G_{Ic-init}$ results were obtained for all composites. On the other hand, $G_{Ic-prop}$ results were obtained for the UD composites but not for the WR composites due to frequent unstable crack growth.
growth. Instead, $G_{Ic}$ in the WR composites was calculated for short periods of stable crack growth ($G_{Ic-stable}$) and for the onset of unstable crack growth ($G_{Ic-onset}$), following the approach suggested by Davies and Moore [58]. To describe further the crack growth behaviour in WR specimens, average values, per specimen, for the number of and length of unstable crack jumps ($n_{I-unstable}$ and $\Delta a_{I-unstable}$), and the number of stable crack growth measurements ($n_{I-stable}$) were obtained. A check on the variation in $E_f$ between UD and WR specimens was also made.

**Mode II ENF**

The standard mode II ENF test was conducted as described in chapter 2 and $G_{IIc-init}$ and $G_{IIc-max}$ results were obtained for each composite. For $G_{IIc-max}$, any observed change in crack length between the initiation and the maximum load points ($\Delta a_{II-max}$) was added to the original crack length, that is, $a = a_o + \Delta a_{II-max}$. This procedure is not consistent with the ESIS protocol but was necessary as the average $\Delta a_{II-max}$ value, which will be presented in section 3.3.3, is significant. Stable crack propagation in the WR composites enabled a series of crack length measurements to be made, and R curves were constructed. These R curves showed a distinct plateau region, giving values for mode II steady-state crack propagation ($G_{IIc-prop}$). A check for any $E_f$ variation between UD and WR specimens was made using raw data from the maximum load point.
3.3 Results and Discussion

3.3.1 Specimen Flexural Modulus

The variation in flexural modulus, $E_f$, between the UD and WR specimens was found to be small, as illustrated by the values for UD/411-45 and WR/411-45 in Table 3.2. Therefore, as indicated in the work by Hudson et al. [59], the effect of $E_f$ on the $G_{Ie}$ and $G_{IIe}$ results should be negligible.

Table 3.2: UD and WR composite $E_f$ results (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>Mode I</th>
<th>Mode II</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$E_f$ (GPa)</td>
<td>$E_f$ (GPa)</td>
</tr>
<tr>
<td>UD/411-45</td>
<td>27.38 (2.14)</td>
<td>13.49 (0.54)</td>
</tr>
<tr>
<td>WR/411-45</td>
<td>25.18 (3.45)</td>
<td>14.03 (0.66)</td>
</tr>
</tbody>
</table>

3.3.2 Mode I interlaminar fracture toughness

Figure 3.1 shows typical load-displacement profiles for UD/411-45 and WR/411-45. A similar difference in the load-displacement profile was also observed for UD/8084 and WR/8084. It is noted though, that single, planar crack growth was observed in all specimens. The UD specimens were characterised by stable crack propagation and a noticeable fibre bridging zone behind the crack tip. In contrast, the WR composites were characterised by load peaks, at which unstable fracture occurred, and short periods of stable fracture between these peaks. This type of stick-slip behaviour has been reported by Ebeling.
et al. [56] for woven roving composites and by Tohgo et al. [60] for angle-ply composites.
The WR composites also had a comparatively short fibre bridging zone. It was observed, through post-fracture surface examination, that bridging fibres in the WR specimens had been interlocked at warp and weft intersections. These points corresponded to the crack length at which unstable fracture occurred, indicating that simultaneous failure of the interlocked bridging fibres was responsible for unstable crack growth. This is consistent with Tohgo et al. [60], who suggested that stick-slip behaviour in angle-ply composites was caused by the development, then failure, of large scale fibre bridging.

Average values of $n_{f-unstable}$, $\Delta a_{f-unstable}$ and $n_{f-stable}$, per WR specimen, are given in Table 3.3. For WR/8084, $n_{f-unstable}$ is slightly smaller than for WR/411-45, but $\Delta a_{f-unstable}$ is greater. More significant though, is the relatively small $n_{f-stable}$ value
for both WR composites, even with increment marks every 1 mm; the UD composites yielded at least 80 values for a similar crack growth distance.

Table 3.3: Average values of $n_{I-unstable}$, $\Delta a_{I-unstable}$ and $n_{I-stable}$ for WR specimens (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>$n_{I-unstable}$</th>
<th>$\Delta a_{I-unstable}$ (mm)</th>
<th>$n_{I-stable}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>WR/411-45</td>
<td>3 (1)</td>
<td>32 (8)</td>
<td>9 (2)</td>
</tr>
<tr>
<td>WR/8084</td>
<td>2 (1)</td>
<td>40 (6)</td>
<td>7 (1)</td>
</tr>
</tbody>
</table>

Figure 3.2 shows R curve profiles for UD/411-45 and WR/411-45. These profiles are also typical for their 8084 counterparts. The first value in each curve signifies the onset of crack growth, $G_{Ic-init}$. The $G_{Ic-prop}$ values for UD specimens were determined from the clear plateau region on the R curves. However, as indicated in Table 3.3, the R curves for WR specimens contained only a small number of $G_{Ic-stable}$ and $G_{Ic-onset}$ points. It should be noted that some $G_{Ic-stable}$ and $G_{Ic-onset}$ points in Figure 3.2 are located at the same crack length. This is due to the restraining effect of the interlocked bridging fibres, whereby crack length at onset points corresponded to the last recorded crack length during stable growth.

The $G_{Ic}$ results, summarised in Table 3.4, are consistent with the order of matrix toughness (0.16 kJ/m$^2$ for 411-45 and 0.43 kJ/m$^2$ for 8084 [61]). Notably, the absolute $G_{Ic-init}$ values for the WR composites are slightly higher. Martin [57] reported that $G_{Ic-init}$ for woven roving composites would increase if the tip of the artificial starter crack is adjacent to the edge of weft yarns. In this study, post-fracture examination revealed the crack tip
Figure 3.2: Mode I R curves for (a) UD/411-45 and (b) WR/411-45 with $G_{Ic-onset}$ highlighted (●).

was in this position in most specimens. The energy increase from $G_{Ic-init}$ to $G_{Ic-prop}$ is similar for UD/411-45 and UD/8084, indicating that the increase in fracture energy due to fibre bridging is similar in both composites. Table 3.4 also shows that, for the same matrix, $G_{Ic-prop}$ and $G_{Ic-stable}$ are similar. This is reasonable since Ebeling et al. [56] reported that stable crack growth in WR composites occurs mainly in the warp regions. For material comparison however, the results from UD composites are statistically more reliable. For WR/8084, $G_{Ic-onset}$ is similar to $G_{Ic-prop}$ and $G_{Ic-stable}$, but for WR/411-45 $G_{Ic-onset}$ shows an increase, suggesting interlocked fibre bridging had a greater toughening effect with lower matrix toughness.

The fracture surface micrograph for UD/411-45, Figure 3.3(a), shows less matrix defor-
Table 3.4: Composite $G_{Ic}$ results (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>$G_{Ic-\text{init}}$ ($\text{kJ}/\text{m}^2$)</th>
<th>$G_{Ic-\text{prop}}$ ($\text{kJ}/\text{m}^2$)</th>
<th>$G_{Ic-\text{stable}}$ ($\text{kJ}/\text{m}^2$)</th>
<th>$G_{Ic-\text{onset}}$ ($\text{kJ}/\text{m}^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UD/411-45</td>
<td>0.19 (0.02)</td>
<td>0.72 (0.09)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>WR/411-45</td>
<td>0.23 (0.05)</td>
<td>-</td>
<td>0.74 (0.11)</td>
<td>0.92 (0.09)</td>
</tr>
<tr>
<td>UD/8084</td>
<td>0.42 (0.08)</td>
<td>1.02 (0.21)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>WR/8084</td>
<td>0.58 (0.11)</td>
<td>-</td>
<td>0.99 (0.25)</td>
<td>1.04 (0.16)</td>
</tr>
</tbody>
</table>

These observations support the conclusion that $G_{Ic}$ is consistent with matrix toughness. In WR specimens, resin-rich regions, such as that shown in Figure 3.3(c), were found adjacent to weft yarns and in front of the insert film. As reported by Davies et al. [62], resin-rich regions introduce a blunting effect for crack growth. Therefore, specimens with the insert film tip placed adjacent to weft yarns show the increase in $G_{Ic-\text{init}}$.

One further observation for WR composites was that of increased matrix deformation in weft regions, seen by comparing Figure 3.3(d) with 3.3(b). Ebeling et al. [56] suggested that a mixed mode (I/II) loading condition is present in such regions of woven roving composites. They attributed this to the effect of yarn orientation on the stress state at the crack tip, whereby an inclined crack tip in the weft regions induces an element of mode II deformation. They also reported increased toughness where this loading condition was present. In the current study, Figure 3.3(d) suggests that weft regions of the WR composites were also subject to a mixed mode loading condition. However, the results did not show any significant influence of this loading condition on $G_{Ic}$ for the WR composites.
5.3.3 Mode II interlaminar fracture toughness

Typical load displacement profiles for UD/411-45 and WR/8084 are shown in Figure 3.4. The profiles are also typical of crack growth behaviour in UD/8084 and WR/8084.

Figure 3.3: Mode I fracture surface micrographs (crack growth from left to right); (a) UD/411-45, (b) UD/8084, (c) resin-rich region in front of the insert film in WR/8084, and (d) weft region in WR/8084.
3.3.3 Mode II interlaminar fracture toughness

Typical load-displacement profiles for UD/411-45 and WR/411-45 are shown in Figure 3.4. The profiles are also typical of crack growth behaviour in UD/8084 and WR/8084. A short period of stable crack growth was observed between the initiation point and maximum load point. After the maximum load point, crack growth was unstable in the UD specimens but relatively stable in WR specimens. As for mode I, there was no crack branching, and single, planar crack growth was observed in all specimens.

![Figure 3.4: Typical mode II load-displacement profiles for (a) UD/411-45 and (b) WR/411-45, showing the non-linear point (NL), crack growth increments (1, 2, etc.) and the maximum load point (max).](image)

Figure 3.4: Typical mode II load-displacement profiles for (a) UD/411-45 and (b) WR/411-45, showing the non-linear point (NL), crack growth increments (1, 2, etc.) and the maximum load point (max).

The additional \( G_{IIc} \) values obtained from the stable crack growth regions were used to construct R curves for WR/411-45 and WR/8084, shown in Figure 3.5. A \( G_{IIc-prop} \) value was obtained from the plateau region which is evident in each R curve. It was suspected that compressive forces may have reduced crack growth rate, and therefore affected \( G_{IIc} \)
plateau values, as the crack approached the central loading point. However, a typical plot of crack length versus time, Figure 3.6, showed this was not the case as crack growth rate increased with time.

Figure 3.5: Mode II R curves with $G_{IIc\_max}$ highlighted (●): (a) WR/411-45 and (b) WR/8084. Each figure contains results from 5 specimens.

The $G_{IIc}$ results are summarised in Table 3.5. Although they are slightly higher for the composites with the tougher 8084 matrix, the matrix effect is not as significant as in mode I. This is consistent with results obtained by Bradley [20] for brittle and toughened carbon-fibre composites. In most WR specimens, post-test inspection revealed the insert film tip adjacent to weft yarns but, unlike $G_{IC\_init}$, this did not increase $G_{IIc\_init}$ values above those for UD specimens. The $G_{IIc\_max}$ values are higher for the WR composites and a further increase is seen for $G_{IIc\_prop}$. It is notable that $\Delta u_{II\_max}$ is also higher for WR composites in comparison to their UD counterparts.
Figure 3.6: Crack length versus time for a WR/411-45 ENF specimen.

Table 3.5: Composite \( G_{IIc} \) and \( \Delta a_{II-max} \) results (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>( G_{IIc-init} ) (kJ/m(^2))</th>
<th>( G_{IIc-max} ) (kJ/m(^2))</th>
<th>( \Delta a_{II-max} ) (mm)</th>
<th>( G_{IIc-prop} ) (kJ/m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>UD/411-45</td>
<td>0.80 (0.18)</td>
<td>2.69 (0.22)</td>
<td>1 (2)</td>
<td>-</td>
</tr>
<tr>
<td>WR/411-45</td>
<td>0.72 (0.14)</td>
<td>3.34 (0.41)</td>
<td>6 (3)</td>
<td>4.10 (0.47)</td>
</tr>
<tr>
<td>UD/8084</td>
<td>1.37 (0.22)</td>
<td>3.73 (0.34)</td>
<td>4 (1)</td>
<td>-</td>
</tr>
<tr>
<td>WR/8084</td>
<td>1.15 (0.13)</td>
<td>5.00 (0.48)</td>
<td>6 (1)</td>
<td>5.46 (0.41)</td>
</tr>
</tbody>
</table>

The fracture surface micrographs for UD/411-45 and UD/8084, in Figures 3.7(a) and 3.7(b) respectively, show hackle mark deformation. This indicates matrix dominated failure. Figure 3.7(c) shows a weft region in front of the insert film in a WR/8084 specimen. The broken fibres found in this region, and observed in other weft regions along the specimen length, are evidence of fibre bridging during the fracture process. As the bridging fibres did not always fracture simultaneously, they would have restrained and slowed crack growth, and reduced stress in the surrounding matrix. For WR specimens, this re-
sulted in increased stable crack growth between the initiation and maximum load points, see $\Delta a_{II-max}$ in Table 3.5, and the establishment of $R$ curves. The bridging fibres also increased energy absorption during crack growth, thus $G_{IIc-max}$ and $G_{IIc-prop}$ increased. Furthermore, reduced hackle mark density was observed for matrix in the warp regions of WR composites, Figure 3.7(d), in comparison to UD composites, Figure 3.7(b). Since hackle mark density reflects the shear stress intensity in the matrix, this observation suggests that the load carried by the matrix was reduced, possibly due to the formation of bridging fibres.

3.4 Conclusion

The mode I results are consistent with the order of matrix toughness. However, this matrix effect is less significant in mode II. The effect of fibre lay-up on initiation values is not significant in both modes; but the WR fibre affected crack growth behaviour and, therefore, the $G_c$ values for crack propagation.

In UD specimens, mode I crack growth was stable but in WR specimens it was characterised by load peaks and unstable fracture due to the formation, then failure, of interlocked bridging fibres. Consequently, WR specimens yielded comparatively few $G_{Ic}$ values. Therefore, despite having similar values, $G_{Ic-prop}$ for the UD specimens is statistically more reliable than $G_{Ic-stable}$ for the WR specimens.
In mode II, specimens exhibited greater visible crack growth between the initiation and maximum load points and exhibited a higher fatigue strength than UD specimens. However, at the maximum load point, unstable fracture occurred in UD specimens. However, crack growth and stable crack growth in WR specimens exhibited the initiation of B fibres bridging fibres in WR/8084 and (d) warp region in WR/8084.

Figure 3.7: Mode II fracture surface micrographs (crack growth from left to right); (a) UD/411-45, (b) UD/8084, (c) bridging fibres in WR/8084 and (d) warp region in WR/8084.
In mode II, WR specimens exhibited greater stable crack growth between the initiation and maximum load points and yielded a higher $G_{IIc-max}$ value than UD specimens. Beyond the maximum load point, unstable fracture occurred in UD specimens. However, continued stable crack growth in WR specimens enabled the construction of R curves and then the determination of a $G_{IIc-prop}$ value, that showed a further increase from $G_{IIc-max}$. Fracture surface examination indicated weft fibre bridging was responsible for stable crack growth, resulting in greater energy absorption, and higher $G_{IIc}$ in WR specimens. From the application perspective, the results show the use of woven roving lay-ups in composite structures can increase interlaminar fracture toughness. However, for the purpose of material comparison and further studies on toughness transfer, it is concluded that unidirectional specimens will provide more reliable and conservative results.
Chapter 4

The influence of fibre volume fraction

4.1 Introduction

A number of mode I critical strain energy release rate \( (G_{Ie}) \) values can be obtained from the standard Mode I DCB test on unidirectional specimens. They are, for crack initiation (either from a starter film or precrack), the first visual observation of crack growth, a 5% increase in initial compliance, and steady-state crack propagation. As discussed by Williams et al. [63], there is contention over the best value to use. The minimum value philosophy prefers the crack initiation values. However, the 5% offset value is more repeatable [64]. The value for steady-state propagation is useful as it more closely represents how the material will perform in practice, but is dependent on the unpredictable energy absorbing mechanisms of bridging fibre formation and fracture during crack growth.
For complete characterisation therefore, the current standards require that all the aforementioned values of $G_{Ic}$ are quoted whenever possible.

Previous work that discussed the influence of fibre volume fraction ($V_f$) on $G_{Ic}$ has not provided a clear understanding on the change in $G_{Ic}$ with $V_f$. Russell [65] varied fibre volume fraction in unidirectional carbon-fibre/epoxy composites by varying the size of the interlaminar resin-rich region. For the range of $V_f$ studied, 56-69%, the results for $G_{Ic}$ at crack initiation were independent of $V_f$, and it was suggested that this independence is due to a matrix deformation zone which was not affected by the presence of fibres. For steady-state crack propagation, Russell did show a gradual increase in the $G_{Ic}$ with $V_f$ in the range of 56-67%. This is reasonable because, as discussed by Bradley [20], fibre bridging has an enhancing effect in brittle-matrix composites. Therefore an increase in $V_f$ is likely to increase fibre bridging and hence $G_{Ic}$ for steady-state crack propagation. However, such an enhancing effect could not explain a comparatively large increase in $G_{Ic}$ for steady-state crack propagation with only a 2% increase of $V_f$ to 69% in Russell’s results. Bradley [20] also varied the $V_f$ of a carbon-fibre composite, with a rubber-toughened epoxy matrix, by altering the size of the interlaminar resin-rich region. The results showed an increase in $G_{Ic}$ for steady-state crack propagation as $V_f$ decreased. It is believed that the ductility of the matrix made the matrix deformation zone larger than the interlaminar resin-rich region. Therefore, the presence of fibres restricted the energy absorption in the matrix. On the other hand, while studying toughness transfer in brittle and ductile-matrix carbon-fibre composites, Hunston [17] noted no significant effect on $G_{Ic}$ with a
5% variation in fibre volume fraction for a given composite system. For epoxy matrix composites reinforced with glass-fibre fabric, Marom [66] reported no consistent trend in $G_{Ic}$ with a change in $V_f$. Understanding these behaviours requires a more detailed investigation on the influence of fibre volume fraction.

The aforementioned studies used fibre volume fraction measured globally, for the laminate or specimen as a whole. This measure does not provide any information on fibre distribution or uniformity of consolidation in a composite, which is usually characterised by resin-rich regions between fibre-dense intralaminar regions. Davies et al. [67] observed that the mode I crack prefers a path through fibre-dense intralaminar regions in unidirectional carbon-fibre composites. Robinson and Song [68] observed similar behaviour for multidirectional composites. These observations suggest that a more relevant measure of fibre volume fraction should be one taken from the local region through which the crack propagates. If the crack prefers fibre-dense intralaminar regions, then it would be the fibre volume fraction in these regions that will influence $G_{Ic}$, especially the propagation value.

Consistent fibre volume fraction and consolidation may therefore be important for $G_{Ic}$ characterisation. In carbon-fibre composites variations in these parameters can be minimised through the use of prepreg and standard cure cycles, even though resin-rich regions are still likely to exist. For glass-fibre composites made with room-temperature-cure thermoset matrix-resins, such as those used for the research presented in this thesis, the situation is different. When fabricated by hand and consolidated using the vacuum bag technique, the matrix-resin will flow. The amount of flow will depend on resin gel time.
and viscosity, or the length of time the vacuum is applied. Consequently, consistent fibre volume fraction and consolidation between laminates may be difficult to achieve.

This chapter investigates the influence of fibre volume fraction on $G_{Jc}$ characterisation of a unidirectional glass-fibre composite made by hand lay-up. Two measures of fibre volume fraction ($V_f$) are defined; for the composite as a whole ($V_{f\text{-global}}$) and for the intralaminar regions adjacent to the delamination crack plane ($V_{f\text{-intra}}$).

### 4.2 Experimental

#### 4.2.1 Materials and fabrication

Composite laminates were made with 20 plies of the Vetrotex P177 unidirectional fibre and with 411-45 as the matrix. A bulk matrix plate was also cast. Variations in $V_{f\text{-global}}$ of four laminates were achieved by varying the length of time for which the vacuum was held. In fabricating a fifth laminate, the procedure was altered to produce a large difference between $V_{f\text{-global}}$ and $V_{f\text{-intra}}$. It required two lay-up steps. The central region for the laminate was made in one mould. Four plies, with Al film at mid-thickness were laid-up, then evacuated to reduce resin content. Fabrication was completed in a second mould with fibre:resin mass ratio of 1:1.5 for the remaining 16 plies. At the appropriate point in the procedure, the central region was transferred to this mould. The completed laminate was evacuated as before. All five laminates were post-cured at 70-75°C for 3
4.2.2 Fibre volume fraction analysis

Laminate 2\(h\) and \(V_{f\text{-global}}\) values are given in Table 4.1. The \(V_{f\text{-global}}\) values were obtained using equation 2.1 in chapter 2. As mentioned in the previous section, laminates 1-4 were made using the standard procedure and laminate 5 was made by the 2-step procedure.

![Image](image.png)

Table 4.1: Laminate 2\(h\), \(V_{f\text{-global}}\) and \(V_{f\text{-intra}}\) (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Laminate</th>
<th>(2h) (mm)</th>
<th>(V_{f\text{-global}}) (%)</th>
<th>(V_{f\text{-intra}}) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.28 (0.12)</td>
<td>33</td>
<td>38 (2.5)</td>
</tr>
<tr>
<td>2</td>
<td>5.95 (0.03)</td>
<td>39</td>
<td>46 (3.6)</td>
</tr>
<tr>
<td>3</td>
<td>5.09 (0.03)</td>
<td>46</td>
<td>51 (2.3)</td>
</tr>
<tr>
<td>4</td>
<td>4.55 (0.04)</td>
<td>53</td>
<td>54 (1.9)</td>
</tr>
<tr>
<td>5</td>
<td>7.17 (0.16)</td>
<td>34</td>
<td>44 (3.0)</td>
</tr>
</tbody>
</table>

The specimen cross-sections shown in Figure 4.1 highlight, qualitatively, the differences in consolidation responsible for \(V_{f\text{-global}}\) variations. At 33\% (laminate 1), there are distinct resin-rich regions between fibre-dense intralaminar regions. At 53\% (laminate 4), the size of the resin-rich regions has reduced and the number of fibres per unit area in the intralaminar regions has increased. Figure 4.1 also shows that while the plies are not perfectly flat, there is a small degree of intermingling in the through-thickness direction.
Figure 4.1: Specimen cross-sections showing resin-rich interlaminar regions and fibre-dense intralaminar regions for $V_f$-global of (a) 33% (laminate 1) and (b) 53% (laminate 4).

Figure 4.2: Specimen cross-section showing typical area selected for $V_f$-intra calculation.
Values of $V_{f-intra}$ were calculated using a cross-section of each laminate. After polishing with 6 $\mu$m diamond paste, backscattered electron images of plies adjacent to the delamination crack plane were acquired. Such an image, with a typical area selected for $V_{f-intra}$ calculation is shown in Figure 4.2. The public domain NIH Image program (version 1.61) was used to determine fibre area fraction and, hence, fibre volume fraction in the selected area. For the image in Figure 4.2, which is from laminate 3, $V_{f-intra}$ is 53%. Ten such images were used to calculate $V_{f-intra}$ for each laminate and the average values are also given in Table 4.1. Laminates 1 and 5 have a similar average value for $V_{f-global}$ but a 6% difference in $V_{f-intra}$. Laminates 3 and 4 have a 7% difference in $V_{f-global}$ but, within scatter, $V_{f-intra}$ is similar.

4.2.3 Mechanical testing

Bulk matrix modulus ($E$), fracture strength ($\sigma$), fracture strain ($\epsilon$), mode I fracture toughness ($G_{Ic}$), deformation zone size ($r_D$), and composite mode I interlaminar fracture toughness ($G_{Ic}$), were evaluated using the standard procedures in chapter 2.

4.2.4 Crack path examination

The method for determining the path taken by the crack during stable fracture in the composite followed that used by Davies et al. [67]. A fractured specimen was wedged open then cast using a viscous resin in such a way that the resin did not flow to the crack.
tip. Then the specimen was cross-sectioned close to the crack tip and polished until the crack was visible.

4.3 Results and Discussion

4.3.1 Matrix properties

The mean (standard deviation) $G_{Jc}$ and $K_{Jc}$ values obtained from the SENB tests were 0.23 (0.01) kJ/m$^2$ and 0.99 (0.02) MPam$^{1/2}$ respectively. The tensile modulus was 3.4 GPa, fracture strength 71 MPa and fracture strain 2.9%. The estimated deformation zone size $r_D$ was 31 µm.

4.3.2 Composite fracture behaviour

Typical load-displacement plots for each laminate are shown in Figure 4.3. The end of the plot corresponds to 70mm of crack growth. For most specimens crack initiation was unstable. When this occurs the ASTM standard suggests that $G_{Ic-init}$ results taken from the non-linear point are invalid as the instability is likely to be due to a problem with the starter film, such as sticking to the DCB arms, folding during laminate fabrication, or being too thick and creating a large resin rich region. However, no sticking was observed during initial loading, post-fracture examination of the specimen did not reveal any folds and film thickness was consistent with that given in the ESIS protocol. Therefore, $G_{Ic-init}$
was calculated using the unstable non-linear points. This issue is discussed further in section 4.3.3 when the $G_{Ic-init}$ results are presented. Furthermore, the unstable initiation prevented a $G_{Ic-5\%}$ value from being obtained.

Figure 4.3: Typical load-displacement plots for (a) laminates 1 and 5, and (b) laminates 2-4.

Crack meandering was not observed in any specimens, indicating a predominantly planar crack front. However, there were notable differences in crack growth behaviour. For laminate 1, Figure 4.3a, crack growth was mostly unstable but for laminates 2-4, Figure 4.3b, it was stable. This increase in stability corresponded to an increase in the amount of bridging fibres visually observed during testing and indicates a balance in the rate of bridging fibre formation and fracture. Laminate 5 has similar $V_{f-global}$ to laminate 1 but the load-displacement plot shows different fracture behaviour and has therefore been placed in Figure 4.3a for comparison. It is clear that crack growth in laminate 5 is compar-
atively stable, with greater force required to propagate the crack and greater displacement required for the crack to grow 70mm. This difference in behaviour is attributed to a difference in the amount of fibre bridging which, as shown in Figure 4.4, was greater in laminate 5 than in laminate 1.

A typical R curve for each laminate, shown in Figure 4.5, also gives an indication of the influence of fibre bridging. The level of the $G_{1c}$ plateau from which $G_{1c-prop}$ is calculated is consistent with the amount of bridging visually observed during testing. Figure 4.5 illustrates two other noteworthy points. First, the R-curves for laminates 2 and 5 are similar even though Table I shows a difference in $V_f-global$. Second, there is no relationship between the length of the fibre bridging zone and $V_f-global$.

The micrograph in Figure 4.6 shows the crack tip in a specimen from laminate 3. The two plies visible are those adjacent to crack initiation point. The behaviour is typical for specimens from each laminate; that is, during stable growth, the crack preferred a path either along the boundary of the resin-rich and fibre-dense intralaminar region, or within the intralaminar region. In addition, the crack always remained within or at the boundary of the adjacent plies as shown, supporting the earlier observation that crack growth was predominantly planar. Whether in the boundary or intralaminar region, the crack is intersecting fibres which are part of the fibre-dense intralaminar region. This suggests that bridging fibres originate from this region. Therefore if $V_f-intra$ reflects the number of fibres available for bridging, the difference observed in Figure 4.4 is probably due to the higher $V_f-intra$ in laminate 5.
Figure 4.4: Fibre bridging in (a) laminate 1 and (b) laminate 5. The arrow indicates a point 20 mm behind the crack tip.
Figure 4.5: Typical R curves for each laminate. The legend refers to the laminate number.
4.3.3 Influence of $V_{f,\text{res}}$ and $V_{f,\text{fib}}$

The $G_{f,\text{res}}$ and $G_{f,\text{fib}}$ results for each laminate are plotted against $V_{f,\text{res}}$ in Figure 4.7. The $G_{f,\text{res}}$ values are independent of $V_{f,\text{res}}$ (and, therefore, $V_{f,\text{fib}}$), and the average (standard deviation) of the $G_{f,\text{res}}$ values in Figure 4.7(b) is $0.04 (0.01)$ kJ/m$^2$, which is very similar to the $G_{f,\text{res}}$. This independence suggests that the deformation zone was formed without noticeable contributes from the fibres to each component. Further (65) also show an interlaminar trend through a quantitative comparison between Figure 4.6 and Figure 4.7.

Figure 4.6: Crack path along the boundary of the resin-rich and fibre-dense intralaminar regions (1) and within the intralaminar region (2).
4.3.3 Influence of $V_{f-global}$ and $V_{f-intra}$

The $G_{Ic}$ and $E_f$ results for each laminate are plotted against $V_{f-global}$ in Figure 4.7. The $G_{Ic-init}$ value is independent of $V_{f-global}$ (and, therefore, $V_{f-intra}$), and the average (standard deviation) for the $G_{Ic-init}$ values in Figure 4.7(a) is 0.24 (0.01) kJ/m² which is very similar to matrix $G_{Ic}$. This independence suggests that the deformation zone was formed without noticeable interference from the fibres in each composite. Russell [65] also drew this conclusion based on $V_{f-global}$ for carbon-fibre composites even though a quantitative comparison between $G_{Ic-init}$ and matrix $G_{Ic}$ was not made.

![Figure 4.7: (a) $G_{Ic-init}$ and $G_{Ic-prop}$, and (b) $E_f$ as a function of $V_{f-global}$ for laminates 1-4 (open symbols) and laminate 5 (closed symbols). (Error bars signify ±1 standard deviation).](image)

The similarity in matrix $G_{Ic}$ and composite $G_{Ic-init}$ is consistent with previous mode I results for glass-fibre composites with brittle vinyl ester matrices [29, 69] and confirms that $G_{Ic-init}$ for such systems is matrix dominated. It is also understandable therefore
that crack initiation in the composite is unstable if it is dominated by the formation of a small damage zone in a brittle matrix. It should be noted however that $G_{Ic-init}$ and matrix $G_{Ic}$ are similar despite the difference in specimen geometry and crosshead displacement rate for the DCB and SENB tests. Such a similarity may not be achieved for tougher, more ductile, matrices for which toughness is loading rate dependent.

The $G_{Ic-prop}$ values for laminates 1-4 rise with $V_{f-global}$ up to 46%, although the rate of increase is diminishing from one point to the next. From 46 to 52% there is no change in $G_{Ic-prop}$ despite a difference in $V_{f-global}$, while $E_f$ continues to increase with $V_{f-global}$, as shown in Figure 4.7(b). The $G_{Ic-prop}$ result for laminate 5 deviates from the trend in $G_{Ic-prop}$ for laminates 1-4. In fact, it is 50% higher than $G_{Ic-prop}$ for laminate 1 which has similar $V_{f-global}$.

The results in Figure 4.7 show that $V_{f-global}$ can be used to predict the behaviour of $E_f$, which is also a global parameter. However, there is no explanation for the behaviour of $G_{Ic-prop}$ or, therefore, the observed differences in fibre bridging discussed in section 4.3.2. A better explanation is provided by the plot of $G_{Ic-prop}$ against $V_{f-intra}$ in Figure 4.8. It shows that the increase of $G_{Ic-prop}$ is consistent with the increase of $V_{f-intra}$. In the case of laminates 1 and 5, this supports the conclusion that the difference in fibre bridging seen in Figure 4.4 is due to the difference $V_{f-intra}$. In addition, a plateau region in Figure 4.7 may have developed due to the similarity in $V_{f-intra}$ for the laminates in question. To summarise, it seems that $V_{f-intra}$ reflects the number of bridging fibres which form during crack growth. Since fibre bridging was the major energy absorbing mechanism
during crack growth, $G_{Ic-prop}$ is consistent with $V_{f-intra}$ within the range studied.

The ability of $V_{f-intra}$ to explain variations in $G_{Ic-prop}$ has been successful in the current study. It also suggests that with consistent $V_{f-intra}$ in unidirectional specimens, $G_{Ic-prop}$ may be even be treated as a material property. However, it is important to note that there was minimal intermingling of plies in each laminate, and that predominantly planar crack growth was observed through the fibre-dense intralaminar regions adjacent to the crack initiation point. The $V_{f-intra}$ parameter may not be useful if crack growth is predominantly through the resin-rich interlaminar region, or if excessive intermingling of plies varies the extent to which it grows through the intralaminar region. Excessive intermingling may also inhibit calculation of $V_{f-intra}$ by the method presented here. In relation to other work, $V_{f-intra}$ may not explain Russell’s $G_{Ic-prop}$ results [65], since $V_{f-global}$ was varied by varying the thickness of the interlaminar resin-rich region. It would also be dif-
difficult to apply to composites reinforced with fabrics, such as those used by Marom [66]. The calculation of $V_{f-intra}$ would not be straightforward and the fabric structure would inhibit planar mode I crack growth.

This study has also highlighted a need for a consistent hand lay-up fabrication procedure.

### 4.4 Conclusion

The influence of fibre volume fraction on $G_{Ic}$ has been investigated for a glass-fibre composite with a brittle vinyl ester matrix, made by hand lay-up. Two fibre volume fraction parameters were defined; a global value for the composite specimen ($V_{f-global}$) and a value for the fibre-dense intralaminar regions ($V_{f-intra}$). The range of $V_{f-global}$ studied was 32-52%. Crack growth was planar and post-fracture examination confirmed that it preferred a path through the fibre-dense intralaminar regions.

Results for crack initiation ($G_{Ic-init}$) were independent of both fibre volume fraction parameters and similar to matrix resin mode I fracture toughness. This was attributed to the formation of the energy absorbing damage zone ahead of the crack tip in the brittle matrix without interference from the fibres in each composite.

The usual measure of composite fibre volume fraction, $V_{f-global}$, does not fully explain the results for steady-state crack propagation ($G_{Ic-prop}$). Two laminates with the same $V_{f-global}$ had different amounts of fibre bridging and different $G_{Ic-prop}$ results. In addition two laminates with a comparatively large variation in $V_{f-global}$ (7%) gave a similar $G_{Ic-prop}$ result. However, the differences in fibre bridging and $G_{Ic-prop}$ were found to
be consistent with $V_{f-intra}$. It is concluded that a fibre volume fraction parameter in the region of the crack path, in this case $V_{f-intra}$, is more relevant for $G_{Ic}$ characterisation, especially for the $G_{Ic-prop}$ value.

This study has also highlighted the need for a consistent hand lay-up fabrication procedure when producing laminates, such as those used in the following toughness transfer studies, whereby consistent $V_{f-global}$ would also indicate consistent $V_{f-intra}$. Then, for composites with matrices of different toughness, differences in $G_{Ic-prop}$ can be attributed clearly to the difference in matrix toughness rather than a large variation in fibre bridging.

5.1 Introduction

Model matrix and composite toughness transfer predictions. fibre Composites has been studied extensively, most notably by Scott and Philips (20), Benson et al. (19), and Bradley (18). These studies cover a range of unidirectional carbon-fibre composites with brittle matrices. The model and matrix toughness predictions would utilise crack initiation using the DCB specimen geometry. In brittle matrices, anything up to three times the toughness can be fully transferred at crack initiation. Then, as the crack propagates, bridging force from across the delamination plane. The energy absorbed by the formation and fracture of
Chapter 5

Mode I toughness transfer using composite DCB specimens

5.1 Introduction

Mode I matrix-to-composite toughness transfer in carbon-fibre composites has been studied extensively, most notably by Scott and Philips [70], Hunston et al. [17], and Bradley [20]. These works cover a range of unidirectional carbon-fibre composites with brittle-thermoset, toughened-thermoset and thermoplastic matrices tested under static loading using the DCB specimen geometry. In brittle-matrix composites it is clear that toughness is fully transferred at crack initiation. Then, as the crack propagates, bridging fibres form across the delamination plane. The energy absorbed by the formation and fracture
of the bridging fibres has an enhancing effect whereby composite $G_{lc}$ for steady-state crack propagation is greater than matrix $G_{lc}$. With tougher matrices, the absolute value of composite $G_{lc}$ for crack initiation and propagation is higher than for the brittle-matrix composites. However, the transfer is only partial when compared to bulk matrix toughness. The fibres restrict matrix deformation zone development and act as rigid fillers when a large zone extends into the fibre-dense intralaminar regions adjacent to the resin-rich interlaminar region [21, 71, 72].

As mentioned in chapter 1, the potential for further delamination under low-velocity impact loading [2] makes the study of rate effects on composite $G_{lc}$ important. A review on this subject by Cantwell and Blyton [30] shows that the trend in the published results varies, but this may be due to the response of the polymer matrix to the increase in rate. Frassine et al. [31, 32] used crack speed as the parameter for comparison of matrix and composite $G_{lc}$ when studying viscoelastic effects in carbon-fibre/PEI and carbon-fibre/PEEK. Their work showed that both matrix and composite $G_{lc}$ decreased with increasing crack speed. Friedrich et al. [22] conducted a similar rate effect and toughness transfer study on a carbon-fibre/PEEK composite using the crack opening displacement rate as the parameter for direct comparison of composite and matrix $G_{lc}$. Although toughness transfer was partial, the results showed that a decrease in bulk matrix $G_{lc}$ corresponded directly to a reduction in composite $G_{lc}$ with increased crack opening displacement rate. These studies by Frassine et al. and Friedrich et al. show that any combined toughness transfer and rate effect study requires reconciliation of a common
loading parameter in bulk matrix and composite specimens.

Toughness transfer studies in glass-fibre reinforced composites are less extensive. However, the increasing use of these composites for structural applications is leading to more research in this area. For a range of unidirectional glass-fibre composites with brittle polyester, vinyl ester and epoxy matrices Compston et al. [29] drew similar conclusions to those already made for brittle-matrix carbon-fibre composites. Burchill and Simpson [73] also reported that rubber-modified vinyl ester matrices can increase mode I fracture toughness in woven roving glass-fibre composites. A rate effect study by Benmedakhene et al. [74] reported an increase in $G_{IC}$ with crosshead displacement rate for a glass-fibre/epoxy composite, but no comparison to matrix $G_{IC}$ was made. In the case of glass-fibre composites, therefore, further study is still required to assess toughness transfer, and the efficacy of toughened matrices, as loading rate increases.

This chapter presents an empirical study of matrix-to-composite mode I toughness transfer at static and dynamic loading rates in unidirectional glass-fibre reinforced composites with brittle and rubber-toughened vinyl ester matrices. The initial crack opening displacement rate is used as the parameter for comparison of matrix and composite $G_{IC}$ results. In particular, the study investigates toughness transfer at crack initiation in the composite, the enhancing effect of fibre bridging during steady-state crack propagation and the relationship between fibre bridging and matrix deformation zone size.
5.2 Experimental

5.2.1 Materials and fabrication

Bulk matrix plates were cast for 411-45, 8084, 9EXL, 8X33 and 8S22, and 24 plies of Owens Corning R25H fibre were used to fabricate a laminate for each matrix. As discussed in the previous chapter, the fibre:resin mass ratio for each ply was strictly maintained at 1:1 during fabrication to minimise variation in intralaminar fibre volume fraction [75]. The laminates and matrix plates were post-cured at 90°C for 4 hours. Composite thickness, $2h$, and global fibre volume fraction, $V_{f-global}$, values are summarised in Table 5.1 (each composite is identified by its matrix).

<table>
<thead>
<tr>
<th>Composite</th>
<th>$2h$ (mm)</th>
<th>$V_{f-global}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>7.43 (0.04)</td>
<td>38</td>
</tr>
<tr>
<td>8084</td>
<td>6.63 (0.16)</td>
<td>42</td>
</tr>
<tr>
<td>9EXL</td>
<td>7.28 (0.10)</td>
<td>39</td>
</tr>
<tr>
<td>8X33</td>
<td>7.09 (0.06)</td>
<td>40</td>
</tr>
<tr>
<td>8S22</td>
<td>6.99 (0.10)</td>
<td>40</td>
</tr>
</tbody>
</table>
5.2.2 Mechanical testing

Matrix tensile and mode I fracture toughness tests

Matrix tensile and mode I fracture toughness tests were conducted as described in chapter 2. However, the fracture toughness was obtained for two crosshead displacement rates; 0.1 and 10 mm/min. Results were obtained for modulus $E$, yield strength $\sigma$, yield strain $\epsilon$, mode I critical strain energy release rate $G_{Ic}$, mode I critical stress intensity factor $K_{Ic}$, and deformation zone size $r_D$.

Composite mode I DCB tests

A static test was conducted at a crosshead displacement rate of 2mm/min as described in chapter 2 with crack growth monitored visually during the test. A dynamic test was conducted at 1000mm/min which was the highest rate possible on the Instron UTM. These rates produce an initial crack opening displacement rate in the DCB specimens of similar magnitude to that of the static and dynamic tests on bulk matrix SENB specimens. This is important for toughness transfer assessment and is discussed further in section 5.2.3. At 1000 mm/min the crack growth could not be monitored visually during the test, therefore a compliance ($C$) calibration was conducted; where $C = \delta/P$. Each specimen was loaded until a small amount of crack growth was achieved, then unloaded. The initial linear portion of the load-displacement plot provided compliance data for the initial crack length. The new crack length was measured, and the loading-unloading procedure repeated. A
minimum of 4 compliance and crack length data points were obtained from each specimen this way. From simple beam theory:

\[ C \propto a^3 \]  

therefore the compliance calibration data was used to construct a plot of \( C^{1/3} \) versus \( a \), which was then used to retrospectively determine crack length at any given point on the load-displacement plot.

5.2.3 Reconciliation of crack opening displacement rate

Toughness transfer may be assessed by comparing matrix and composite \( G_{ic} \) results and, as shown by Friedrich et al. [22], the crack opening displacement rate can be used as the parameter for the comparison. Such an approach removes the possibility of matrix rate sensitivity affecting the assessment. Friedrich et al. used compact tension specimens to obtain matrix \( G_{ic} \) but the crack opening displacement rate can also be reconciled in matrix SENB and composite DCB specimens [76]. The COD rate \( (C\dot{\delta}D) \) in the DCB specimens was estimated using the expression, derived from beam theory, by Smiley and Pipes [23]:

\[ C\dot{\delta}D = \frac{3 \, c \, h \, d \, x^2}{2 \, a_o^2} \]  

(5.2)
where \( \dot{chd} \) is crosshead displacement rate and \( x \) an arbitrary set distance behind the crack tip, and in this study \( x = 1.08 \text{mm} \). This value of \( x \) was chosen since \( \text{COD} \) from equation 5.2 agrees well with \( \text{COD} \) obtained empirically with a transverse strain sensor placed 1.08mm behind the crack tip on the same glass-fibre/viny. ester DCB specimens [76].

The \( \text{COD} \) in SENB specimens was obtained experimentally using the geometrical method given by Broek [47]:

\[
\text{COD} = \frac{\dot{x}_m}{1 + \left[ \frac{a-x}{x} \right]^{\frac{3}{2}}} \quad (5.3)
\]

where \( \dot{x}_m \) is the opening displacement rate at the mouth of the U-notch. The \( \text{COD} \) was obtained by loading the SENB specimens on the Instron UTM while recording the time required for the notch mouth to open 0.1mm. The opening distance was monitored using an optical microscope. Table 5.2 shows that the \( \text{COD} \) is the same order of magnitude in the SENB and DCB specimens for each loading condition. Toughness transfer will be assessed by comparing composite and matrix \( G_{IC} \) for each loading condition.

<table>
<thead>
<tr>
<th>Loading condition</th>
<th>Test</th>
<th>( chd ) rate (mm/min)</th>
<th>( COD ) rate (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Static</td>
<td>SENB</td>
<td>0.1</td>
<td>6.8 \times 10^{-3}</td>
</tr>
<tr>
<td></td>
<td>DCB</td>
<td>2</td>
<td>1.1 \times 10^{-3}</td>
</tr>
<tr>
<td>Dynamic</td>
<td>SENB</td>
<td>10</td>
<td>6.6 \times 10^{-1}</td>
</tr>
<tr>
<td></td>
<td>DCB</td>
<td>1000</td>
<td>5.7 \times 10^{-1}</td>
</tr>
</tbody>
</table>
5.3 Results

5.3.1 Matrix tensile properties

The matrix tensile properties are given in Table 5.3. The modulus is lower for the toughened systems, and the yield strain is higher. A similar trend is evident for the yield strength except for 411-45 where the value of 62 MPa is lower than that quoted by the manufacturer (81 MPa) [37]. However, all specimens were tested under the same conditions and the low value for 411-45 reflects the difficulty of testing brittle materials. It is also noted that the $\sigma$ and $\epsilon$ values for 411-45 are lower than those obtained in chapter 4. This is attributed to batch variability of the 411-45 resin.

Table 5.3: Matrix $E$, $\sigma$ and $\epsilon$ (±1 standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Matrix</th>
<th>$E$ (GPa)</th>
<th>$\sigma$ (MPa)</th>
<th>$\epsilon$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>3.64 (0.17)</td>
<td>62 (7)</td>
<td>1.96 (0.37)</td>
</tr>
<tr>
<td>8084</td>
<td>3.31 (0.07)</td>
<td>73 (1)</td>
<td>3.99 (0.38)</td>
</tr>
<tr>
<td>9EXL</td>
<td>3.09 (0.05)</td>
<td>64 (1)</td>
<td>2.80 (0.18)</td>
</tr>
<tr>
<td>8X33</td>
<td>2.75 (0.07)</td>
<td>57 (1)</td>
<td>4.26 (0.23)</td>
</tr>
<tr>
<td>8S22</td>
<td>2.70 (0.05)</td>
<td>55 (1)</td>
<td>3.88 (0.08)</td>
</tr>
</tbody>
</table>

5.3.2 Matrix mode I fracture toughness

The fracture behaviour was linear elastic for the 411-45 and 8084 matrices under both loading conditions. Non-linearity was observed for the rubber-modified matrices and the
5% offset point $P_Q$ provided raw data for calculation of $G_{IC}$ in the static and dynamic tests, as illustrated for 8S22 in Figures 5.1(a) and 5.1(b) respectively.

Figure 5.1: Typical load-displacement plots for 8S22 matrix SENB specimens tested under (a) static and (b) dynamic loading.

It is noted that these plots show stable fracture behaviour after $P_{max}$ in the static test but unstable fracture in the dynamic test. Similar behaviour was observed to a lesser degree for 9EXL and 8X33. All the tests satisfied the ASTM validity criterion of $P_{max}/P_Q < 1.1$, although for the static test on 8S22 the validity was borderline, as $P_{max}/P_Q = 1.1$.

The results for matrix $G_{IC}$, $K_{IC}$ and $\tau_D$ are given in Table 5.4. Toughness is clearly improved by the rubber additives, and the increase in damage zone size is consistent with the increase in toughness. The toughness of 8084 is rate sensitive which is consistent with previous work [76], however the toughness of 411-45 and the rubber-modified matrices
shows little rate sensitivity. It is noted that the $G_{IC}$ value for 411-45 is lower than the value obtained in chapter 4, but this may be due to batch variability.

<table>
<thead>
<tr>
<th>Matrix</th>
<th>$G_{IC}$ (kJ/m$^2$)</th>
<th>$K_{IC}$ (MPa m$^{1/2}$)</th>
<th>$r_D$ (µm)</th>
<th>$G_{IC}$ (kJ/m$^2$)</th>
<th>$K_{IC}$ (MPa m$^{1/2}$)</th>
<th>$r_D$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>0.17 (0.01)</td>
<td>0.72 (0.04)</td>
<td>21</td>
<td>0.16 (0.02)</td>
<td>0.71 (0.01)</td>
<td>21</td>
</tr>
<tr>
<td>8084</td>
<td>0.75 (0.10)</td>
<td>1.46 (0.08)</td>
<td>64</td>
<td>0.49 (0.01)</td>
<td>1.20 (0.10)</td>
<td>43</td>
</tr>
<tr>
<td>9EXL</td>
<td>1.35 (0.03)</td>
<td>2.01 (0.03)</td>
<td>159</td>
<td>1.22 (0.05)</td>
<td>1.91 (0.03)</td>
<td>143</td>
</tr>
<tr>
<td>8X33</td>
<td>1.67 (0.07)</td>
<td>1.90 (0.02)</td>
<td>177</td>
<td>1.35 (0.20)</td>
<td>1.77 (0.09)</td>
<td>154</td>
</tr>
<tr>
<td>8S22</td>
<td>1.89 (0.12)</td>
<td>2.13 (0.06)</td>
<td>241</td>
<td>1.97 (0.45)</td>
<td>2.26 (0.26)</td>
<td>272</td>
</tr>
</tbody>
</table>

5.3.3 Composite mode I interlaminar fracture toughness

Under static loading, initial instability in the 411-45 and 8084 composites was followed by relatively stable crack growth, and similar behaviour was observed in the dynamic tests. The initial instability is consistent with the behaviour observed in bulk matrix SENB tests. In the 9EXL, 8X33 and 8S22 composites, crack growth was stable throughout the static test, as illustrated by the typical load-displacement profile for 8S22 in Figure 5.2(a). In the dynamic test however, crack growth was unstable at or just after initiation before becoming stable, as shown Figure 5.2(b). Consequently, $G_{IC-5\%}$ values could only be obtained from static tests on the 9EXL, 8X33 and 8S22 composites. Extensive fibre bridging was observed during the static and dynamic tests and corresponded to stable crack propagation. This stability enabled $R$ curves to be constructed and $G_{IC-prop}$ determined for all specimens.
Figure 5.2: Typical load-displacement plots for 8S22 composite DCB specimens tested under (a) static and (b) dynamic loading.

The composite $G_{Ic}$ results given in Table 5.5 are consistent with order of matrix $G_{Ic}$, indicating that the rubber-modified matrices have toughened the composite. The $G_{Ic-init}$ value for the 8084 composite shows a loading rate effect and this is also consistent with the matrix $G_{Ic}$, but there is no significant matrix or loading rate effect on $E_f$.

The fracture surface micrographs from static test specimens are shown in Figure 5.3. The starter crack tip is shown at the left edge of each micrograph, and the crack growth direction was left to right. The micrographs show greater matrix deformation in the composites with rubber-modified matrices, which is consistent with the higher $G_{Ic}$ results. Examination of the opposite surfaces indicated cohesive matrix failure as the crack propagated from the crack tip, but it is also evident in Figure 5.3 that propagation was towards the
Table 5.5: Composite $G_{IC}$ and $E_f$ results ($\pm 1$ standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>$G_{IC\text{-init}}$ ($\text{kJ/m}^2$)</th>
<th>$G_{IC\text{-5%}}$ ($\text{kJ/m}^2$)</th>
<th>$G_{IC\text{-prop}}$ (GPa)</th>
<th>$E_f$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>0.15 (0.01)</td>
<td>-</td>
<td>0.58 (0.01)</td>
<td>34 (3)</td>
</tr>
<tr>
<td>8084</td>
<td>0.81 (0.08)</td>
<td>-</td>
<td>1.10 (0.19)</td>
<td>34 (3)</td>
</tr>
<tr>
<td>9EXL</td>
<td>0.60 (0.06)</td>
<td>0.82 (0.05)</td>
<td>1.71 (0.30)</td>
<td>33 (3)</td>
</tr>
<tr>
<td>8X33</td>
<td>0.80 (0.11)</td>
<td>1.09 (0.04)</td>
<td>1.80 (0.06)</td>
<td>32 (2)</td>
</tr>
<tr>
<td>8S22</td>
<td>0.94 (0.13)</td>
<td>1.23 (0.08)</td>
<td>2.17 (0.04)</td>
<td>32 (3)</td>
</tr>
</tbody>
</table>

The crack is at the interface in each composite after approximately 100-200 $\mu$m of growth, but the 5% offset in initial compliance for the statically loaded toughened composites corresponded to 1-2mm of crack growth. Therefore, the $G_{IC\text{-5\%}}$ values in Table 5.5 probably include energy absorbed by fibre bridging mechanisms. The interfacial failure shown in Figure 5.3 also suggests that the crack prefers to propagate through the fibre-dense intralaminar regions, and is consistent with previous observations of mode I crack growth in a glass-fibre/vinyl ester composite [75]. The features on the fracture surfaces of the dynamic test specimens, shown in Figure 5.4, are similar to those shown for static test specimens.
Figure 5.3: Fracture surface micrographs showing the initiation region in DCB specimens tested under static loading (crack growth from left to right); (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33 and (e) 8S22.
Figure 5.4: Fracture surface micrographs showing the initiation region in DCB specimens tested under dynamic loading (crack growth from left to right); (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33 and (e) 8S22.
5.4 Discussion

5.4.1 Toughness transfer under static loading

The toughness transfer at crack initiation in the composite has been assessed by plotting $G_{Ic-init}$ against matrix $G_{Ic}$ in Figure 5.5. (The diagonal line drawn across this and subsequent graphs represents a 1:1 relationship between the plotted $G_{Ic}$ values. The error bars signify ±1 standard deviation).

![Graph showing composite $G_{Ic-init}$ versus matrix $G_{Ic}$ - static test results.](image)

Figure 5.5: Composite $G_{Ic-init}$ versus matrix $G_{Ic}$ - static test results.
The scatter in $G_{Ic-init}$ results can be affected by variation in the thickness of the resin-rich interlaminar region, and position of the starter crack in relation to the adjacent fibres. Nevertheless, the trend in toughness transfer is reasonably clear. Transfer is complete in the 411-45 and 8084 composites. In the toughened composites, $G_{Ic-init}$ is less than matrix $G_{Ic}$, indicating partial toughness transfer.

The partial toughness transfer can be explained by comparing the observed deformation zone size, $r_D$, in the toughened matrix and its composite counterpart. The $r_D$ of 8S22 bulk matrix is shown in Figure 5.6(a). It agrees reasonably well with the estimated $r_D$ given in Table 5.4. The $r_D$ observed in the composite DCB specimen, shown in Figure 5.6(b), is slightly smaller than for the bulk matrix. However, comparison of the observed $r_D$ in the composite with a micrograph of the composite cross-section at the same magnification, shown in Figure 5.6(c), suggests that fibres are likely to be contained within the deformation zone. The fracture process for partial transfer in these composites seems therefore, to be consistent with that already described by Jordan and Bradley [21] for toughened carbon-fibre composites. The fibres have restricted energy absorption by acting as rigid fillers within the deformation zone. Then, as the formation of the deformation zone has redistributed load away from the crack tip, the fibres have debonded and initiated crack growth before $r_D$ has developed to the same magnitude as in the bulk matrix. Furthermore, the continuity of the deformation zone in both the matrix and composite is consistent with the stable fracture behaviour observed during the static SENB and DCB tests.
Figure 5.6: Deformation zone radius $r_0$ in (a) 8S22 matrix SENB specimen, (b) 8S22 composite DCB specimen tested under static loading, and (c) cross-section of the 8S22 composite.
Previous work has compared the composite $G_{Ic-prop}$ results to matrix $G_{Ic}$ when assessing toughness transfer and the fibre bridging effect during steady-state crack propagation [17, 20]. Following this approach, $G_{Ic-prop}$ has been plotted against matrix $G_{Ic}$ in Figure 5.7(a). The fibre bridging observed during the DCB tests has enhanced composite $G_{Ic}$ such that $G_{Ic-prop}$ is greater than matrix $G_{Ic}$, but the general trend indicates that the enhancing effect of fibre bridging becomes less significant as matrix $G_{Ic}$ increases.

![Figure 5.7: Composite $G_{Ic-prop}$ versus (a) matrix $G_{Ic}$ and (b) composite $G_{Ic-init}$ - static test results.](image)

However, the partial toughness transfer at initiation in the toughened composites indicates that the maximum contribution to energy absorption by the matrix is more accurately reflected by $G_{Ic-init}$, not matrix $G_{Ic}$, and any increase in composite $G_{Ic}$ above $G_{Ic-init}$ is due mainly to fibre bridging. This suggests an alternative approach to assessing the effect.
of fibre bridging, whereby $G_{IC{\text{-}prop}}$ is plotted against $G_{IC{\text{-}init}}$ as in Figure 5.7(b). It shows that fibre bridging has again had an enhancing effect. However, the trend is opposite to that shown for $G_{IC{\text{-}prop}}$ versus matrix $G_{IC}$ in Figure 5.7a, that is, $G_{IC{\text{-}prop}}$ is increasing with $G_{IC{\text{-}init}}$. This suggests that the enhancement of composite $G_{IC}$ due to fibre bridging is greater than that indicated by comparison of $G_{IC{\text{-}prop}}$ to matrix $G_{IC}$, particularly for the toughened composites.

Following Bradley’s approach [20], the fibre bridging effect is also assessed by expressing $G_{IC{\text{-}prop}}$ as a ratio of matrix $G_{IC}$ and $G_{IC{\text{-}init}}$ in Table 5.6. The $G_{IC{\text{-}prop}}/\text{matrix } G_{IC}$ ratio indicates that the fibre bridging effect is greater in the brittle-matrix composites, and this is consistent with the earlier work on carbon-fibre composites [17, 20]. The values of approximately 1 for the toughened composites suggest the $G_{IC{\text{-}prop}}$ is dominated by the matrix. However, the $G_{IC{\text{-}prop}}/G_{IC{\text{-}init}}$ ratio for the toughened composites is approximately twice the $G_{IC{\text{-}prop}}/\text{matrix } G_{IC}$ ratio, which suggests that $G_{IC{\text{-}prop}}$ is dominated as much by fibre bridging as it is by the matrix. Clearly, for composites with tough matrices, the approach to evaluating the effect of fibre bridging on $G_{IC{\text{-}prop}}$ based on $G_{IC{\text{-}init}}$ provides quite different conclusions to the approach based on the matrix $G_{IC}$.

<table>
<thead>
<tr>
<th>Composite</th>
<th>$G_{IC{\text{-}prop}}$/matrix $G_{IC}$</th>
<th>$G_{IC{\text{-}prop}}/G_{IC{\text{-}init}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>3.48</td>
<td>3.93</td>
</tr>
<tr>
<td>8084</td>
<td>1.52</td>
<td>1.36</td>
</tr>
<tr>
<td>9EXL</td>
<td>1.27</td>
<td>2.88</td>
</tr>
<tr>
<td>8X33</td>
<td>1.08</td>
<td>2.25</td>
</tr>
<tr>
<td>8S22</td>
<td>1.15</td>
<td>2.31</td>
</tr>
</tbody>
</table>
Friedrich et al. [22] explained, qualitatively, that $G_{Ic-prop}$ is a summation of the energy absorbed by a number of fracture mechanisms which occur during steady-state crack propagation, namely, matrix deformation and fracture, and the fibre bridging mechanisms of debonding, pull-out and fracture. Friedrich et al. also suggested that as the deformation zone size increases and encompasses more fibres, the energy absorbed by fibre bridging mechanisms is also likely to increase. This concept is used to provide an explanation for the trend observed in Figure 5.7(b) where evaluation of the fibre bridging effect is based on $G_{Ic-init}$. The $G_{Ic-prop}$ value can be divided into two simpler $G_{Ic}$ components, (i) for matrix deformation and fracture, taken as $G_{Ic-init}$ and (ii) fibre bridging mechanisms, which can be quantified by the difference between average $G_{Ic-prop}$ and $G_{Ic-init}$ values ($G_{Ic-FB}$). The $G_{Ic-FB}$ values are plotted against matrix deformation zone size in Figure 5.8; note that deformation zone diameter, $2r_D$, is used as bridging fibres would have formed from both sides of the delamination plane. It is clear that an increase in $G_{Ic-FB}$ relates to an increase in matrix $2r_D$, indicating that more bridging fibres have formed in the toughened composites than in the brittle-matrix composites.

In absolute terms, therefore, the fibre bridging effect is more significant in the toughened composites. It is noted that Figure 5.6 has already shown $r_D$ to be greater in the bulk matrix than the composite. Nevertheless, Figure 5.8 suggest that an increase in bulk matrix $r_D$ will increase the $r_D$ in the composite, resulting in greater energy absorption by the fibre bridging mechanisms, ie. $G_{Ic-FB}$. This supports the trend shown in Figure 5.7(b) and, consequently, the use of $G_{Ic-init}$ instead of matrix $G_{Ic}$ to assess the fibre
bridging effect.

Energy absorption by fibre bridging mechanisms in unidirectional specimens may also be influenced by fibre-matrix adhesion [78, 79]. Although this study has used vinyl ester based matrices with the same reinforcement, there may be a variation in fibre-matrix adhesion between composites with the rubber-modified matrices and those with just the base resin matrix. However, all the results are clearly consistent with matrix toughness and deformation zone size, indicating that any bond strength variation did not have a significant effect. It is also noted that the alternative approach presented for the study of toughness transfer and the fibre bridging effect uses the $G_{Ic-init}$ value, obtained from the non-linear (NL) point. Davies [64] has reported that the selection of the NL point is operator dependent and leads to a greater scatter in $G_{Ic-init}$ between different research
groups, for the same material, than the $G_{ic-5\%}$ value. Therefore, re-evaluation of the load-displacement plots presented in this study by a different operator may produce different $G_{ic-init}$ results to those given in Table 5.5. However, even if the difference is large, it is unlikely to affect the trend in the results or the general conclusions. Furthermore, $G_{ic-5\%}$ is likely to be influenced by fibre bridging mechanisms and cannot always be obtained, especially at higher loading rates.

### 5.4.2 Toughness transfer under dynamic loading

The composite $G_{ic-init}$ values are plotted against matrix $G_{ic}$ in Figure 5.9. The trend and position of the points are similar to that shown for the static test results, except for 8084 where the matrix $G_{ic}$ is lower due to its rate sensitivity. The toughness transfer for 8084 is not as complete as it was in the static test and this may also be due to the rate sensitivity of the matrix $G_{ic}$. The partial toughness transfer in the toughened composites is due to the same mechanisms already discussed for the static loading condition. Compared to the 8S22 bulk matrix, Figure 5.10(a), the deformation zone in the composite, Figure 5.10(b), is smaller due to the presence of fibres. Further comparison with the composite cross-section in Figure 5.6(c) shows that fibres are also present within the deformation zone.
Figure 5.9: Composite $G_{Ic-init}$ versus matrix $G_{Ic}$ - dynamic test results.
In the dynamic case the deformation zone is not continuous in either the 8S22 matrix or composite. This discontinuity is consistent with the unstable crack growth after \( r_{cr} \) in the SENB test on the toughened matrix and, in just after, the initiation point in the DCB test on the composite and therefore. The loading rate has not affected significantly the magnitude of \( r_{cr} \) in the SENB test, however it does seem to be higher in the composite, that has resulted in a shorter deformation zone and apparent bridging fibres in the matrix. The relationship of this behaviour may be due to a variation in crack speed. However further investigations of this relationship are needed.

The composite DCB specimens, Figure 5.10, are not smooth, the strain of the interface was observed to be increasing in the crack zone.

Figure 5.10: Deformation zone radius \( r_{de} \) in (a) 8S22 matrix SENB specimen, and (b) 8S22 composite DCB specimen tested under dynamic loading.

5.5 Conclusion

The transfer of matrix toughness to composite made from toughened matrix materials (8S22) under static and dynamic loading has been assessed in unidirectional glass-fibre
In the dynamic case the deformation zone is not continuous in either the 8S22 matrix or the composite. This discontinuity is consistent with the unstable crack growth after $P_{max}$ in the SENB test on the toughened matrices and at, or just after, the initiation point in the DCB tests on the composite counterparts. The loading rate has not affected significantly the magnitude of $r_D$ or matrix $G_{Ic}$ as calculated according to the ASTM procedure, however it does seem to have affected fracture behaviour after $P_{max}$. In the composite, this has resulted in a short period of unstable crack growth from the initiation point until sufficient bridging fibres formed to stabilise crack growth. The change in fracture behaviour may be due to a change from ductile to brittle crack growth, caused by increasing crack speed. However, further study is required to provide a full explanation of this behaviour.

The composite $G_{Ic-prop}$ value is plotted against $G_{Ic-init}$ in Figure 5.11. Again due to matrix $G_{Ic}$ rate sensitivity, the position of 8084 has changed compared to the static test results, otherwise the trend in $G_{Ic-prop}$ and the enhancing effect of fibre bridging is similar to that seen for static test results. This trend also indicates that examination of the relationship between $G_{Ic-FB}$ and matrix $2r_D$ would yield similar conclusions to those already made for the static test condition.

5.5 Conclusion

The transfer of matrix toughness to composite mode I interlaminar fracture toughness ($G_{Ic}$) under static and dynamic loading has been assessed in unidirectional glass-fibre
reinforced composites with brittle and rubber-modified vinyl ester matrices. Composite DCB specimens were tested at 2 and 1000 mm/min, and matrix SENB specimens were tested at 0.1 and 10 mm/min. The initial crack opening displacement rate was used as the parameter for comparison of matrix and composite $G_{lc}$ results.

Under static loading, matrix $G_{lc}$ is fully transferred at crack initiation, $G_{lc-init}$, in the composites with brittle matrices. The $G_{lc-init}$ value is increased by the use of rubber-modified matrices, however, the toughness transfer is only partial as the fibres affect matrix deformation zone development. A plot of composite mode I strain energy release rate for steady-state crack propagation, $G_{lc-prop}$, versus matrix $G_{lc}$ indicates the effect of fibre bridging is diminishing with increased matrix toughness. This approach and conclusion are consistent with previous work.

Figure 5.11: Composite $G_{lc-prop}$ versus composite $G_{lc-init}$ - dynamic test results.
However, the partial transfer of matrix toughness at crack initiation in the composite indicates that the maximum contribution to energy absorption by the matrix is more accurately reflected by $G_{l_c-init}$, not matrix $G_{l_c}$. A plot of $G_{l_c-prop}$ versus $G_{l_c-init}$ shows that the enhancing effect of fibre bridging is greater than that shown by $G_{l_c-prop}$ versus matrix $G_{l_c}$. It was found that an increase in energy absorption by fibre bridging mechanisms is consistent with an increase in matrix deformation zone size. It is concluded that an increase in bulk matrix deformation zone size will increase the deformation zone size in the composite. Hence, more fibres are encompassed by the deformation zone, resulting in greater energy absorption through fibre bridging mechanisms. The dynamic tests showed that 8084 matrix $G_{l_c}$ and, consequently, $G_{l_c-init}$ of its composite counterpart, are rate sensitive. Otherwise, the $G_{l_c-init}$ and $G_{l_c-prop}$ results for each composite show no loading rate effect. Toughness transfer and the enhancing effect of fibre bridging were similar to that reported for the static loading condition.

Although toughness transfer is partial, the use of the toughened vinyl ester matrices has increased $G_{l_c}$ for both loading conditions. The efficacy of the rubber-modified matrices at higher loading rates now needs to be assessed.
Chapter 6

Mode I toughness transfer using composite SENB specimens

6.1 Introduction

As discussed in the previous chapter, the difference in crack tip loading rate caused by the difference in composite DCB and matrix SENB or CT specimen geometry can be reconciled using the crack tip opening displacement parameter \[ G_{1c} \]. When this parameter is used for toughness transfer assessment through comparison of matrix and composite \( G_{1c} \), the possibility of matrix toughness rate sensitivity affecting the assessment is removed. However, it is clear that high rate toughness transfer studies would be simpler with matrix and composite SENB specimens of the same geometry. Then, the crosshead displacement rate could be used as the parameter for comparison of the \( G_{1c} \) results.

Recently, three-point bend composite specimens have been used to characterize mode I interlaminar fracture toughness. Hojn [86] used such a specimen under static loading to characterize \( G_{1c} \) in a cross-ply carbon/carbon composite. However, as noted by several researchers, the primary advantage of this specimen lies in the case of testing under impact loading. In fact, Saha et al. [82, 83] have studied dynamic mode I fracture toughness in carbon/epoxy and carbon/epoxy/carbon hybrid composites using 3-point bend specimens in a universal impact test machine to study mode I impact behavior of a glass-fiber/PEEK composite. The key advantage of this approach is that it is similar to the standard SENB specimen used for matrix \( G_{1c} \) evaluation.

This chapter studies the extent of matrix to composite mode I toughness transfer with increasing crosshead displacement rates for matrix and composite SENB specimens of the same dimension. The specimens are tested at crosshead displacement rates ranging from 1 mm/min up to 1.64 m/s.
could be used as the parameter for comparison of the $G_{Ic}$ results.

Recently, three-point bend composite specimens have been used to characterise mode I interlaminar fracture toughness. Hojo [80] tested such a specimen under static loading to characterise $G_{Ic}$ in a cross-ply carbon/carbon composite. However, as noted by Wang [81], the potential for this specimen lies in the ease of testing under impact loading. Indeed, Sohn et al [82, 83] have studied dynamic mode I fracture toughness in carbon-fibre and carbon/kevlar hybrid composites using 3-point bend specimens in a universal testing machine and in a Charpy impact machine. Similarly, Cantwell [84] conducted Charpy impact tests to study mode I impact behaviour of a glass-fibre/PET composite. For toughness transfer studies, the advantage of this specimen is that it is similar to the standard SENB specimen used for matrix $G_{Ic}$ evaluation.

This chapter studies the extent of matrix-to-composite mode I toughness transfer with increased loading rate. Bulk matrix and composite SENB specimens of the same dimension are tested at crosshead displacement rates ranging from 1 mm/min up to 1 m/s.

### 6.2 Experimental

#### 6.2.1 Materials and specimen fabrication

Bulk matrix plates were cast for 411-45, 8084, 9EXL, 8X33 and 8S22, and 36 plies of Owens Corning R25H fibre were used to fabricate a laminate for each matrix. The lam-
inates and matrix plates were post-cured at 90°C for 4 hours. The composite laminates were sectioned then adhered (with super glue), to produce unidirectional SENB specimens with fibres parallel to the \( W \) dimension, as shown in Figure 6.1. The central portion of the specimen corresponded to the initial laminate thickness (2\( h \)). The dimensions of the composite SENB specimens were the same as the matrix specimens, that is, \( W \) 16 mm, \( t \) 8 mm and notch depth 6 mm. After notching, the artificial starter crack length was 2 mm and the crack length (\( a \)) was in the range 0.45 < \( a/W \) < 0.55. Composite thickness 2\( h \) and global fibre volume fraction \( V_{f\text{-global}} \) are summarised in Table 6.1 (each composite is identified by its matrix).

<table>
<thead>
<tr>
<th>Composite</th>
<th>2( h ) (mm)</th>
<th>( V_f ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>9.88 (0.10)</td>
<td>43</td>
</tr>
<tr>
<td>8084</td>
<td>9.69 (0.20)</td>
<td>44</td>
</tr>
<tr>
<td>9EXL</td>
<td>10.11 (0.31)</td>
<td>42</td>
</tr>
<tr>
<td>8X33</td>
<td>10.86 (0.18)</td>
<td>39</td>
</tr>
<tr>
<td>8S22</td>
<td>10.19 (0.15)</td>
<td>41</td>
</tr>
</tbody>
</table>

6.2.2 Mode I fracture toughness testing

Mode I fracture toughness testing of both matrix and composite SENB specimens was conducted in accordance with ASTM standard D5045 [18], as described in chapter 2. The specimens were tested at crosshead displacement rates of 1, 10, 100 and 1000 mm/min and 1 m/s. Tests up to 1000 mm/min were conducted on the Shimadzu UTM. Load-displacement plots were recorded on the digital data recorder at sampling times of 100
ms, 10 ms, 1 ms and 100 µs for 1, 10, 100 and 1000 mm/min respectively. The 1 m/s tests were conducted using an instrumented drop weight impact tester with an optical fibre displacement sensor [85, 86], as shown in Figure 6.2. A strain gauge attached to the falling dart was used to measure load. The optical fibre was fixed on the top of the specimen, close to the impact point. A laser light source was introduced to the fibre and received by a position sensitive detector (PSD) which measured the displacement of the fibre during the test. The load and displacement signals, sampled at 1 µs, were stored in a personal computer. A 1.5 mm thick silicone rubber pad was placed on the specimen at the point of impact to reduce signal oscillation [87]. (It should be noted that the energy absorbed by system compliance, loading pin penetration and sample compression (U_i) could not be obtained for test speeds above 100 mm/min. Therefore U_i for tests above 100 mm/min was estimated by extrapolating the U_i results from the lower rate tests.)

The matrix and composite G_{IC} was evaluated according to the ASTM standard as described in section 2.3.2, that is, G_{IC} at the maximum load point (P_{max}), or the load corresponding to a 5% increase in initial compliance (P_Q), where P_{max}/P_Q < 1.1. However, in composite mode I interlaminar fracture testing, the non-linear point is also used to calculate a G_{IC} value for crack initiation. Therefore, in this study, G_{IC} values from the NL point (G_{IC-NL}) and the 5% offset or maximum load point (G_{IC-offset}) will be reported for matrix and composite. (Note that the composite G_{IC-NL} value used here is equivalent to composite G_{IC-init} obtained from DCB specimens in the previous chapter).
6.3 Results and discussion

6.3.1 Matrix mode | fracture toughness

The influence of matrix toughness on the SENB fracture toughness is shown in Figure 6.4. The fracture toughness estimated at a final compliance of 1 mm/mm for both SENB and SENB specimens is shown in Figure 6.5. However, the other values were obtained at a different final compliance.

Figure 6.1: Composite SENB specimen geometry.

Figure 6.2: Apparatus for drop weight impact testing of SENB specimens.

Bridge circuit
Strain amplifier
Amplifier
Wave memory
PC
Laser diode
Diode driver
Power supply

Rubber pad
Optical fibre
Strain gauge
Falling dart
6.3 Results and discussion

6.3.1 Matrix mode I fracture toughness

The load-displacement plots for 411-45 and 8084 tested from 1 mm/min to 1000 mm/min on the Shimadzu UTM showed linear elastic fracture behaviour. Therefore, only the \(G_{IC-NL}\) result was obtained. For the rubber-modified resins, which exhibited significant non-linear behaviour after the NL point, \(G_{IC-NL}\) and \(G_{IC-offset}\) values were obtained. However, the offset % varied with loading as shown, for example, for 8S22 in Figure 6.3.

![Load-displacement plots for 8S22 matrix SENB specimens tested at (a) 1 mm/min and (b) 1000 mm/min.](image)

Figure 6.3: Load-displacement plots for 8S22 matrix SENB specimens tested at (a) 1 mm/min and (b) 1000 mm/min.

At 1 mm/min a \(G_{IC}\) was evaluated from the \(P_Q\) point: corresponding to a 5% change in initial compliance. At 1000 mm/min on the other hand, fracture occurred at \(P_{max}\) where
the change in compliance was only 1%. This difference in compliance offset % influenced
the trend in results and is discussed further when all the results have been presented.

A typical plot of load and displacement against time, obtained from the instrumented im-
pact tests conducted at 1 m/s, is shown for 8S22 in Figure 6.4(a). There is minimal signal
oscillation and the load-displacement plot shown in Figure 6.4(b) is reasonably linear. As
discussed by Béguelin and Kausch [88], who conducted high rate mode I fracture tests on
polycarbonate CT specimens, the linearity indicates that the loading condition is similar
to a static test even though the rate is higher and equation 2.2, which is based on linear
elastic fracture mechanics, can be used to evaluate $G_{IC}$. The load-displacement profile
in Figure 6.4(b) is typical for all matrix specimens tested at 1 m/s and only $G_{IC} = N_L$ was
obtained (using $P_{max}$).

Figure 6.4: Test data for 8S22 matrix impacted at 1 m/s; (a) load and displacement v.
time, and (b) load v. displacement.
The $G_{lc-NL}$ results are plotted against crosshead displacement rate in Figure 6.5(a). (For clarity in this and subsequent figures, scatter bars are only shown for results with standard deviation greater than 10% of the average value). The $G_{lc-NL}$ is clearly higher in the rubber-modified matrices with 8S22 having highest toughness. The 8084 matrix is rate sensitive, with $G_{lc-NL}$ decreasing linearly with increasing test rate. This is consistent with previous results [76]. Otherwise, the $G_{lc-NL}$ results for 411-45 and rubber-modified matrices show no loading rate effect. The $G_{lc-offset}$ results are presented in Figure 6.5(b). (Note that at 1000 mm/min in 9EXL and 8X33, fracture was linear elastic, therefore $G_{lc-offset}$ was not obtained). After an initial plateau at the lower testing rates, $G_{lc-offset}$ begins to decrease with increasing loading rate. The points in the plateau region were calculated from $P_Q$, indicating a 5% offset in initial compliance. However, as noted earlier, the change in initial compliance did not reach 5% before fracture occurred at the higher test rates, and $G_{lc-offset}$ was evaluated using $P_{max}$. Furthermore, the reduction in offset percentage, as shown alongside the relevant points Figure 6.5(b), is consistent with the reduction in $G_{lc-offset}$ with increased loading rate.

The trends in the results can be explained through examination of the matrix deformation zone. Thin sections were cut, parallel to the $W$ dimension, from 8S22 specimens tested at 1 mm/min and 1 m/s. The sections were polished then examined in transmission mode through an optical microscope. The micrographs are shown in Figure 6.6. At both test rates, the depth of the deformation zone is similar and suggests that the initial response of the matrix is not affected by loading rate. This observation is consistent with the trend
in $G_{ic-NL}$. However, in the 1 mm/min specimen the deformation zone in Figure 6.6(a) is continuous, whereas in the 1 m/s specimen it is discontinuous. Visual measurements from the surface of fractured 8S22 specimens show a decrease in the deformation zone extension with increased loading rate. The extension was 1, 0.6, 0.45 and 0.35 mm for 10, 100 and 1000 mm/min and 1 m/s respectively. At 1 mm/min crack growth was stable and the deformation zone extended through the entire width of the specimen. These observations suggest a change from ductile to brittle crack growth, caused by increasing crack speed. Similar behaviour was observed by Béguelin [85] for high rate tests on rubber-modified PMMA.
Figure 6.6: Deformation zones in 8S22 matrix tested at (a) 1 mm/min and (b) 1 m/s.
The differences in fracture behaviour after the NL point have an effect on the approach taken to characterise high rate $G_{IC}$. The existence of a 5% offset point on a load-displacement plot seems to depend on deformation zone extension, due to crack growth, up to a critical distance. If the critical distance is not reached, the compliance offset % at the fracture point, ie. $P_{max}$, is less than 5%. Then, the trend in $G_{IC}$-offset reflects the variation in offset % at $P_{max}$. In order to test the dependence of $G_{IC}$-offset on deformation zone extension, the offset results were re-evaluated using $P_Q$ for a 1% change in compliance. These results, shown in Figure 6.7, suggest that when the offset % is consistent, there is no effect of loading rate on $G_{IC}$-offset. This concurs with the conclusion suggested by the trend in the $G_{IC}$-NL results. Furthermore, the results suggest that either the NL point or a point defined by a consistent offset % should be used for high rate $G_{IC}$ characterisation.

![Figure 6.7: Matrix $G_{IC}$-offset (1% offset) v. crosshead displacement (chd) rate.](image-url)
6.3.2 Composite mode I interlaminar fracture toughness

The 411-45 composite exhibited linear elastic fracture behaviour, consistent with the bulk matrix. However, the remaining composites showed slightly different fracture behaviour compared to the bulk matrix. For test rates of 1, 10 and 100 mm/min, the 8084 composite showed some non-linear behaviour, as illustrated in Figure 6.8(a), and a $G_{Ic-offset}$ result was obtained. In addition, $G_{Ic-offset}$ results were obtained for 9EXL and 8X33 at 1000 mm/min, and for the 8S22 composite the offset was 5%, as illustrated in Figure 6.8(b), compared to only 1% in the bulk matrix. A typical load-displacement plot for a composite SENB specimen 1 m/s is shown in Figure 6.8(c). Similar to the bulk matrix results, the plot is reasonably linear and only $G_{Ic-NL}$ results were obtained.

The $G_{Ic-NL}$ results are plotted against crosshead displacement rate in Figure 6.9(a). They show that the toughened matrices have increased composite toughness. The 8084 composite shows some rate sensitivity, however the other composites show no loading rate effect. These trends are consistent with bulk matrix $G_{Ic-NL}$. In addition, the $G_{Ic-NL}$ results agree well with $G_{Ic-init}$ results from the mode I DCB tests in chapter 5.
Figure 6.8: Load-displacement plots for composite SENB specimens (a) 8084 tested at 1 mm/min, and (b) 8S22 at 1000 mm/min and (c) 8S22 at 1 m/s.
Figure 6.9: Composite $G_{lc}$: (a) $G_{lc-NL}$ and (b) $G_{lc-offset}$ v. crosshead displacement (chd) rate, (note the offset %).

The composite $G_{lc-offset}$ results are presented in Figure 6.9(b). They are consistent with the order of matrix toughness. The plateau regions for the toughened composites correspond to a consistent 5% offset point. These regions indicate no loading rate effect and the reduction in $G_{lc-offset}$ is consistent with a reduction in offset % as marked alongside the relevant point. These trends are consistent with the bulk matrix $G_{lc-offset}$ results.

The only exception is the 8084 composite. The compliance offset for 8084 was 2% at each rate for which $G_{lc-offset}$ was obtained, and the downward trend of the results is consistent with matrix $G_{lc-NL}$. 

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The electron micrographs in Figure 6.10 show typical fracture surfaces of composite specimens tested at 1 mm/min. There is greater deformation in the composites with the rubber-modified matrices which is consistent with the higher $G_{Ic}$ values. Furthermore, there is similar matrix deformation on the fracture surfaces of specimens tested at 1 m/s, shown in Figure 6.11. This observation supports the conclusion that, for all except 8084, there is no effect of loading rate on $G_{Ic-NL}$.

The $G_{Ic-offset}$ and offset % results in Figure 6.9(b) suggest greater deformation zone extension or crack growth in the composite specimens than in the bulk matrix counterparts. This can also be explained through examination of the micrographs in Figures 6.10 and 6.11. After initial cohesive matrix failure, the crack has grown into the fibre dense intralaminar region after a similar distance in both composites. Therefore, the greater offset % in the composite specimens is most likely caused by bridging fibres, which have increased stable crack growth before increasing crack speed has caused unstable failure. The $G_{Ic-offset}$ values may therefore include energy absorbed by fibre bridging mechanisms such as debonding and pull-out. Overall, the conclusions drawn from the fracture surface examination regarding the order of $G_{Ic}$ and influence of fibre bridging mechanisms on $G_{Ic}$ offset values are consistent with those drawn from the mode I DCB tests.
Figure 6.10: Fracture surfaces of composite SENB specimens tested at 1 mm/min (crack growth from left to right); (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33 and (e) 8S22.
Figure 6.11: Fracture surfaces of composite SENB specimens tested at 1 m/s (crack growth from left to right); (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33 and (e) 8S22.
To be consistent with the analysis of bulk matrix results, composite $G_{Ic-offset}$ was also evaluated for a 1% change in compliance. These results, presented in Figure 6.12, indicate no loading rate effect in the toughened composites; consistent with composite $G_{Ic-NL}$ and the conclusions drawn from the bulk matrix results.

![Graph](image)

Figure 6.12: Composite $G_{Ic-offset}$ (1% offset) v. crosshead displacement (chd) rate.

### 6.3.3 Toughness transfer

As discussed in the previous sections, the selection of a consistent point for $G_{Ic}$ characterisation is important when studying loading rate effects. For toughness transfer assessment therefore, matrix and composite $G_{Ic}$ will be compared for the NL point and, for a consistent offset point, namely, 1% and 5%. Composite $G_{Ic}$ has been plotted against matrix...
$G_{IC}$ in Figure 6.13. (The diagonal line drawn represents a 1:1 relationship between the plotted $G_{IC}$ values). Figure 6.13(a) shows complete toughness transfer at the NL point in the 411-45 and 8084 composites. The downward trend in the points for 8084 reflects the rate sensitivity of the matrix toughness. In the toughened composites, transfer at the NL point is partial. The toughness transfer assessment using 1% and 5% $G_{IC_{-offset}}$ results is presented in Figure 6.13(b). (NB. For the 8084 composite $G_{IC_{-offset}}$ is plotted against matrix $G_{IC_{-NL}}$). The transfer in the 8084 composite is greater than 1, however, this enhancement is most likely due to the extra energy absorbed by fibre bridging mechanisms. The $G_{IC_{-offset}}$ values for the toughened composites are therefore also likely to be affected by fibre bridging. Nevertheless, toughness transfer is partial.

Figure 6.13: Toughness transfer: (a) composite $G_{IC_{-NL}}$ v. matrix $G_{IC_{-NL}}$, and (b) composite $G_{IC_{-offset}}$ v. matrix $G_{IC_{-offset}}$, open symbols for 1%, closed symbols for 5%.
The partial toughness transfer can be explained by examining deformation zone depth in the composite in relation to fibre content. Micrographs from thin sections of the 8S22 composite, tested at 1 mm/min and 1 m/s, are shown in Figure 6.14. The magnification is the same as the bulk matrix micrographs presented previously in Figure 6.6, and the deformation zone depth is similar. Comparison with the composite cross-section of the same magnification, shown in Figure 6.14(c), suggests that fibres are likely to be contained within the deformation zone. The fracture process for partial transfer in these composites seems therefore, to be consistent with that already described by Jordan and Bradley [21] for toughened carbon-fibre composites and for the toughened DCB specimens in chapter 5. That is, the fibres have restrict energy absorption by acting as rigid fillers within the deformation zone. Then, as the formation of the deformation zone redistributes the load away from the crack tip, the fibres debond and initiate crack growth.

Overall, this particular aspect of the toughness transfer research has shown that the composite SENB specimen is useful for high rates mode I fracture studies. One disadvantage though, is that only $G_{IC-NL}$ and $G_{IC-off}$ values can be obtained, whereas the standard mode I DCB composite test can provide $G_{IC-NL}, G_{IC-off}$ and $G_{IC}$ for steady-state crack propagation. However, as discussed by Williams et al. [63], there is contention over the most appropriate value to use for $G_{IC}$ characterisation. The $G_{IC}$ propagation value may represent how the composite performs in practice, but the major energy absorbing mechanism is fibre bridging. The formation of bridging fibres, especially in fabric composites, is unpredictable and may also depend on small variations in intralaminar fibre volume.
fraction as shown in chapter 4. Therefore, the most conservative value is often preferred, and this is provided by $G_{Jc-NL}$.

### 6.4 Conclusion

Mode I toughness transfer in glass-fibre reinforced composites, with brittle and rubber-modified vinyl ester matrices, has been investigated using SENB specimens. Test rates ranged from 1 mm/min to 1 m/s. Toughness transfer was assessed by comparing matrix and composite $G_{Jc}$ for the same test rate.

The matrix $G_{Jc}$ results show that the rubber additives toughened the base resins. When $G_{Jc}$ is evaluated in accordance with the ASTM standard, using the load point $P_Q$ (defined by 5% offset in initial compliance) or the maximum load point $P_{max}$ on a load-displacement plot, the results for toughened matrices suggest a loading rate effect whereby $G_{Jc}$ decreases with increased loading rate. This effect is related to a decrease in deformation zone extension and offset % at $P_{max}$ with increased loading rate. When $G_{Jc}$ is evaluated at the load-displacement non-linear point, or for a consistent offset %, there is no loading rate effect. This conclusion is supported by the deformation zone depth in the 8S22 toughened matrix, which does not vary with loading rate. It is concluded that either the NL point, or a point defined by a consistent offset % should be used for high rate $G_{Jc}$ characterisation.
Figure 6.14: Deformation zones in 8S22 composite tested at (a) 1 mm/min and (b) 1 m/s, and (c) 8S22 composite cross-section.
The trends in composite $G_{lc}$ were similar to those observed for bulk matrix $G_{lc}$. Toughness transfer was complete in the brittle-matrix composites but only partial in the toughened composites. Nevertheless, the toughened matrices still improved composite $G_{lc}$ and show that the rubber-modified matrices would increase mode I delamination resistance in composite structures subject to low-velocity impact.

Chapter 7

The influence of matrix toughness and loading rate on mode II interlaminar fracture toughness

7.1 Introduction

Delamination is one of the major failure modes in composite structures subject to low-velocity transverse impact loading. Initial damage is usually in the form of shear matrix cracks in layers close to the point of impact, or tensile matrix cracks in back-face layers where bending introduces high tension stresses. These matrix cracks then initiate mode II-dominated delamination. The presence of these delaminations within the com-
Chapter 7

The influence of matrix toughness and loading rate on mode II interlaminar fracture toughness

7.1 Introduction

Delamination is one of the major failure modes in composite structures subject to low-velocity transverse impact loading. Initial damage is usually in the form of shear matrix cracks in layers close to the point of impact, or tensile matrix cracks in back-face layers where bending introduces high tensile stresses [2–4]. These matrix cracks then initiate mode II dominated delamination. The presence of these delaminations within the com-
posite structure will adversely affect structural integrity, with compressive strength and fatigue performance most seriously affected [2, 5, 6]. Since impact-induced delamination is mode II dominated [2, 42], high rate test mode II interlaminar fracture tests have been conducted to gain insight into the failure process and potential toughening mechanisms. Under static loading, the use of tougher matrices increases composite mode II interlaminar fracture toughness \( (G_{IIc}) \) [10, 20], and a similar effect has been reported for composite \( G_{IIc} \) at higher rate tests. Smiley and coworkers [22, 24], Maikuma et al. [25] and Blackman et al. [28] have all reported higher \( G_{IIc} \) for tough PEEK-matrix/carbon-fibre composites than for the brittle epoxy-matrix counterparts. Similar conclusions were made by Compston et al. [29] for glass-fibre composites with a range of brittle thermoset matrices.

In a practical situation, the rate at which the composite structure is impacted is unpredictable. Therefore, the effect of a variation in loading rate on mode II delamination resistance is also important. The aforementioned studies, used to highlight the matrix effect, show a decrease in \( G_{IIc} \) with increased loading rate [22, 24, 25, 28, 29]. However, Cantwell [89] has reported an increase in \( G_{IIc} \) for carbon-fibre/PEEK with increased loading rate, as have Todo et al. [90] for a carbon-fibre/polyamide-6 composite. However, Todo et al. also reported a decrease of \( G_{IIc} \) with increasing loading rate when the same polyamide-6 matrix was reinforced with glass-fibre composites. Through fracture surface examination, Todo et al. attributed the different \( G_{IIc} \) trend to variation in dominant the failure mode, with interfacial failure more dominant in the glass-fibre composite and co-
hesive matrix failure more dominant in carbon-fibre composite. It is clear then that large discrepancies in the effect of loading rate exist. This point was made by Cantwell and Blyton [30] in a more extensive review of loading rate effects on composite $G_{IIc}$. However, the authors also concluded that the bulk of published experimental data shows no significant reduction in $G_{IIc}$ due to increased loading rate.

The previous chapters have shown that the addition of rubber to the bulk vinyl ester matrix improves both matrix $G_{Ic}$ and $G_{Ic}$ of its glass-fibre reinforced composite counterpart at static and dynamic loading rates [91, 92]. However, given the susceptibility to impact-induced mode II delamination, this chapter now investigates the influence of matrix toughness and loading rate on composite $G_{IIc}$.

### 7.2 Experimental

#### 7.2.1 Materials

A 24-ply composite laminate was fabricated for each matrix with Owens Corning R25H fibre, and post-cured at $90^\circ$C for 4 hours. Composite thickness, $2h$, and fibre volume fraction, $V_{f\text{-global}}$ are given in Table 7.1.
Table 7.1: Composite $2h$ and $V_{f-global}$, and matrix $G_{lc}$ ($\pm 1$ standard deviation in parentheses).

<table>
<thead>
<tr>
<th>Composite</th>
<th>$2h$ (mm)</th>
<th>$V_{f-global}$ (%)</th>
<th>matrix $G_{lc}$ (kJ/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>411-45</td>
<td>7.23 (0.13)</td>
<td>39</td>
<td>0.16</td>
</tr>
<tr>
<td>8084</td>
<td>6.79 (0.20)</td>
<td>41</td>
<td>0.49</td>
</tr>
<tr>
<td>9EXL</td>
<td>7.09 (0.25)</td>
<td>40</td>
<td>1.21</td>
</tr>
<tr>
<td>8X33</td>
<td>7.16 (0.15)</td>
<td>39</td>
<td>1.35</td>
</tr>
<tr>
<td>8S22</td>
<td>7.10 (0.08)</td>
<td>40</td>
<td>1.97</td>
</tr>
</tbody>
</table>

7.2.2 Mode II interlaminar fracture testing

Mode II tests were conducted using the ENF specimen, in accordance with the ESIS procedure outlined in chapter 2. The influence of loading rate was investigated by testing the specimens at crosshead displacement rates of 1, 10, 100 and 1000 mm/min, and 3 m/s. Tests up to 1000 mm/min were conducted on the Shimadzu UTM. Load-displacement plots were recorded on the digital data recorder at sampling times of 1 s, 100 ms, 10 ms and 1 ms for 1, 10, 100 and 1000 mm/min respectively. The 3 m/s impact tests were conducted using the instrumented drop weight impact tester used in chapter 6 for the mode I SENB tests at 1 m/s. The fixture set-up for the mode II ENF specimen is shown in Figure 7.1. In this case, the optical fibre was fixed on underside of the specimen, directly below impact point. As before, a 1.5 mm thick silicone rubber pad was placed on the specimen at the impact point to reduce signal oscillation [87]. Load and displacement signals were recorded as a function of time at sampling rate of 2 $\mu$s.
Figure 7.1: Apparatus for drop weight impact testing of ENF specimens.
The contribution of kinetic energy $G_{IIc}$ to composite $G_{IIc}$ from the 3 m/s impact was estimated as follows [24, 89]:

$$G_{IIc} = 0.078 \rho h \dot{\delta}^2$$  \hspace{1cm} (7.1)

where $\rho$ is material density and $\dot{\delta}$ crosshead displacement rate. In this study, $G_{IIc}$ was approximately 0.1% of $G_{IIc}$ and hence regarded as negligible.

Results were obtained for $G_{IIc-init}$ and $G_{IIc-max}$. (It is noted that the ESIS protocol also requires a $G_{IIc}$ value for a 5% offset in initial compliance. This value was not obtained for the composites tested in this study as unstable fracture occurred before a 5% increase in compliance.) The flexural modulus, $E_f$, was evaluated using the compliance data from the NL point on the load-displacement plot. Based on the results in chapter 3, crack growth was expected after the NL point. Therefore, following Jar et al. [93] and assuming $E_f$ is independent of crack length, the effective crack length ($a_{eff}$) at the maximum load point was estimated for each test rate using the Russell and Street expression for compliance [54]:

$$C = \frac{2L^3 + 3a_{eff}^3}{8E_f W h^3}$$  \hspace{1cm} (7.2)

where $C$ is compliance at the maximum load point. The influence of matrix toughness was assessed by comparing composite $G_{IIc}$ with the order of matrix $G_{Ic}$ obtained from
the ASTM standard test. Matrix $G_{1c}$ was evaluated in chapter 5 but, for convenience, has been presented again here in Table 7.1.

### 7.3 Results

The load-displacement profiles for 411-45 and 8S22 tested at 1 mm/min, shown in Figure 7.2, are similar. A non-linear (NL) point can be identified and at the maximum load point unstable fracture occurred. This profile is typical for all composites tested up to 1000 mm/min on the Shimadzu UTM.

![Load-displacement profiles](image)

Figure 7.2: Load-displacement plot for composite mode II tests at 1 mm/min (a) 411-45 and (b) 8S22.
A plot of load and displacement against time from a 3 m/s impact test on 8S22 is shown in Figure 7.3(a). After some initial oscillation, the load signal becomes more linear with increasing time. However, there is still some disturbance which hinders the identification of a NL point. On the other hand, the displacement signal shows no oscillation, and just prior to the maximum load point there is a definite inflection point where the displacement shows a sharp increase. Kusaka et al. [94] showed that an inflection point in the signal from a strain gauge attached to the surface of a mode II ENF specimen corresponds to initiation of crack growth. Todo et al. [86] subsequently showed that the inflection points from a strain gauge and the optical fibre displacement sensor correspond well. Therefore, for the 3 m/s test, the inflection point in Figure 7.3(a) was taken to indicate crack initiation. The inflection and maximum load points are magnified in Figure 7.3(b) and the load and displacement data points used for $G_{IIc-init}$ and $G_{IIc-max}$ evaluation are indicated.

The $G_{IIc-init}$ and $E_f$ results are presented in Figure 7.4. The average $G_{IIc-init}$ values show a slight increase with loading rate, however, when scatter is taken into account, this trend is not so conclusive. The $G_{IIc-max}$ and $a_{eff}$ results are presented in Figure 7.5, and they show no significant effect of loading rate or matrix toughness.
Figure 7.3: Test data for 8S22 composite ENF specimen tested at 3 m/s (a) load and displacement v. time, (b) as (a) but with inflection and maximum load points magnified.
Figure 7.4: Composite $G_{IIc-init}$ (closed symbols) and $E_f$ (open symbols) v. crosshead displacement (chd) rate; (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33, and (e) 8S22. (Error bars signify ±1 standard deviation)
Figure 7.5: Composite $G_{IKc-max}$ (closed symbols) and $a_{eff}$ (open symbols) v. crosshead displacement (chd) rate; (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33, and (e) 8S22. (Error bars signify $\pm 1$ standard deviation)
7.4 Discussion

Although the $G_{IIc\text{-init}}$ results indicate a slight rate effect, this trend may be influenced by the definition of initiation. For tests up to 1000 mm/min initiation was defined by the NL point on a load-displacement plot, whereas at 3 m/s it was defined by the inflection point on the displacement-time curve. This difference in approach may be responsible for the trend in Figure 7.4, especially since $G_{IIc\text{-init}}$ obtained from the 3 m/s tests was consistently higher than $G_{IIc\text{-init}}$ from the lower rate tests. Therefore, the following discussion is based only on $G_{IIc\text{-max}}$ results.

7.4.1 Influence of loading rate

The insignificant effect of loading rate on $G_{IIc\text{-max}}$ is consistent with the bulk of previous mode II rate effect studies [30]. Fracture surface micrographs are shown for each composite at the lowest and highest test rates, that is 1 mm/min and 3 m/s, in Figures 7.6 and 7.7 respectively. The micrographs were taken close to the initiation point, corresponding to the region of stable crack growth covered by $a_{eff}$. The crack growth direction was from left to right. Between rates there is no significant difference in matrix deformation which supports the conclusion of no rate effect. However, it was noted by Cantwell [89] that bridging fibres in unidirectional specimens may hide matrix rate sensitivity. Therefore, further tests with offset fibre plies in the delamination crack plane may be required to confirm the current results.
Figure 7.6: Fracture surfaces of composite mode II ENF specimens tested at 1 mm/min; (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33 and (e) 8S22.
Figure 7.7: Fracture surfaces of composite mode II ENF specimens tested at 3 m/s; (a) 411-45, (b) 8084, (c) 9EXL, (d) 8X33 and (e) 8S22.
7.4.2 Influence of matrix toughness

Under mode II loading, sigmoidal-shaped matrix microcracking in the deformation zone ahead of the crack tip is a major energy absorbing mechanism, and these microcracks are responsible for the hackle mark features shown in Figures 7.6 and 7.7. This failure process is more tortuous than in mode I and is the commonly accepted reason for absolute $G_{IIC}$ to be greater than $G_{Ic}$. Furthermore, Bradley [20] has suggested that the microcracking provides similar redistribution of load away from the crack tip in both brittle and toughened matrices. Consequently, the increase in composite $G_{IIC}$ with matrix toughness is not as significant as in mode I. Nevertheless, the bulk of the literature on mode II work shows a matrix effect and given the difference in matrix $G_{Ic}$ for the current glass-fibre/vinyl ester composites, improved $G_{IIC}$ in the toughened composites was expected.

Verification of the unexpected trend in $G_{IIC_{-\text{max}}}$ was sought through microscopic examination of matrix deformation. The fracture surface micrographs show extensive, but similar, matrix hackle mark deformation between composites in both sets of micrographs, which seems to support the conclusion that there is no matrix effect. However, Hibbs et al. [95], who conducted static mode II tests on carbon-fibre reinforced composites with brittle and rubber-toughened epoxy matrices, showed an increase in composite $G_{IIC}$ with matrix toughness even though all fracture surfaces showed hackle mark deformation. Therefore, the similarity in hackle mark deformation is not sufficient on its own to support the trend in $G_{IIC_{-\text{max}}}$. 

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Examination of thin sections from mode I composite specimens in the previous chapters indicated that an increase in composite $G_{Ic}$ with matrix $G_{Ic}$ is related to an increase in deformation zone size. A similar approach was taken here to gain further understanding of the $G_{Ic-max}$ results. Thin sections were cut and polished from the 411-45 and 8S22 composites tested at 1 mm/min and 3 m/s. These specimens were chosen as they represent the extremes of bulk matrix $G_{Ic}$ and test rate. Optical micrographs of the thin sections, taken in transmission mode, are presented in Figure 7.8. They show no significant difference in deformation zone size in either composite at either test rate. Therefore, the results of both the microscopic examination of fracture surface deformation and through-thickness deformation zone size strongly support the conclusion, drawn from the experimental results, that there is no effect of matrix toughness.

Although the trend in $G_{Ic-max}$ is supported by the microscopic examinations, an explanation for the insignificant matrix effect is still required. Todo et al. [96] modelled mode II failure from an artificial starter crack, using experimental data from similar glass-fibre/vinyl ester specimens, and reported crack initiation in the direction of the fibre-matrix interface. In addition, in-situ observation of mode II failure by Friedrich et al. [22] and Bradley [20] showed that the growth of the microcracks ahead of the crack tip, which are responsible for the hackle mark formation, are likely to be impinged by the fibre-dense regions either side of the interlaminar region. It is suspected therefore that mode II failure for these glass-fibre/vinyl ester composites systems is controlled by the interface.
Figure 7.8: Thin sections of mode II ENF specimens showing deformation zone depth in (a) 411-45, test rate 1 mm/min; (b) 8S22, 1 mm/min; (c) 411-45, 3 m/s; (d) 8S22, 3 m/s. Crack growth direction from left to right.
Specifically, the stress concentration at the interface was sufficient to initiate crack growth towards the interface, and then unstable crack growth from the same point (i.e. $a_{eff}$) in each composite, before any significant increase in $G_{IIc}$ due to increased matrix toughness became apparent. The conclusion that the interface in glass-fibre composites has a dominant role in the failure process is consistent with previous work by Todo et al. [90]. Furthermore, all the matrices are vinyl ester based and the interface bond strength is likely to be similar, hence interface controlled failure is likely to produce a similar result in each composite.

It is noted that similar mode II work on glass-fibre composites indicated a loading rate and a matrix effect [29]. However, the variation in matrix toughness was achieved through the use of different thermoset matrices (polyester, vinyl ester and epoxy) and since the fibre was the same in these composites, the interface bond strength would also vary. Therefore, direct comparison with the current results may not be appropriate.

### 7.4.3 Influence of specimen thickness

Previous mode II studies have reported an increase in $G_{IIc}$ with specimen thickness [89, 97]. Davies [98] has subsequently shown that friction between crack faces is significant in thicker specimens, and when a folded PTFE separator film is placed between the crack faces of a 5 mm thick carbon-fibre/PEEK specimen, $G_{IIc}$ is reduced to the value obtained from 3mm thick specimens. It is possible, therefore, that the results in Figures 7.4 and

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7.5 were influenced by specimen thickness or friction, so further investigation was made with specimens cut from the original 7 mm thick laminates, milled to a thickness of 4.5 mm and tested at 1 mm/min on the Instron UTM.

![Figure 7.9](image)

**Figure 7.9**: Composite $G_{IIC-init}$ v. specimen thickness, with test system identified for each set of results and (b) Composite $G_{IIC-max}$ v. specimen thickness.

The current mode II study of brittle and toughened glass-fibre reinforced composites

The $G_{IIC-init}$ results are plotted against thickness in Figure 7.9(a). The results for UD/411-45 and UD/8084 from chapter 3 are also included. The thicker specimens show higher $G_{IIC-init}$, which suggests friction is significant. However, another point of note is that a reliable $G_{IIC-init}$ value is dependent on accurate identification of the NL point, which as shown for mode I [64], may be more difficult to identify than an offset or a maximum load point. Also, as indicated in Figure 7.9(a), the test set-up and data acquisition system
has not been consistent for all the mode II tests, which may be another source of variability for NL identification. Therefore, due to all the above factors, $G_{IIc-init}$ may not be a reliable result to use for the work conducted here.

The unreliability of the $G_{IIc-init}$ results is highlighted even more by the $G_{IIc-max}$ results in Figure 7.9(b), which show no influence of specimen thickness. There is also no influence of matrix toughness which confirms the original results. The reasonably good agreement in $G_{IIc-max}$ between thick and thin specimens indicates no significant friction in the thicker specimens. Where the artificial starter crack is either an Al or polymer film (other than PTFE), Davies [98] suggested that a PTFE separator should be placed between the crack faces. The results for $G_{IIc-max}$ presented here suggest that the PTFE separator is not necessary for the systems studied here.

### 7.4.4 Future work

The current mode II study of brittle and toughened glass-fibre/vinyl ester composites shows no matrix effect, which is an unusual result. Therefore, a number of directions for future work are proposed in order to provide further understanding of the interlaminar fracture behaviour of these composites. Evaluation of bulk matrix $G_{IIc}$ would indicate the efficacy of the rubber-modified matrices under mode II loading, and assist toughness transfer assessment, however there is no standard test method for bulk matrix $G_{IIc}$. Chai [99] has proposed the use of an adhesive bond test to evaluate resin $G_{IIc}$ but this
approach would require detailed study as results can be influenced by bond thickness.

Further interlaminar fracture testing could also be conducted. New configurations for the composite mode II test are under investigation [100]. They include the stabilised end-notch-flexure (SENF) specimen which promotes stable crack growth. This configuration could be utilised to determine if the elimination of unstable crack growth caused by the ENF test enhances the sensitivity of composite $G_{IIc}$ to variations in bulk matrix toughness. Another obvious step to take is mixed-mode testing (I/II). It is believed that tougher matrices improve resistance to impact induced mode II delamination in composite structures [2, 30]. However, the loading conditions under impact are complex [4] and through-thickness damage is likely to result from mixed-mode loading. This has provided the motivation for previous mixed-mode (I/II) studies [28]. Although the $G_{IIc}$ results in this thesis show no matrix effect, the significant improvement in composite $G_{Ic}$ shown in previous chapters may lead to improved results for $G_{I/IIc}$, and hence, impact-induced delamination resistance. For the same reason, the influence of matrix toughness on energy absorption and delamination damage area in plate structures should also be studied.

7.5 Conclusion

The influence of loading rate and matrix toughness on the mode II interlaminar fracture toughness has been investigated in glass-fibre reinforced composites with brittle and rubber toughened vinyl ester matrices. End-notch-flexure specimens were tested at crosshead...
displacement rates ranging from 1 mm/min to 3 m/s.

Composite $G_{IIc}$ at the maximum load point was compared with the order of matrix $G_{Ic}$, and showed no significant effect of loading rate or matrix toughness. Microscopic examination of fracture surfaces and deformation zone depth shows similar deformation features in each composite which do not vary over the range of test rates studied. These observations support the mechanical test results. The absence of a clear loading rate effect is consistent with bulk of published mode II work, but the absence of a clear matrix effect is not. It is suggested that failure in these glass-fibre/vinyl ester composites is interface controlled, with unstable failure initiated before an increase in composite $G_{IIc}$ due to increased matrix toughness becomes apparent.

Overall, the results suggest that the use of rubber-toughened vinyl ester matrices in glass-fibre composites will not improve resistance to mode II dominated impact-induced delamination. However, through-thickness damage in an impacted composite structure is likely to involve mixed-mode loading. It is proposed, therefore, that further work is undertaken to study the matrix effect on composite mixed-mode (I/II) interlaminar fracture toughness, and on delamination resistance in plate structures subjected to transverse impact loading.
Chapter 8

Conclusions and future work

The research conducted in this thesis has assessed matrix-to-composite toughness transfer in the mode I and mode II interlaminar fracture of glass-fibre reinforced composites with brittle and rubber-toughened vinyl ester matrices. Composite structures are susceptible to delamination under low-velocity transverse impact loading, therefore the main aspects considered were the extent of toughness transfer and the influence of loading rate. This chapter summarises the main conclusions and directions for future work.

Two preliminary studies on aspects of mode I and II interlaminar fracture toughness \((G_{Ic} \text{ and } G_{IIc})\) characterisation were conducted. The first investigated the influence of fibre lay-up by comparing \(G_{Ic} \text{ and } G_{IIc}\) of unidirectional and woven roving fibre specimens, the latter lay-up representing more closely in-service composite structures. Results showed unidirectional specimens would provide the most reliable and conservative results.
for further toughness transfer studies. The second study, on the influence of fibre volume fraction, highlighted the importance of a consistent hand lay-up fabrication method, in order to produce consistent fibre volume fraction in each composite. Then, for the further toughness transfer studies, any variation in \( G_{Ic} \), particularly the propagation value, could be directly attributed to the difference in matrix toughness rather than any large variation in fibre bridging.

The details of the mode I and mode II interlaminar fracture tests used for the toughness transfer studies are shown in Table 8.1. Double cantilever beam (DCB) specimens and single-edge-notched bend (SENB) specimens were used for the mode I tests, and end-notch-flexure (ENF) specimens for the mode II tests. In the mode I tests, bulk matrix \( G_{Ic} \) was evaluated using SENB specimens. Hence, a direct comparison of composite and matrix \( G_{Ic} \), using the parameter stated in Table 8.1, allowed a qualitative assessment of toughness transfer. There is no established method for determining matrix \( G_{IIc} \), therefore a qualitative assessment of toughness transfer was made by comparing composite \( G_{IIc} \) with the order of matrix \( G_{Ic} \).

<table>
<thead>
<tr>
<th>Mode</th>
<th>Specimen geometry</th>
<th>Test rates</th>
<th>( G_{Ic} ) comparison parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>DCB</td>
<td>2 &amp; 1000 mm/min</td>
<td>crack opening displacement rate</td>
</tr>
<tr>
<td>I</td>
<td>SENB</td>
<td>1 mm/min - 1 m/s</td>
<td>crosshead displacement rate</td>
</tr>
<tr>
<td>II</td>
<td>ENF</td>
<td>1 mm/min - 3 m/s</td>
<td></td>
</tr>
</tbody>
</table>

For the range of rates covered in mode I and mode II, all results show no significant effect of loading rate. Therefore, the influence of matrix toughness holds for all test rates.
Figure 8.1 summarises the effect of matrix toughness on composite mode I and mode II interlaminar fracture toughness. The legend shows failure mode, test specimen geometry and the values plotted for a particular symbol.

Results for composite $G_{Ic}$ at crack initiation ($G_{Ic-init}$) are plotted against matrix $G_{lc}$ (□). They show an increase in $G_{Ic-init}$ with increased matrix $G_{lc}$. The diagonal line repre-
resents a 1:1 relationship between the plotted $G_{Ic}$ values. It is clear that toughness transfer is complete in the brittle matrix composites but only partial in the toughened composites. This partial transfer is due to the presence of fibres within the energy absorbing matrix deformation zone. Furthermore, it was concluded from the DCB tests that $G_{Ic-init}$ is a more accurate reflection of the maximum contribution of matrix toughness to composite $G_{Ic}$. Therefore, the enhancing effect of fibre bridging was assessed by plotting $G_{Ic-prop}$ against $G_{Ic-init} (O)$. This plot shows greater $G_{Ic-prop}$ in the toughened composites and is related to the larger deformation zone depth in the toughened composites, which encompasses more fibres, thus encouraging greater fibre bridging.

Composite and matrix SENB specimens were used to assess mode I toughness transfer at higher loading rates. It was found that for high rate tests, $G_{Ic}$ should be characterised using the non-linear (NL) point on the load-displacement plot ($G_{Ic-NL}$) (or for a consistent offset in initial compliance). The use of a point corresponding to a 5% offset in initial compliance, as recommended by the ASTM standard for static tests, does not always exist at high loading rates. The existence of the 5% offset point relies on deformation zone extension, which does not always occur due to an increase in crack speed after initiation at high loading rates. When $G_{Ic}$ is evaluated from the NL point, there is no loading rate effect. Composite $G_{Ic-NL}$, plotted against matrix $G_{Ic-NL}$ in Figure 8.1 (△), is higher in the toughened composites. Toughness transfer in the brittle-matrix composites is almost complete, but only partial in the toughened composites, again due to the presence of fibres. Also, $G_{Ic-NL}$ agrees well with $G_{Ic-init}$ obtained from DCB specimens.
The composite $G_{IIc}$ results from the maximum load point are plotted against matrix $G_{Ic}$ (■). There is no significant matrix effect, which is not consistent with bulk of published mode II work. However, in these composites, it is suspected that failure is interface controlled, with unstable fracture initiated before an increase in composite $G_{IIc}$ due to increased matrix toughness becomes apparent.

Knowledge of delamination resistance in composite structures is useful as part of a damage tolerance methodology for assisting design. Measurement of $G_c$ requires a preexisting crack [101], and the damage tolerance methodology is concerned with the load bearing capability of a structure with the assumption of a crack or defect, usually in a critical load-bearing region. This is a reasonable approach since composite structures are susceptible to delamination cracking caused by incidental impacts, such as dropped tools, while in service. Barely visible impact damage (BVID) caused by such impacts is particularly dangerous as it is difficult to detect visually, and other means such as ultrasonic non-destructive testing, are time-consuming and expensive. A design philosophy will often incorporate a need for a composite to demonstrate its ability to maintain structural integrity, with the assumption of a defect in a critical region [102]. For example, the compression after impact (CAI) test is commonly used to assess compressive strength in composites with impact-induced delamination. Stress and strain values from conventional tensile tests can also be used to predict delamination initiation [103]. However, $G_c$ provides more fundamental knowledge on energy absorption at the crack tip and on the likelihood of further delamination. Evaluation of $G_c$ and deduction of toughening
mechanisms from interlaminar fracture toughness tests will, therefore, provide greater confidence in the ability of the composite to retain structural integrity during its expected service lifetime.

Based on the assumption of existing delamination damage, the results of this thesis show that rubber-toughened vinyl ester matrices will enhance resistance to further mode I delamination in glass-fibre composite structures under static or low-velocity impact loading. It is acknowledged that rubber-toughened vinyl ester resins may be more expensive, in terms of material and production costs, than the base resin. Therefore it would be reasonable to restrict the application of rubber-toughened matrices to regions of high mode I loading. On the other hand, the $G_{IIc}$ results in this thesis do not indicate that utilisation of the rubber-toughened vinyl ester will enhance the delamination resistance under further mode II loading.

The explanation for the absence of a matrix effect in mode II would benefit from further investigation. Evaluation of bulk matrix $G_{IIc}$ would be particularly useful. A standard test method for matrix $G_{IIc}$ does not exist, however Chai [104] has shown that an adhesive bond test with a DCB type specimen could be used. This approach could provide a ranking for matrix $G_{IIc}$ and test the ability of the rubber additives to increase energy absorption under mode II loading. Further study could include investigation of the influence of fibre-matrix interface bond strength on $G_{IIc}$. With the improvement in $G_{Ic}$ obtained through the use of the toughened vinyl ester, study of the matrix effect on mixed-mode (I/II) interlaminar fracture toughness is suggested. For the same reason, a study of the ma-
trix effect on energy absorption and delamination resistance in composite plate structures subjected to low-velocity transverse impact loading would also yield useful information. Finally, as glass-fibre/vinyl ester composites are of interest to the marine industry, the effect of long-term exposure to a marine environment on $G_c$ should be investigated, especially for the rubber-toughened composites.
Bibliography


[43] Material synthesised by PJ Burchill according to information provided by BF Goodrich.


