Chapter 3

Standard Delamination Testing

Procedures of Unidirectional Composites

In this chapter some of the standard procedures for delamination testing of unidirectional polymer composites will be examined. The main objective is to explore the possibilities of applying the standard testing methods to interlayer toughened composites and to establish parameters which may influence fracture toughness measurements. Existing standards for mode I and mode II delamination testing will be overviewed, in order to define influential parameters which can help in defining realistic fracture toughness values. Also, these results will be used as a benchmark to estimate fracture toughness improvements due to tough interlayers.
3.1 Background

The goals for any fracture toughness test are to obtain consistent results, to simulate in-situ cracking and to give a fracture toughness value that can reliably characterize tested materials for structural applications. In the case of composite materials all these requirements predominantly depend on the successful simulation of a starter crack. The starter crack must represent a site in a structure where crack initiation is most likely to occur. These sites are usually voids and pre-existing cracks that exist due to manufacturing errors and/or fatigue loading during the service life of the component. Since composites are multi-phase materials successful simulation of a starter crack is not always a straightforward task, and various standards exist to represent this.

Three major standardization organizations: the American Society for Testing and Materials (ASTM), European Structural Integrity Society (ESIS) and Japanese Industrial Standards Group (JIS), developed and adopted similar standards for mode I delamination testing of composite materials. While the ASTM and ESIS [97, 98] standards use a 15\(\mu\)m thick non-adhesive insert film for simulating a starter defect, the JIS standard suggests, in addition to the film, the use of a mode I pre-crack, obtained by clamping the test specimen close to the end of the insert film and than wedging open the specimen. Also, because of this pre-cracking, JIS tests allow the use of a thicker insert film of up to 30\(\mu\)m [99,100]. Thicker insert films allow easier handling during laminate manufacturing, while the sharp pre-crack serves to simulate a real-life crack inside the material. All standards use the same data-reduction procedures.

After initiation of the crack in unidirectional composites, the major cause of resistance to delamination is fiber bridging. This is a phenomenon where unidirectional fibers are pulled out from the matrix during the crack propagation and form bundles which can bridge the crack, slow-down its propagation and absorb vast amounts of
fracture energy. It is noticed that this phenomenon is completely absent during de-
lation between non-unidirectional composite plies [97, 101]. Hence, fibre bridging
can be considered as an artifact of delamination in unidirectional composite materials.
Therefore, any $G_{Ie}$ value calculated beyond the implanted insert (i.e. propagation frac-
ture toughness values) is questionable for composite characterization, and an initiation
value of $G_{Ie}$ is more valuable, as stated in the ASTM standard [97]. The main reason for
this is the fact that unidirectional composites are rarely employed in real-life composite
structures and so, any fracture toughness value which is influenced by fiber bridging
can overestimate fracture toughness and make material characterization unreliable for
real-life structural applications. However, obtaining consistent initiation values for the
mode I strain energy release rate ($G_{Ie}^{\text{ini}}$) is not an easy process. Different problems can
occur due to the presence of the insert film which can make the test results unreliable
and inconsistent. Pre-cracking can solve some of these problems but is likely to produce
undesirable effects, such as fibre bridging, which can adversely effect the testing results.

Generally, it is known that both approaches have advantages and disadvantages.
The main advantage of using the 15µm insert film as a starter crack is in testing
without any special preparation of the specimens. The film is simply embedded in
the mid-region of the composite during manufacturing and specimens can be tested
immediately, without any additional preparation. A reason for using the pre-cracking
technique is that it diminishes the influence of the thick resin-rich region in front of
the film starter crack making crack development more realistic, in terms of in-service
conditions. Also, any film irregularity (waviness, folding or crimping of the film and
bridging of the starter crack during testing) can be avoided by pre-cracking. However,
pre-cracking can invoke fiber bridging prior to testing and hence specimens can exhibit
higher initiation fracture toughness values.
For mode II fracture toughness, there is no ASTM standard testing procedure. Some recommendations from the ESIS protocol [98] suggest the use of both pre-cracked and unpre-cracked specimens, while the JIS standard [102] recommends mode I pre-cracking noting that almost identical results were obtained from the pre-cracked and unpre-cracked specimens. The ambiguity lies in the fact that there still remains some disagreement over whether the lowest (i.e. the most conservative) values of \( G_{IIc} \) are obtained from thin films or from pre-cracks [98].

### 3.2 Materials and specimen preparation

The materials used in this part of the study were Dow Derakane 8084 vinyl-ester resin with unidirectional E-glass (Colan AR106) fiber reinforcement. The fibres were obtained as a woven fabric, and unidirectional fiber plies were made by removing all weft yarns, except two end yarns, which were used for binding. The linear density of the fibers in the warp yarn was 1.2 tex \( (g/m) \), so the areal weight of the unidirectional ply was 354 \( g/m^2 \). Vinyl-ester resin was mixed with additives which serve to control the speed of the resin solidification process, and thus obtain sufficient time for the manufacturing process. For this purpose 1.5\% methyl-ethyl-ketone peroxide (MEKP) was used as a ‘promoter’ (supplied VE was pre-promoted by the manufacturer with 0.3\% of cobalt naphthenate) together with 0.2\% of 2,4pantanedione \((2,4P)\) used as a ‘retarder’ of the chemical reaction.

Each laminate was fabricated by hand in a wet lay-up. Alternate layers of liquid resin and fiber plies were placed inside a dam on a flat mould plate. At the end of the lay up procedure, a caul plate was placed on top of the laminate to insure uniform thickness. The vacuum bagging technique was than applied to cure the laminate under atmospheric pressure and at room temperature. The manufacturing procedure is illus-
trated in Figure 3.1. After an initial room temperature cure in the vacuum bag, each laminate was post-cured at 90°C for 4 hours, in order to obtain uniform properties for the laminate and complete the curing process.

![Mould design for wet lay-up fabrication](image)

Figure 3.1: Mould design for wet lay-up fabrication [103]

Test specimens were cut from the laminates using a water-cooled diamond saw, then dried in a vacuum oven for 12 hours, prior to testing. The fiber volume fraction ($V_f$) for each specimen was calculated using the following formula [98]:

$$V_f = \frac{(FAW)}{(FD) \cdot 2h} \cdot \frac{N}{100\%}$$

where $FAW$ is fiber areal weight (354 $g/m^2$), $N$, the number of plies (20 in this case), $FD$, the fiber density (2.56 $g/cm^3$ for glass [101]) and $2h$, the specimen thickness.

The thickness of all specimens varied from 5.2-5.5mm, and therefore, the fiber volume fraction was kept between 50.28% and 53.18%, thus minimizing its influence on the fracture toughness. A piece of aluminum film coated with mold release agent, 15 $\mu$m thick and 57mm long, was inserted between the mid-plies of each laminate to simulate a crack.
3.3 Experimental procedures

3.3.1 Mode I testing

Two different test procedures were followed for this study. The first one was as reported in the ASTM and ESIS standards [97, 98], and it required specimens with a foil starting defect and without any pre-cracks. The second one followed a slightly modified JIS standard [99, 100], where specimens had to be pre-cracked. Pre-cracking was performed under fatigue mode I loading until the pre-crack length was between 2 and 5mm, and during this process, the cyclic load applied was kept at around 80% of the load required to initiate a crack in the double cantilever beam (DCB) specimen under static loading. This level of loading was found to be sufficient to produce the desired pre-crack length over an acceptable period of time, without causing undesirable premature fracture. A frequency range of 0.5-0.8 Hz was used with an amplitude between 2 and 4mm. Both the pre-cracking and testing were conducted in displacement control on an Instron 4505 Universal Testing Machine, and the typical DCB specimen geometry is shown in Figure 3.2.

![Figure 3.2: The nominal double cantilever beam (DCB) specimen geometry for 60% of glass fibres, suggested by the ESIS standard [98]: initial crack length - $a_0 = 50\text{mm}$, width - $B = 20\text{mm}$, thickness - $2h = 5 \pm 0.1\text{mm}$.](image)

The length of the specimens were 120mm and the width and thickness were 20mm and 5mm, respectively. Loading was applied via the aluminum blocks at the end of
the specimens and the cross-head speed was 1mm/min. Crack propagation was monitored using 1 and 5mm guide marks on the side of specimen, and a load-displacement plot provided data for each crack length used for the calculation of $G_{Ic}$, using the experimental compliance method (Berry’s method) [98], with the standard correction factors for large displacements being used. The strain energy release rate is given by the expression:

$$G_{Ic} = \frac{nP\delta}{2Ba}$$

(3.2)

where $P$ is the applied load, $\delta$ is displacement, $B$ is average width of specimen, $a$ is measured crack length and $n$ is a slope factor calculated from the logarithmic plot of crack length $a$ versus compliance $C$, using the assumption that the compliance is given by the expression:

$$C = K \cdot a^n; \text{ where } n \leq 3$$

(3.3)

Values of $G_{Ic}$ were plotted as a function of crack length, giving a fracture resistance curve (R-curve). The values of $G_{Ic}$ at crack initiation ($G_{Ic}^{ini}$) and after $\sim$50mm of crack growth ($G_{Ic}^{prop}$), when the R-curve reaches its plateau, were of major interest in this study. Crack initiation was defined as the first deviation from linearity on the force-displacement curve.

### 3.3.2 Mode II testing

The geometry of the end-notched flexure (ENF) specimens was the same as that for DCB test. The specimen was placed in a 3-point bend fixture with a half-span length $L$ set to 50mm, as shown in Figure 3.3.
The ratio of the original crack length to half-span length, \(a_o/L\), was 0.5. Testing speed was 1 mm/min and a load-displacement curve was obtained for the calculation of the mode II critical strain energy release rate, \(G_{IIc}\), based on the Direct Beam Theory [98], where \(G_{IIc}\) is expressed as:

\[
G_{IIc} = \frac{9a^2P\delta}{2B(2L^3 + 3a^3)}
\]  

where \(L\), \(a\), and \(B\) are half-span length, starting defect length and width of the ENF specimen, respectively, while \(P\) and \(\delta\) are the force and displacement recorded during the testing. Two values of \(G_{IIc}\) were calculated, initiation values, using values of force and displacement from the first non-linear points on the force-displacement curves; and maximum values, using the maximum force point and the related displacement. These characteristic points are as depicted in Figure 3.4.

Micrographs of the fracture surfaces and side view of the fatigue crack propagation were taken using a Cambridge S360 scanning electron microscope. The specimens were coated with a thin layer of gold. The specimen used for the side view images was carefully polished prior to the testing.
3.4 Results and discussion

3.4.1 Mode I testing

Figure 3.5 illustrates R-curves for the two sets of specimens used in this study. The exact initiation and propagation values of $G_{IC}$ are as given in Table 3.1. Figure 3.5 depicts trend lines calculated by the least square method using the test data from at least five specimens. Both curves are second-order functions in the form of $G_{IC} = Aa^2 + Ba + C$, where A, B and C are calculated constants. The major difference in the results obtained from these two set of specimens can be found in their initiation fracture toughness values ($G_{IC}^{ini}$), when the crack increment is equal to zero (i.e. $a = a_0$). This difference can be explained by the different pre-crack conditions as the fracture behavior of all specimens was similar.

Stability in crack propagation was noticed for all specimens and can be related to
Table 3.1: Average initiation and propagation $G_{It}$ values regarding different pre-crack conditions (standard deviation in parentheses)

<table>
<thead>
<tr>
<th>pre-crack condition</th>
<th>$G_{Im}^{in}/[J/m^2]$</th>
<th>$G_{Ie}^{prop}/[J/m^2]$</th>
<th>spec. tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>foil starter crack</td>
<td>426.6 (26.5)</td>
<td>703.4 (28.8)</td>
<td>12</td>
</tr>
<tr>
<td>foil and fatigue pre-crack</td>
<td>535.1 (23.2)</td>
<td>702.2 (39.7)</td>
<td>5</td>
</tr>
</tbody>
</table>

Figure 3.5: R-curves for specimens with the foil starter crack and specimens with the foil starter crack plus 2mm fatigue pre-cracks

Fiber-bridging phenomena, where fibers are pulled out from the fracture surface and bridge the half-arms of the crack, giving slow and stable propagation. This fracture event was visually observed during the testing, while its influence on the stability of the crack propagation was also reported by Compston and Jar [101]. Also, extensive fiber bridging can be the reason for increases in toughness after the crack initiation, regardless of the pre-crack conditions. It is obvious that the pre-crack conditions did not have any influence on $G_{Ie}^{prop}$. This was expected since crack propagation is hardly dependent upon the starter crack condition after the crack initiation occurs. SEM images of the crack propagation surfaces (for $a=25-30mm$), shown in Figures 3.6(b) and 3.7(b), clearly illustrate broken fibers pulled out from the resin as evidence of the
fiber bridging. This supports the conclusion that fibre bridging predominantly governs the fracture energy absorption processes during crack propagation and that they are not functions of the pre-crack conditions.

Figure 3.6(a), shows the fracture surface ahead of the starter film. The crack rapidly grows towards the resin-fiber interface after a short period of propagation through the thick resin rich region in front of the foil tip, as also reported by Todo and Jar [71]. This interfacial failure continued further with increasing crack length, together with fiber bridging. It can be assumed that an increase in the number of the fibers involved in the fiber bridging process is followed by an increase of $G_{Ic}$, until a plateau value is reached and fiber bridging saturation occurs.

![Figure 3.6](image)

(a) Specimen with foil starter crack. The zone in front of the foil tip shows crack propagation through the resin rich region and subsequent growth towards fibre-resin interface (b) The zone after 25mm of crack growth shows some evidence of fibre bridging in the form of unaligned broken fibres at the fracture surface marked by arrows

The third column in Table 3.1 shows the number of tested specimens required to obtain consistent and acceptable results (all standards request a minimum of five specimens to be tested). The first group of specimens (with the foil insert) exhibited numerous problems during testing. Folding and crimping together with the foil waviness
and bridging of the starter crack were all products of the manufacturing procedure. Therefore, in order to obtain 5 specimens with consistent results, 12 of them had to be tested. This finally gave a standard deviation for the sample of 5 of around ±10%, compatible to those reported elsewhere [97, 101, 104]. With careful foil preparation and handling, before and during the manufacturing, the problems can be diminished but never completely avoided. Conversely, the pre-cracked specimens gave consistent results and did not exhibit any of the aforementioned problems. Obviously, pre-cracking ensured that the foil insert is completely separated from the half-arms of the starter crack along its whole length and that the initial crack extends through the resin rich region. This ensures that neither film insert anomalies nor the resin rich region have any influence on the testing.

![Figure 3.7: Fracture surface micrographs of the specimens with foil insert plus 2mm fatigue pre-crack (crack propagates from left to right): (a) Specimen with foil and fatigue pre-crack (depicted zone is in front of the foil). Fatigue zone near to the fatigue crack tip contains smooth fracture surface without visible damaged fibers ;(b) After 25mm of crack growth fiber bridging is fully active, producing significant number of broken fibers at the fracture surface, marked by arrows](image)

Figure 3.7(a) shows failure due to the fatigue pre-crack loading. No broken fibres were observed on the fracture surface suggesting the absence of fibre bridging. There are two possible explanations for the higher $G_{Ic}^{ini}$ values produced by the pre-cracking.
Firstly, fatigue pre-cracking could cause small amounts of fibre bridging, not visible under the microscope but still sufficient to produce the toughness increase. Secondly, by pre-cracking, a considerable plastic zone can be developed in front of the crack tip. The plastically deformed material around the crack tip can 'blunt' the crack, causing any further crack propagation to be more energy consuming. However, fatigue pre-cracking usually produces very sharp cracks with minimized plastic zone size around the crack tip. Therefore, crack blunting is not expected to significantly influence the results, while fibre bridging can be a possible reason for the noticed fracture toughness increase.

Figure 3.8(a) clearly shows how the fatigue pre-crack grows through the resin rich region. This observation was in agreement with similar analysis done by Kageyama et al. [61] showing that even the mode I pre-crack produced by the wedge-opening method 'prefers' interlaminar, rather than interfacial propagation. This provides evidence that the fatigue pre-crack can negate any anomaly caused by the presence of the foil insert film. Figure 3.8(b) is a high magnification image of the fatigue pre-crack ending in the fibre rich region. Any further crack growth from this point cannot be influenced by the film insert but only by fiber bridging since the crack will clearly interact with fibres causing their breakage and debonding from the matrix.

The problems during this investigation with unpre-cracked specimens are usually observed during any DCB testing [97]. Their existence is inevitable as is their influence on experimental error. Various attempts were made to avoid these problems and their influence. Using a polymer film (PTFE for instance) instead of aluminum foil inserts can solve some of the problems [97], by allowing easier debonding of the film during testing. However, sometimes even that cannot make the definition of $G_{Ic}^{ini}$ consistent enough, due to the existence of the resin rich region in front of the insert tip together
Figure 3.8: Micrographs of the fatigue crack growth path (crack propagates from left to right): (a) the crack path in front of the foil; (b) fatigue pre-crack tip after 2mm of propagation with the influence of the insert tip bluntness [71]. Therefore, the standards have two additional definitions of $G_{Ic}$, beside the $G_{Ic}^{ini}$ defined from the first non-linear point of the load-displacement curve (usually marked as NL). The first is measured at the point where delamination is visually observed and the second at the point where the line that represents the initial compliance increased for 5%, starting from the origin, intersects with the load-displacement plot (or where load has reached a maximum value) [98]. They are usually marked as VIS and 5% offset, respectively, and they are marked on the typical load-displacement curve obtained during DCB testing in Figure 3.9. The last two definitions are meant to be used to minimize any of the insert film influences on fracture initiation. However, a shortcoming of this approach is the very big difference between NL and the other two $G_{Ic}$ values, sometimes as high as 20% [97]. That makes the R-curve almost ‘flat’, where there is almost no difference between VIS and 5% offset values and $G_{Ic}^{prop}$, suggesting that these two definitions of $G_{Ic}^{ini}$ do not represent crack initiation in the best way. The results shown in Figure 3.5 indicate that pre-cracking can solve all problems with the insert film and obtain a properly defined R-curve. It
was noticed during the testing that using the pre-cracked specimens can make the test itself more reliable and easier to perform, without having any of the problems induced by the artificial insert film.

![Diagram](image.png)

Figure 3.9: Typical load-displacement plot under mode I loading - arrows mark the onset of non-linearity (NL), a usual position of visually observed delamination onset (VIS) and 5% offset point (5%). \( C_0 \) is the initial compliance line and \( C_0 + 5\% \) is the initial compliance increased by 5%, [98]

Based on the presented results, fatigue pre-cracking can be introduced as an alternative method for obtaining the NL \((G_{Ic}^{ini})\) values and used together with the visual and 5% offset values of \(G_{Ic}\). These two latter definitions of \(G_{Ic}\) are not based on physical evidence found during delamination onset [97], but are suggested by the standards to increase the consistency of the results. With the pre-cracking technique, the consistency is high without the large overestimation of \(G_{Ic}^{ini}\) observed with the VIS and 5% offset \(G_{Ic}\) values. The shortcoming of this approach is in the highly time-consuming specimen preparation procedure. In this study the pre-cracking was performed using a frequency in the range of 0.5 to 0.8 Hz with an amplitude of approximately 2 to 4mm.
The number of cycles required for the desired crack length was around 20,000, resulting in approximately 8 hours of fatigue loading (per specimen) being necessary to obtain a pre-crack 2-5mm long.

Finally the overall applicability of DCB testing, with and without the pre-cracking, should be mentioned. The word 'applicability' is used here to describe how close laboratory testing is to real-life, in-service conditions. The insert film successfully simulates a void inside the composite which can be, in most cases, the origin for the in-situ crack initiation and growth. It is recommended in the ASTM and ESIS standards that only a 15 µm thick insert film (or thinner) can be used, without pre-cracking, for a realistic simulation. The problem is undesirable resin rich regions which can significantly lower the DCB testing credibility. On the other hand, the existence of small fatigue cracks is also a reality for any real-life composite structure. During the lifetime of the structure, it is more likely that catastrophic failure would occur due to cracks originating from the sharp fatigue cracks than from the blunt voids. However, low and high velocity impacts are also highly likely to occur during the lifetime of the composite structure. An accidental impact by a pebble during aircraft landing or take-off or a tool dropped during a maintenance procedure can be examples of this. These accidents can produce numerous delamination sites by initiating cracking from the pre-existing voids and/or sharp fatigue cracks. Therefore, both approaches have equal credibility because both of them successfully simulate possible failure sites in real-life composite structures and can be equally used for that purpose in mode I delamination testing of unidirectional composites.
Mode II testing

Unlike mode I DCB testing, mode II testing is still not fully standardized by the ASTM. The existing European protocol for composite delamination testing (ESIS) [98] defines two different testing coupon geometries: End-Notched Flexure and End-Loaded Split (ENF and ELS) and three different procedures for specimen preparation. The JIS suggests mode I pre-cracking with the ENF specimen geometry [99, 102]. The ESIS protocol recommends testing of both pre-cracked (under tension or shear) and unpre-cracked specimens for a new material because there are still some uncertainties about which method will give the most relevant initiation values of $G_{IIc}$ [98, 105, 106].

For this study the DCB specimen preparation procedure was followed for mode II ENF specimens. The results are given in Table 3.2.

<table>
<thead>
<tr>
<th>pre-crack condition</th>
<th>$G_{IIc}^{\text{ini}} [J/m^2]$</th>
<th>$G_{IIc}^{\text{max}} [J/m^2]$</th>
<th>spec. tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>foil starter crack</td>
<td>631.5 (137)</td>
<td>3093.2 (196.1)</td>
<td>5</td>
</tr>
<tr>
<td>foil and fatigue pre-crack</td>
<td>547 (140.1)</td>
<td>2757.3 (294.1)</td>
<td>5</td>
</tr>
</tbody>
</table>

Table 3.2: Average initiation and maximum $G_{IIc}$ values regarding different pre-crack conditions (standard deviation in parentheses)

Crack growth for all tested specimens was completely unstable. Also, a considerably larger scatter (compared to the mode I results) was noticed. The results clearly show that the mode II ENF test is not as sensitive to the pre-crack conditions as the mode I DCB test.

Differences between $G_{IIc}$ values of pre-cracked and unpre-cracked specimens cannot be explained simply by the different pre-crack conditions, due to large experimental scatter usually recorded under mode II loading. A support for this conclusion can be found in Figure 3.10 (a) and (b). Apart from the initiation areas, there was no difference in fracture surface morphology between the two specimens. Both fracture surfaces were formed by the characteristic 'hackle' pattern, usually observed after mode II failure.
Figure 3.10: Mode II fracture surface micrographs (crack propagates from bottom to top): (a) specimen with foil and fatigue pre-crack: damage zone near to the fatigue crack tip reveals the smooth fracture under the mode I fatigue loading which transforms to rough ‘hackle’ fracture pattern (b) specimen with foil insert film in front of its tip - the ‘hackle’ fracture pattern is no different to that obtained from the pre-cracked specimens.

The significantly larger scatter for $G_{IIc}^{\text{ini}}$ is a consequence of certain difficulties faced in the acquisition of the non-linear point on the load-displacement curves. The definition of this point, presented in Figure 3.4, plays the most important role in the calculation of $G_{IIc}^{\text{ini}}$. The maximum value of $G_{IIc}$ was defined with much higher accuracy and also exhibited no dependance on pre-cracking.

### 3.5 Summary

Although being standardized, mode I delamination testing remained complex. The use of various crack initiators, like aluminium or PTFE (Teflon) films, can cause fibre disturbance, undesirable resin rich regions and consequently invalid test results. Fatigue mode I pre-cracking was suggested as a promising method in obtaining reliable and consistent fracture toughness results at the point of the crack initiation. The pre-cracking possibly caused some fibre-bridging but not as significant as that observed after pre-cracking by the wedge opening technique as reported elsewhere [100]. Mode
II exhibited very little pre-crack condition sensitivity.

Fatigue pre-cracking can be a useful method for achieving conservative fracture toughness results. However, due to the various obstacles, like complicated and time consuming specimen preparation procedures, in most cases it may not be the best solution for the most effective testing. An alternative, especially when a comparative study is concerned, can be the testing without fatigue pre-cracking by following the existing test standards. Therefore, pre-cracking will not be involved in the following comparative testing of interlayered specimens, but some preliminary results on its influence on fracture toughness will be presented.