NEAR-THRESHOLD FATIGUE OF ADHESIVE JOINTS:
EFFECT OF MODE RATIO, BOND STRENGTH
AND BONDLINE THICKNESS

by:

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Abstract

Near-Threshold Fatigue of Adhesive Joints: Effect of Mode Ratio, Bond Strength and Bondline Thickness

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The main objective of the project was to establish a fracture-mechanics energy-based approach for the design of structural adhesive joints under cyclic loading. This required understanding how an adhesive system behaved near its fatigue threshold, and how the key factors affected this behavior in a fresh undegraded joint. The investigated factors were mode ratio (phase angle), substrate material, surface treatment and surface roughness (both affecting the bond strength), bondline thickness and load ratio.

It was first required to understand how the adhesive system behaved under quasi-static loading by examining a fracture mechanics-based design approach for adhesive systems with different substrate materials and geometries. Experiments were initially performed to characterize the strength of aluminum and steel adhesive systems based on the fracture envelope, critical strain energy release rate as a function of the mode ratio. Ultimate failure loads of aluminum and steel adhesive joints, having different overlap end conditions and different geometries were then experimentally measured. These values were compared with the failure loads extracted from the fracture envelope. Considering the toughening behavior of the adhesive in the fracture mechanics analyses, a very good agreement (average of 6%) was achieved between the predictions and experiments for all types of overlap end conditions and geometries.

Different fatigue threshold testing approaches, which are commonly used in the literature or suggested by the ASTM standard, were evaluated for the cracked and intact fillet joints. Based on the experimental and analytical studies, the most appropriate technique for fatigue testing and characterization of adhesive systems was suggested.
Comparing the mixed-mode near-threshold behavior of different adhesive systems with the fracture behavior and fatigue mode-I and mixed-mode high crack growth rates showed the high sensitivity of the mixed-mode near-threshold fatigue to the subtle changes in the interfacial bond strength.

In order to make a baseline for the design of adhesive joints under cyclic loading, similar to the previous fracture tests and following the energy-based approach, fatigue behavior was characterized as a function of the loading mode ratio for aluminum and steel adhesive joints.

The effect of substrate material, surface treatment, bondline thickness, surface roughness and fatigue testing load ratio on the near-threshold fatigue behavior of adhesives joints was evaluated experimentally. The experimental observations were then explained using finite element modeling.

To generalize the conclusions, the majority of experiments and studies covered a broad range of crack growth rates, as low as fatigue threshold and as high as $10^{-2}$ mm/cycle. Having understood the significant testing and design parameters, an adhesive system can be designed based on a safe cyclic load that produces an insignificant (for automotive industry) or reasonably low but known crack growth rate (for aerospace industry).
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<tr>
<td>CFRP</td>
<td>Carbon Fiber Reinforced Polymer</td>
</tr>
<tr>
<td>FEA</td>
<td>Finite element analysis</td>
</tr>
<tr>
<td>FEM</td>
<td>Finite element method</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>XPS</td>
<td>X-ray photoelectron spectroscopy</td>
</tr>
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</table>

\[\begin{align*} 
&a \quad \text{Crack length} \\
&A_d \quad \text{Damage zone area} \\
&A_r \quad \text{Projected (apparent) profile area} \\
&b \quad \text{Constant in the Paris law equation} \\
&B \quad \text{Specimen width} \\
&C \quad \text{Joint compliance} \\
&c_1-c_4 \quad \text{Regression coefficients for normalized crack length versus compliance equation} \\
&C_1-C_{12} \quad \text{Constants for beam deflection of CLS joints} \\
&d \quad \text{Crack tip distance from the thinner arm of an ADCB} \\
&D \quad \text{Flexural rigidity per unit width} \\
&E \quad \text{Elastic modulus of adherend} \\
&E_a \quad \text{Elastic modulus of adhesive} \\
&E_t \quad \text{Tensile tangent modulus} \\
&F \quad \text{Actuator force in DCB testing} \\
&F_1 \quad \text{Applied force on the upper loading pin of a DCB} \\
&F_2 \quad \text{Applied force on the lower loading pin of a DCB} \\
&G \quad \text{Strain energy release rate} \\
&G_s \quad \text{Shear modulus} \\
&G_I \quad \text{Mode I strain energy release rate} \\
&G_{IC} \quad \text{Mode I critical strain energy release rate} \\
&G_{II} \quad \text{Mode II strain energy release rate} \\
&G_{IIIC} \quad \text{Mode II critical strain energy release rate} \\
\end{align*}\]
$G_a$  Shear modulus of adhesive

$G_c$  Critical strain energy release rate under quasi-static loading

$G_{const}$  Strain energy release rate in which the effect of adherend constraint first becomes evident

$G_{ci}^i$  Crack initiation strain energy release rate under quasi-static loading

$G_{cs}^s$  Steady-state critical strain energy release rate under quasi-static loading

$G_{th}$  Fatigue threshold strain energy release rate

$h$  Adherend thickness

$H$  Lower adherend thickness for DCB and ADCB specimens

$h_l$  Lower adherend thickness

$h_u$  Upper adherend thickness

$l_d$  Damage zone length

$L_{rl}$  Ratio of the actual to the projected profile length in longitudinal direction

$L_{rt}$  Ratio of the actual to the projected profile length in transverse direction

$m$  Slope of the Paris law equation

$M$  Bending moment per unit width

$N$  Number of cycles

$P$  Force per unit width

$P_{Exp}$  Experimental failure load per unit width

$P_i$  Crack initiation force per unit width

$P_{Pred}$  Predicted failure load per unit width

$R$  Force or displacement ratio in a fatigue cycle

$r_p$  Plastic zone radius

$R_a$  Arithmetic average of the roughness profile

$R_{lo}$  Developed length of the roughness profile, based on ISO 4287

$t$  Adhesive thickness

$t_d$  Damage zone thickness

$v$  Beam deflection

$w$  Specimen length from the loading pins (for DCB and ADCB), initial overlap length (for CLS)

$x$  Distance from the crack tip
Greek symbols

\( \alpha \) Calibration constant
\( \delta_{\text{min}} \) Minimum displacement in a fatigue cycle
\( \delta_{\text{max}} \) Maximum displacement in a fatigue cycle
\( \sigma_{\text{eq}} \) Von Mises stress
\( \sigma_p \) Proportional limit of adhesive
\( \sigma_y \) Yield stress
\( \sigma_{yy} \) Opening stress
\( \psi \) Phase angle (mode ratio)
\( \nu \) Poisson ratio
Chapter 1

Introduction

1. Motivation

Adhesively bonded joints promote a more uniform stress distribution than other methods of joining such as bolting, riveting and spot welding which create high stress concentrations. Residual stresses associated with conventional fusion welding are also greatly reduced in adhesive joints. In addition, bonding provides a smoother appearance to the final assembly and the ability to join different materials in complex shapes.

Adhesives are currently used in many areas in the manufacture of automobiles, but mostly in noncritical secondary structures. One of the issues that has limited the use of adhesives is uncertainty about long-term performance under cyclic loads and adverse environmental conditions, and the lack of a solid understanding about parameters that may affect the cyclic behavior of adhesive systems. Cyclic loads can produce fatigue failure in adhesive joints at stress levels much lower than they can withstand under monotonic loading. It is, therefore, of prime importance for automotive engineers to be able to develop and recommend adhesive systems (i.e. the substrate and the adhesive) that will possess an adequate service life under the given operating conditions and applied loads.

A reliable fracture mechanics approach for the design of joints that will experience cyclic loading is based on the threshold strain energy release rate, $G_{th}$, below which no crack propagation occurs. $G_{th}$ and fatigue crack growth rates can be affected by the joint geometry, the properties of the substrate, the loading conditions such as the mode ratio, load ratio, frequency, and environmental parameters such as humidity and temperature. In order to establish an approach for fatigue design of adhesive joints, it is crucial to first understand which parameters are key in defining an adhesive system. This needs to be done by studying the effect of different parameters on the performance of the system. The findings not only can help in establishing the design protocol, but also can guide an engineer to improve the performance of the adhesive system.

Numerous factors have complicated the development of an acceptable fatigue design
procedure for joints in automotive applications: 1) The fatigue behavior of adhesives depends on various adhesive and adherend properties in an unknown way, making it difficult to generalize published results. 2) The influence of loading parameters such as mode ratio, frequency and load ratio, and adhesive system parameters such as bondline thickness and surface roughness are not fully understood. 3) The effect of temperature and humidity on adhesive joint fatigue performance is unpredictable. 4) The majority of research are for fatigue regions above the threshold, i.e. has followed the aerospace approach based on the measurement and prediction of crack growth rates. This may be inappropriate for mass produced automotive designs.

2. Objectives

The objective of this PhD research was to develop a method to predict the maximum cyclic load that can be applied on an adhesively bonded joint with an arbitrary geometry in the room temperature, dry condition. The method should be based on the fatigue threshold, should be applicable to the design of automotive structures with aluminum and steel adherends, and incorporate test data that are relatively straightforward to obtain for new adhesive systems. To achieve this primary objective, using an adhesive with application in automotive industries, the following were found necessary:

(a) Examine and verify a fracture-mechanics design approach for quasi-static loading of highly toughened epoxy adhesives.

(b) Recommend a suitable fatigue threshold testing approach. This included assessing different threshold testing techniques and the validity of the measured $G_{th}$ for the design of joints with different initial conditions of the overlap end.

(c) Develop a similar energy-based fracture-mechanics design approach for cyclic loading of the tested adhesive system. This included the characterization of the energy-based design parameter under cyclic loading.

(d) Identify and understand the effect of loading factors (mode ratio and load ratio) and adhesive system parameters (adhesive fillet, bondline thickness, substrate material and surface roughness) on the design criterion parameter, $G_{th}$. To further extend the applicability of this research, the majority of studies covered both the threshold and cyclic crack growth rates.

3. Thesis Outline

In order to accomplish these objectives, it was required to establish the adhesive system,
types of specimens, surface treatment technique and specimen preparation approach. This was guided by first evaluating an energy-based fracture mechanics approach to the prediction of adhesive joint failure loads under quasi-static loading. Quasi-static fracture tests were performed to characterize the fracture behavior of aluminum and steel adhesive systems by defining a fracture envelope; i.e. the steady-state critical strain energy release rate versus the loading phase angle. The fracture envelope was then used to predict the final failure load for different geometries of typical aluminum and steel fracture joints (cracked lap shear and single lap shear) with different overlap initial conditions. The predictions were then compared with experimental results. The results are presented in Chapter 2.

In collaboration with a research associate from the NRC Aluminum Technology Centre and industrial members of the research team, various elements of the aforementioned quasi-static testing and analyses have been presented in *Engineering Fracture Mechanics*¹ and several conference presentations².

The load frames for the fatigue tests were located at Engineering Materials Research, Downsview, ON. However, no crack length measurement apparatus and fatigue testing control systems were available. Different crack length measurement techniques were assessed for accuracy, convenience and price. The fatigue testing control software and the capability to remotely control the fatigue experiments were developed as part of the project.

Aluminum and steel joints were used to study the effect of mode ratio and interfacial bond strength on the fatigue threshold and crack growth rate. Four different adhesive systems were produced, each with different failure modes, interfacial or cohesive. This was accomplished by changing the substrates, surface treatment and adherend surface roughness. As with the fracture envelope, a fatigue threshold envelope was developed for fatigue design of adhesive joints. The analytical solutions were developed for the calculation of the strain energy release rate for the double cantilever beam, asymmetric double cantilever beam and cracked lap

shear joints used in the experiments. These fatigue experiments and analyses are presented in Chapter 3. The contents of the chapter have been published in *Engineering Fracture Mechanics*¹, and it was partly presented in the 19th *Canadian Materials Science Conference*².

Chapter 4 considers the fatigue threshold and slow crack growth rate behavior of a highly-toughened epoxy adhesive as a function of the starting condition (fatigue precrack and fillet), testing approach and interfacial bond strength. The experiments in Chapter 3 were conducted following the force-controlled threshold testing approach as per an ASTM standard. However, Chapter 4 compares different threshold testing techniques to suggest the most suitable one for the testing and design of adhesive joints. The contribution of the crack initiation phase to the fatigue life of a joint is assessed, and the suitability of defining the threshold based on the ASTM test, which ignores the fatigue crack initiation phase, is discussed. Finally, the effect of interfacial bond strength on fatigue and fracture was investigated by changing the adhesive batch, adherend roughness and surface pretreatment. Chapter 4 has been published in the *International Journal of Adhesion and Adhesives*³.

Fatigue threshold measurements in the thesis are performed at a constant load or displacement ratio. An appendix in Chapter 4 investigates the dependency of the measured $G_{th}$ on the load or displacement ratio. Different approaches to present the fatigue results are considered, and an approach which eliminates the effect of load and displacement ratio is presented.

One of the key experimental observations of Chapters 3 and 4 was the dependency of the mixed-mode crack path on the fatigue crack growth rate or equivalently, the applied strain energy release rate. Such dependency attributed significantly to the mixed-mode near-threshold fatigue behavior of adhesive joints. Chapter 5 presents some hypotheses to explain this behavior, which will be presented in the 33rd *Annual Meeting of the Adhesion Society*⁴.

After establishing the fatigue design criterion and the experimental approach to measure

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the required data, $G_{th}$ or $G$ at a given $da/dN$, the effect of different adhesive system parameters was investigated. Chapters 6 and 7 present the effects of adhesive thickness on both the fatigue and fracture of the adhesive joint. Chapter 6 focuses on the test results and has been submitted to *Engineering Fracture Mechanics*\(^1\). Some elements of this chapter have also been presented in a conference\(^2\).

Chapter 7 presents finite element analyses of the experimental trends reported in Chapter 6, explaining the results in terms of the effect of adhesive thickness and the applied strain energy release rate on the stress distribution in the bondline, the stress triaxiality at the crack tip, and the damage zone size in the adhesive layer. Chapter 7 has also been submitted to *Engineering Fracture Mechanics*\(^3\).

The effects of surface treatment and adhesive batch variation on interfacial bond strength and fatigue were studied in Chapters 3 and 4. Chapter 8 seeks to understand if surface roughness, another factor that can affect the bond strength, is a significant design parameter and whether changing surface roughness is a relatively cost-effective approach to improve the fatigue performance of adhesive joints. These fatigue studies covered both the fatigue threshold strain energy release rate, $G_{th}$, and fatigue crack growth rates, while the strain energy release rate for crack initiation, $G_c^i$, and the steady-state value, $G_c^s$, were measured under quasi-static loading. Finite element model modeling, scanning electron microscopy (SEM) analyses and adherend topography measurements were used to explore the observed experimental trends. This chapter has been submitted for publication in *The Journal of Adhesion*\(^4\).

Chapter 9 presents the conclusions and the recommended future work.

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Chapter 2

Fracture Load Predictions and Measurements

1. Introduction

A variety of strain and stress-based failure criteria have been proposed to predict the strength of adhesive joints [1-5]. The maximum stresses at the end of the joint overlap are difficult to predict accurately, and consequently it has been proposed that failure can be based on the integral of stress over some characteristic length [5-6]. A related critical stress/strain approach based on finite element modeling assumes that an adhesive joint fails when a “damage zone” extending from the end of the joint overlap reaches a specific value. This critical size and the adhesive failure criterion governing the development of the damage zone are calibrated by comparison with the measured failure loads on joints that mimic those used in the application of interest [7-8]. These approaches have been used to predict the strengths of joints when cracking first appears at the end of the overlap, rather than the ultimate strength of joints.

Another finite element approach is cohesive zone modeling, which has been used, for example, to predict failure loads in single lap shear joints (elastic and plastically deforming) [9-11]. The approach attempts to mimic the global deformation and stress in the damage zone at the tip of a developing crack, and involves the selection of traction-separation parameters that achieve this in mode I and mode II. However, the choice of these parameters is based largely on trial and error, and will not be unique in general (i.e. experimental results can be matched with more than one set of parameters). Moreover, numerical instabilities can affect finite element solutions involving cohesive zone models [12].

For joints in which the adherends remain elastic, an analytical fracture-based method has been shown to accurately predict the final fracture loads of joints bonded with relatively brittle epoxy adhesives [13-17]. This approach was based on characterizing the strength of an adhesive system using a fracture envelope; i.e. the steady state critical strain energy release rate as a function of the loading phase angle. The energy release rate for a particular joint was calculated using a closed-form expression for the J-integral in a cracked adhesive sandwich. The calculated energy release rate and phase angle for the joint were then compared to the steady state critical
strain energy release rate, $G_c^*$, at the corresponding phase angle $\psi$. The experimentally measured fracture envelope was used and the fracture load was extracted. This approach was shown to accurately predict the ultimate loads of joints made with two relatively brittle epoxy adhesives of moderate strength bonded to aluminum adherends [16].

The objective of the present work was to assess the performance of this approach with an epoxy adhesive that was an order of magnitude tougher than those used in earlier work. The adhesive also displayed a much more prominent R-curve, compared to the previously tested brittle adhesives [14,16], and it was not obvious that the critical strain energy release rate could still form an appropriate failure criterion. Joint strength was predicted using the measured fracture envelope, $G_c^*(\psi)$, and compared with measured fracture loads for two commonly used joints, the cracked-lap shear (CLS) and the single-lap shear (SLS), made with either steel or aluminum adherends.

2. Specimen Preparation

The fracture envelope was measured using double cantilever beam (DCB) specimens fabricated from 12.7 mm×19.05 mm (1/2”×3/4”) AA6061-T651 flat bars (yield stress $\sigma_y=275$ MPa), and having a bondline thickness of 0.4 mm, controlled by placing spacing wires in the bondline. Prior to bonding, the aluminum adherends were abraded using a silicon carbide nylon mesh abrasive pad which gave an average roughness of $R_a = 0.77±0.02 \mu m$. The aluminum bars were then pretreated using the P2 etching process [18]. By inserting a thermocouple in the bondline, the adhesive temperature was monitored during cure to ensure that the adhesive remained at 180°C for 30 min, the manufacturer recommended curing schedule. Cracked lap shear (CLS) and single lap shear (SLS) joints were made from AA7075-T651, $\sigma_f=500$ MPa, in order to prevent yielding of the adherends during the fracture tests. The surface preparation and the curing procedure were as with the DCB joints. To remove the excessive adhesive after curing and achieve a specimen of uniform width, the sides of the joints were carefully milled to avoid edge damage and overheating. To improve the visibility of the crack, the bondline was then sanded with P100 sandpaper, and a thin coating of diluted correction fluid was applied. The geometries of the DCB, CLS and SLS joints are shown schematically in Fig. 1. As explained below, the CLS specimens were used with several end configurations: one that had a foil precrack and two that had adhesive fillets and no precrack.
To evaluate the failure load prediction technique with another system, DCB, CLS and SLS specimens were also manufactured from AISI 4140 flat steel bars. The bars were first abraded with an aluminum oxide abrasive pad, giving an average roughness of $R_a = 0.84 \pm 0.03 \mu$m, followed by degreasing using acetone and ethanol. DCB specimens were manufactured
from 12.7 mm×19.05 mm (1/2”×3/4”) flat bars. As was the case with the aluminum joints, the bondline thickness was 0.4 mm.

3. Fracture Envelope

3.1. Experimental Approach

Crack growth must be measured over a range of mode ratios (phase angles) in order to obtain the fracture envelope (\(G_c^s\) vs. phase angle) for a given adhesive system, defined as the combination of adhesive, pretreatment, adherend, bondline thickness and cure schedule. The load jig shown in ref. [14] provides a convenient way of measuring the critical strain energy release rate, \(G_c\), as a function of the phase angle using a single DCB specimen. By adjusting the pin locations in the link arms (Fig. 2), the load jig allows many different combinations of moments to be applied to the arms of the DCB specimen using a single actuator and specimen geometry. The load jig is statically determinate, and the specimen loads, \(F_1\) and \(F_2\) are given from equilibrium considerations of the link-arm system as [14]

\[
F_1 = F \left(1 - \frac{s_1}{s_3}\right)
\]

\[
F_2 = F_1 \frac{s_1}{s_2} \frac{1}{\left(1 + \frac{s_3}{s_4}\right)}
\]

where \(s_1, s_2, s_3\) and \(s_4\) are defined in Fig. 2 as the distances between the pin centers.

A load frame with a capacity of 10 kN was used to load the specimen under displacement control. The load was increased at a constant cross-head speed of 1 mm/min. The crack length was measured from the center of the loading pins on the DCB specimens using a microscope mounted on a micrometer stage having an accuracy of 0.01 mm. The accuracy in the crack length measurement decreased as the phase angle increased, due to the less opening of the crack. However, it is believed that the accuracy in the measured crack length was always better than 0.1 mm, which incurred a very insignificant error in the measured \(G_c\) (about 0.1%). The crack front along the width of the DCB joint was fairly uniform, therefore, a crack length measurement based on the crack observed on the edge of the specimen was accurate enough for \(G_c\) measurements, and a very insignificant difference would be observed if the \(G\) calculation was
based on the crack length in the middle of the specimen’s width. The maximum difference between the crack at the edge of the specimen and the crack front in the middle of the specimen was measured to be about 2 mm, which, if assuming the crack length according to the middle of the specimen’s width, could result in only a 2% increase in the $G_c$. Crack growth was stable in this system so that many crack extension events could be recorded with a single specimen.

Fig. 2 Schematic illustration of the load jig [14] (not to scale). The distance between the holes on the load jig is 25.4 mm.

References [14] and [16] describe the procedure to measure the fracture envelope for two relatively brittle single-part epoxy adhesives. For those adhesives, it was appropriate to start and stop the cross-head displacement repeatedly until new cracking was observed in the damage zone ahead of the macro-crack at the critical load for the measured crack length. However, the single-part, heat-cured toughened epoxy adhesive used in the present study was found to be much tougher and more visco-elastic than those two adhesives, and therefore the crack growth was less abrupt, being more of a gradual tearing of the bondline. Consequently, relying solely on visual observation of the damage zone while manually starting and stopping the cross-head iteratively, led to overloading the specimen and recording loads greater than the true critical load corresponding to $G_c$. In principle, this problem could be resolved by choosing a very small
cross-head speed; however, a small crosshead speed would greatly increase the test duration, and due to the visco-elastic nature of the adhesive, creep crack growth might occur at loads below that corresponding to $G_c$. For the present very tough adhesive system, a better approach was to start and stop the cross-head displacement repeatedly in the vicinity of the expected fracture load (each time at a constant crosshead speed of 1.0 mm/min) until a drop in the applied load was observed. This maximum load prior to the drop was taken as the critical fracture load for the measured crack length if visual inspection through the microscope confirmed that the macro-crack had propagated. After measuring the new macro-crack length, the DCB was unloaded and the same procedure was followed again beginning at the new crack length. In this way many $G_c$ measurements could be obtained for a single DCB. Although this measurement procedure differed from that utilized in [13-17], where the crack tip was considered as the furthest advanced micro-crack, there was a negligible effect on the steady state $G_c$ beyond the rising part of the R-curve (Fig. 3), since the length of the damage zone was usually small compared to the overall crack length. For instance, for a specimen tested at a loading phase angle of $16^\circ$, such a difference in crack tip definition resulted in only a 2%-4% reduction in the calculated $G_c^s$.

![Graph](image_url)

Fig. 3  Typical R-curve behavior of a DCB joint. Aluminum specimen tested at a phase angle of $\psi=27^\circ$.

### 3.2. DCB Data Analysis, $G$ Calculation and Mode Partitioning
The calculation of the phase angle and critical energy release rate, $G_c$, of DCB specimens has been done using beam theory [14] or a beam-on-elastic foundation approach [19]. The beam theory approach neglects the presence of the adhesive, while the beam-on-elastic-foundation model accounts for the additional compliance of the adhesive layer.

3.2.1. Beam Theory [14]

Assuming that the adhesive layer of the test specimen is thin, and neglecting shear deformation, the energy release rate per unit area of crack extension, $G$, and the nominal phase angle (mode ratio) of loading, $\psi = \arctan\left(\sqrt{\frac{G_{II}}{G_I}}\right)$, can be expressed as

$$G = \frac{(F_a)^2}{2D} \left[1 + \left(\frac{F_2}{F_1}\right)^2 - \frac{1}{8} \left(1 + \left(\frac{F_2}{F_1}\right)^2\right)^2\right]$$

$$\psi = \arctan\left[\frac{\sqrt{3}}{2} \frac{F_1 + 1}{F_2 - 1}\right]$$

where $a$ is the crack length measured from the loading pins and $D$ is the flexural rigidity per unit width of the adherends, given under plane stress by

$$D = \frac{Eh^3}{12}$$

with $E$ and $h$ being the Young’s modulus and thickness of the adherends, respectively. Note that the loads are positive in the direction of actuator force, $F$, as depicted in Fig. 2, and that $F_1$ and $F_2$ in Eq. (3) are also defined per unit width and are, respectively, the forces at the upper and lower loading pins of the specimen. From Eq. (2) it is seen that the ratio $F_1/F_2$ is only a function of the chosen load jig geometry ($s_1, s_2, s_3$, and $s_4$), and hence the nominal phase angle of loading (Eq. (4)) is independent of the crack length, $a$, of the specimen. $s_1, s_2, s_3, s_4$ are defined positive as shown in Fig. 2.

3.2.2. Beam-on-Elastic-Foundation Model
An analytical beam-on-elastic-foundation model for the DCB energy release rate has been presented [19]. If $F_1$ and $F_2$ (Fig. 2) are transformed to $f_1$ and $f_2$ through the equations

$$f_1 = \frac{F_1 - F_2}{2}$$

$$f_2 = -\frac{F_1 + F_2}{2}$$

then the energy release rate in an adhesive with a thickness $t$ is given by

$$G = \frac{12a^2}{E(h-t)^3} \left[ f_1^2 \Phi_I^2 + \frac{3}{4} f_2^2 \Phi_{II}^2 \right]$$

(7)

where $a$ is the crack length, and $E$ and $h$ are, respectively, the elastic modulus and thickness of the adherends. $\Phi_I$ and $\Phi_{II}$ are given by

$$\Phi_I = 1 + 0.667 \frac{h}{a} \left(1 - t/h\right)^{1.5} \left[1 + t/h(2E/E_a - 1)\right]^{0.25}$$

(8)

$$\Phi_{II} = 1 + 0.206 \frac{h}{a} \left[1 - \frac{t}{h}\right] \left[1 + \frac{2tE\alpha}{G_s h}\right]$$

where $\alpha = 2.946$ is a calibration constant that was determined using a finite element analysis [19]. $E_a$ and $G_a$ are the elastic and shear modulus of the adhesive, respectively. The phase angle is given by

$$\psi = \arctan \left[ \frac{\sqrt{3} F_1 \Phi_{II}}{2 F_2 \Phi_I} \right]$$

(9)

3.3. Experimental Results for DCB Specimens

During the first several crack growth sequences, the measured critical energy release rate, $G_c$, increased with crack length, becoming almost constant after the crack propagated a certain
distance (Fig. 3). Such resistance curves are observed commonly in the interlaminar fracture of composites and in adhesive joints [16,20-27], and result from the growth of a damage zone ahead of the crack tip, which increases the joint toughness as the volume of yielded and micro-cracked material expands. The rising portion of the R-curve ends when the damage zone, or the plastic zone, reaches a steady-state size [16,26]. The length of the rising portion of the curve increased with increasing mode ratio and with increasing bondline thickness, both of which also caused the steady-state size of the crack tip damage zone to grow. This is illustrated in Figs. 4 and 5. The steady state critical strain energy release rate, $G_{cs}$, at each phase angle was considered to be the average value over the “plateau” (steady state) region (Fig. 3). To measure the length of the rising part of the R-curve, the starting point was the tip of the aluminum foil precrack. The end of the rising part was determined using an algorithm that identified the start of the plateau based on the change in the slope of $G(a)$.

![Graph showing $G_c$ vs. crack length for phase angles of 16° and 55°. Two representative experiments have been shown for each phase angle. Crack extension was measured from the tip of the precrack.](image)

Fig. 4 $G_c$ vs. crack length for phase angles of 16° and 55°. Two representative experiments have been shown for each phase angle. Crack extension was measured from the tip of the precrack.

Under mode I loading, the crack propagated in the mid-plane of the bondline. Increasing the phase angle resulted in a crack path which was closer to the more highly strained arm of the DCB joint, and left more shear hackles. This change in the crack path with increasing phase
angle was in accordance with expectations [20,28]. The crack path in the aluminum DCB joints was fully cohesive. The steel DCBs also failed cohesively, although there were scattered small patches of interfacial failure as well. The percentage of the fracture surface that was interfacial failure for steel joints increased with phase angle, and was about 10%-20% of the total fracture surface area for phase angles from $0^\circ$ to $55^\circ$.

![Graph showing change in the length of the rising part of the R-curve with phase angle. Different symbols are used for separate tests performed at the same phase angle.](image)

**Fig. 5** Change in the length of the rising part of the R-curve, with phase angle. Different symbols are used for separate tests performed at the same phase angle.

To verify that the measured $G_c^s$ was independent of the adherend width, two specimens were made, one 19 mm (3/4") wide and the other 25.4 mm (1") wide. When tested at a phase angle, $\psi = 16^\circ$, the $G_c^s$ values for the two specimens differed by less than 1%; therefore, a width of 19 mm was used for all specimens. The independence of the fracture behavior on specimen width over this range was also observed in [14,16]. The number of data points on the plateau region, used to measure the $G_c^s$, of the 19 mm and 25.4 mm wide specimens were 26 and 33, respectively.

It was of interest to investigate the effect of cross-head speed on the fracture behavior of the adhesive over a range of speeds that span typical quasi-static testing. A specimen was tested at $\psi = 16^\circ$ at a cross-head speed of 0.5 mm/min to obtain 20 $G_c^s$ data points on the plateau. The test was then continued at a cross-head speed of 5 mm/min, and 20 more data points on the plateau were measured. The average $G_c^s$ values for the two cross-head speeds differed by less
than 3%, showing that the steady-state critical strain energy release rate was relatively insensitive to the strain rate over this range. It is expected that differences in $G_c^s$ would be observed if a much wider range of cross-head speeds was chosen.

The fracture envelope for the aluminum adhesive system was defined by measuring $G_c^s$ at seven different phase angles as shown in Fig. 6 (beam theory calculation). It is observed that the dependence of $G_c^s$ on $\psi$ was very small from $\psi = 0^\circ$ to $\psi = 30^\circ$, becoming more significant at higher phase angles, similar to what was previously reported for more brittle adhesive systems [15,16]. However, the $G_c^s$ values were much higher than in these earlier studies. For example, the average mode I ($\psi = 0^\circ$) $G_c^s$ for the current adhesive system was 3860 J/m$^2$ (calculated using the beam-on-elastic-foundation model), compared to 212 J/m$^2$ for Cybond 4523GB [16] and 794 J/m$^2$ for ESP 310 adhesive [15].

![Fig. 6 Measured fracture envelope for aluminum adhesive system calculated using beam theory. Given values are average $G_c^s$ (±1 SD).](image)

A third-order polynomial was fitted to the measured fracture envelope, $G_c^s$ versus $\psi$. Equations (10) and (11) give the relation using the beam theory and the beam-on-elastic-foundation models, respectively:

\[
G_c^s = 3237.9 + 13.342 \psi - 0.6832 \psi^2 + 0.0345 \psi^3, \quad R^2 = 0.98 \tag{10}
\]

\[
G_c^s = 3850.8 + 10.996 \psi - 0.9495 \psi^2 + 0.0405 \psi^3, \quad R^2 = 0.98 \tag{11}
\]
Beam theory, which does not include the compliance of adhesive in the total joint compliance, resulted in a lower $G_c^s$ (16% smaller at $\psi = 0^\circ$).

A commonly used equation for fitting the experimental data at different phase angles is

$$\frac{G_I}{G_{IC}} + \frac{G_{II}}{G_{IIIC}} = 1$$

(12)

where $G_{IC}$ and $G_{IIIC}$ are steady-state critical energy release rates under pure mode I and pure mode II, respectively, and $G_I$ and $G_{II}$ are the critical components of the mode I and II energy release rates under mixed-mode crack growth conditions [29]. The measured $G_I$ and $G_{II}$ critical values, with the number of tested specimens and data points at the seven phase angles of Fig. 6 are listed in Table 1. The $G_{IIIC}$ at $\psi = 90^\circ$ was extrapolated from a curve fit of Fig. 6. The sixth column of Table 1 shows that the mode I and II energy release rates could be reasonably fitted using Eq. (12). The standard deviation of the steady-state critical strain energy release rate was less than 10% of the mean at each of the seven phase angles (5% on average), which was comparable to previous experience with much more brittle epoxy adhesives [14].

Table 1  Measured critical values of $G_I$ and extrapolated $G_{II}$ at different phase angles for the aluminum system fracture envelope. $N$ and $M$ represent the total number of data points and the number of specimens, respectively.

<table>
<thead>
<tr>
<th>Phase Angle (degree)</th>
<th>$G_I$ (J/m$^2$)</th>
<th>$G_{II}$ (J/m$^2$)</th>
<th>$G_I / G_{IC} + G_{II} / G_{IIIC}$</th>
<th>$N$</th>
<th>$M$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average</td>
<td>Standard Deviation</td>
<td>Average</td>
<td>Standard Deviation</td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>3255</td>
<td>151</td>
<td>0</td>
<td>0</td>
<td>1.0</td>
</tr>
<tr>
<td>16</td>
<td>3166</td>
<td>168</td>
<td>264</td>
<td>14</td>
<td>0.98</td>
</tr>
<tr>
<td>27</td>
<td>2842</td>
<td>180</td>
<td>767</td>
<td>49</td>
<td>0.90</td>
</tr>
<tr>
<td>48</td>
<td>2958</td>
<td>152</td>
<td>3668</td>
<td>189</td>
<td>1.05</td>
</tr>
<tr>
<td>54</td>
<td>2484</td>
<td>58</td>
<td>4699</td>
<td>110</td>
<td>0.94</td>
</tr>
<tr>
<td>55</td>
<td>2314</td>
<td>85</td>
<td>4821</td>
<td>176</td>
<td>0.89</td>
</tr>
<tr>
<td>65</td>
<td>1915</td>
<td>205</td>
<td>8978</td>
<td>962</td>
<td>0.92</td>
</tr>
</tbody>
</table>

A similar procedure was used to measure the fracture envelope of the steel adhesive system. The experiments were conducted at phase angles of $\psi = 0^\circ$, 16$^\circ$, 27$^\circ$ and 48$^\circ$, yielding the third-order polynomial fits given in Eqs. (13) and (14), for the beam theory and the beam-on-
Several studies in the literature have noted an effect of substrate stiffness on the critical strain energy release rate for quasi-static fracture. Yan et al. [30] observed that the $G_c^s$ for an adhesive system with steel adherends was less than that of the same adhesive with more flexible aluminum adherends. This was attributed to elevated stress levels in the crack tip region for the stiffer steel joint. On the other hand, Bell and Kinloch [31] compared the $G_c^s$ of aluminum, steel and CFRP joints and found that $G_c^s$ increased with adherend stiffness. This was attributed to the shape and the size of the plastic zone ahead of the tip and within the adhesive layer which was believed to be affected by the transverse elastic modulus of the adherends. Later however, further experiments with a similar adhesive showed that, the differences between the aluminum and steel joints were less pronounced [32], and that the differences between the CFRP and the metallic adherends were due to water absorption by the CFRP substrates. Comparing the fracture envelope of the aluminum adhesive system, Eqs. (10) and (11), with the corresponding relations for the steel adhesive system, Eqs. (13) and (14), it is observed that a slightly lower $G_c^s$ was measured for the steel system. This was attributable to the different crack path in the steel joints, which was mostly cohesive, but had scattered small patches of interfacial failure as explained above. Overall, there was no difference that could be attributed to the modulus of the adherend material.

To predict the failure load in aluminum and steel CLS and SLS fracture joints, Eqs. (10) and (13) were considered to be the fracture envelopes of aluminum and steel systems, respectively. The reason for using the beam theory model, rather than the beam-on-elastic-foundation model, is explained in Section 4.1.

### 4. Quasi-Static Fracture Tests on CLS and SLS Joints

Nine CLS and eleven SLS joints of varying overlap and arm lengths were made from 25.4×19.05 mm (1”×3/4”) flat bars of aluminum AA 7075-T651 in order to achieve a range of joint strengths to test the model predictions (Tables 2 and 3). This alloy and thickness were sufficient to prevent adherend yielding before adhesive fracture. The SLS joints had one arm...
longer than the other so that a higher bending moment was applied at the overlap end of the longer arm. This ensured that a crack initiated and grew from only one end of the joint.

In order to investigate the effect of initiation on the ultimate strength, three different conditions at the overlap ends were considered: a small adhesive fillet, a large adhesive fillet and a precracked condition. The small adhesive fillet was formed by removing the excess adhesive from the end of the overlap using a small spatula before curing. The large adhesive fillet was formed by not removing this excess adhesive prior to curing. The precracked condition was formed by embedding a piece of 20 µm thick aluminum foil that had been folded over to make an unbonded double layer in the adhesive at the end of the overlap. The resulting conditions at the overlap end are schematically shown in Fig. 7.

![Fig. 7 Different overlap end conditions. a) Small Fillet, b) Large Fillet and c) Precracked.](image)

In addition to the aluminum joints, six CLS and six SLS joints were bonded using 25.4 mm×19.05 mm (1”×3/4”) AISI 4140 steel flat bars (Tables 4 and 5). AISI 4140 in this thickness did not yield during the fracture experiments. The steel joints were made with the same three conditions at the end of the overlap as were the aluminum joints.

All specimens were loaded to ultimate fracture on a servo-hydraulic load frame at a constant displacement rate. The crack initiation and growth were monitored during the loading of the specimen using a CCD camera with a high-magnification lens, giving a field of view of 3 mm. The camera was mounted on a motorized linear stage to follow the crack tip as it was advancing with increasing load. The measured force and crack length as a function of time were used to calculate the corresponding $G$. The video recording was also analyzed to identify the approximate crack initiation load.

As with the DCB joints, the fractures were entirely cohesive for the aluminum CLS and SLS joints, while the steel fracture surfaces were predominately cohesive with scattered, small
patches of interfacial failure that covered about 20% of the total fracture surface.

4.1. Data Analysis, *G* Calculation and Mode Partitioning of CLS and SLS Joints

To calculate the energy release rate, an “adhesive sandwich” was isolated in the CLS and SLS joints as shown in Fig. 8, and the J-integral for this sandwich was calculated as [13]:

\[
G = \left[ \frac{F_1^2}{2(Eh)_1} + \frac{M_1^2}{2D_1} \right] + \left[ \frac{F_2^2}{2(Eh)_2} + \frac{M_2^2}{2D_2} \right] - \left[ \frac{F_0^2}{2(Eh)_0} + \frac{M_0^2}{2D_0} \right]
\]

where \(F\) and \(M\) are, respectively, the tensile force and the bending moment per unit width in the adherends at the crack tip, while \(E\) and \(h\) are the elastic modulus and the beam thickness. Subscripts 1, 2 and 0 denote the respective cross-sections, as shown in Fig. 8. \(D\) is the flexural rigidity per unit width of the beam (Eq. (5)). Note that it is not necessary to assume the existence of a crack in the adhesive layer – if there is no crack, the strain energy release rate is calculated in the same way and represents the value for a crack just beginning to grow from the end of the overlap.

\[
\frac{\partial^4 v_i}{\partial x^4} - \frac{P}{D_i} \frac{\partial^2 v_i}{\partial x^2} = 0
\]

which has the general solution
\[ v_i = C_{i1} x + C_{i2} + C_{i3} \cosh(\lambda_i x) + C_{i4} \sinh(\lambda_i x) \quad (17) \]

where
\[ \lambda = \sqrt{\frac{P}{D_i}} \quad (18) \]

CLS and SLS joints are divided, respectively, into two and three beam sections denoted by the subscript, \( i \). The constants, \( C_{ii} - C_{id} \), were determined using the boundary conditions together with the beam theory relations for bending moments and shear forces. At the loading pins, the transverse deflections and bending moments were considered to be zero. At the overlap ends, continuity implies that the displacements and slopes match for the two beam sections. For CLS joints, this yields eight linear equations in eight unknowns for which a closed-form solution exists [15,16]. For SLS joints, a set of twelve equations in twelve unknowns was solved numerically [15,16]. The Appendix gives the constants for the CLS joints and the set of equations for the SLS joints.

The phase angles for the CLS and SLS joints can be calculated by using the following expressions [15]:

\[ G_i = \frac{[M_2(0^-)]}{4D_i} \quad (19) \]

\[ G_{ii} = G - G_i \quad (20) \]

\[ |\psi| = \arctan \left( \frac{G_{ii}}{G_i} \right) \quad (21) \]

where \( M_2(0^-) \) is the bending moment immediately to the left of the crack tip.

The \( G \) calculation and mode partitioning for the CLS and SLS joints presented above are based on beam theory, and to maintain consistency, the fracture envelope derived from beam theory (Eqs. (10) and (13) for the aluminum and steel adhesive systems, respectively, was used to predict the ultimate fracture load in the CLS and SLS joints. As mentioned previously, the simpler beam theory model neglects the compliance of the adhesive layer and underestimates the strain energy release rate. However, simplifying the analysis using beam theory for both the fracture envelope DCB calculations and those for the actual CLS and SLS joints produces offsetting errors that have a small net effect on the ultimate fracture load prediction. This approach was successfully followed in ref. [16]. Having calculated the phase angle, \( P_{pred} \) is the force that yields the critical energy release rate, \( G_c^* \), from the fracture envelope at the calculated phase angle, \( \psi \).
4.2. Experimental Results and Predictions

4.2.1. Aluminum Adhesive System

As expected from the adhesive R-curve behavior, cracks started to propagate at loads between 30% and 90% of the ultimate failure load. The subcritical crack propagated in a stable manner for several centimeters before the ultimate critical load was attained and final fast fracture occurred. The measured crack initiation and final failure loads for the aluminum CLS and SLS joints with different initial conditions are given in Tables 2 and 3. Figure 9 compares the approximate crack initiation strain energy release rate, $G_c^i$, with the corresponding steady state critical strain energy release rate, $G_c^s$, for the CLS and SLS joints with the three different overlap end conditions. The given $G_c^s$ for each specimen was the value at the ultimate load of the joint and was based on the phase angle calculation of the initial geometry of the specimen. As will be discussed below, this phase angle was only slightly different from the phase angle at the critical actual crack length corresponding to final rupture. It is clear that for all three overlap end conditions, the crack started propagating at significantly lower $G_c^i$ values than the $G_c^s$. As mentioned before, this behavior is attributed to the damage zone development ahead of the crack tip, which occurred in the quasi-static loading of CLS and SLS joints as well as in the DCBs used to measure the fracture envelope.

Since crack initiation occurred well below the joint ultimate load and was followed by a period of stable crack growth, the ultimate fracture load did not depend on the initial condition at the end of the joint overlap (fillet or precrack), which is consistent with the results for the adhesive system of ref. [17].

Phase angles that can be achieved by changing the geometry of CLS and SLS joints are in the relatively narrow range of 45º to 55º, as can be seen in Tables 2 and 3. Figure 5 shows that the crack propagation associated with the rising part of the DCB R-curve for phase angles in this range was between 40 and 60 mm for aluminum specimens. This length corresponded to a change in the appearance of the fracture surfaces of the SLS and CLS joints, which displayed two distinct regions corresponding to slow, stable crack extension and then fast fracture. An example of this is shown in Fig. 10 for two of the SLS joints. For the tested CLS and SLS specimens this change in the failure surface occurred at an average of 60±10 mm from point of crack initiation, which was approximately the length of crack growth seen during the rising part of the R-curve in DCB tests, implying that the SLS and CLS joints reached their ultimate strength and became unstable when the adhesive damage zone had fully developed at $G_c^s$.\[22\]
This crack growth pattern was confirmed using the CCD camera to monitor the crack length during CLS and SLS testing. As expected from the failure surface, after the crack initiated, it propagated stably with a gradually increasing crack speed from approximately 0.02 mm/s to about 0.35 mm/s under an increasing joint load until very close to the moment of final fracture when the crack speed increased sharply and the joint broke. The recorded crack length at the onset of fast fracture was consistent with the location of the change in the failure surface seen in Fig. 10.

Table 2  Geometry and initial conditions of aluminum CLS specimens, measured crack initiation forces, $P_i$, and final failure loads, $P_{\text{Exp}}$, per unit width, compared with predicted failure loads, $P_{\text{Pred}}$, using a 60 mm subcritical crack length. SF, LF and P stand for small fillet, large fillet and precracked, respectively.

<table>
<thead>
<tr>
<th>Specimen, Initial Condition</th>
<th>$L_1, L_2$ (mm)</th>
<th>$\psi$ (°)</th>
<th>$P_i$ (kN/m)</th>
<th>$P_{\text{Exp}}$ (kN/m)</th>
<th>$P_{\text{Pred}}$ (kN/m)</th>
<th>Error (%), ($P_{\text{Pred}} - P_{\text{Exp}}$) / $P_{\text{Exp}}$</th>
<th>Displ. Rate (mm/min.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C12C, SF</td>
<td>308, 190</td>
<td>51.8</td>
<td>2747</td>
<td>3787</td>
<td>4021</td>
<td>6</td>
<td>1.50</td>
</tr>
<tr>
<td>C4A, SF</td>
<td>312, 135</td>
<td>49.4</td>
<td>1299</td>
<td>3027</td>
<td>3139</td>
<td>4</td>
<td>0.25</td>
</tr>
<tr>
<td>C12B, LF</td>
<td>316, 152</td>
<td>50.1</td>
<td>2384</td>
<td>3779</td>
<td>3398</td>
<td>-10</td>
<td>0.35</td>
</tr>
<tr>
<td>C13C, LF</td>
<td>316, 130</td>
<td>49.3</td>
<td>-</td>
<td>3705</td>
<td>3063</td>
<td>-17</td>
<td>1.00</td>
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<tr>
<td>C4C, LF</td>
<td>234, 190</td>
<td>51.9</td>
<td>1454</td>
<td>4023</td>
<td>4085</td>
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<tr>
<td>C12A, P</td>
<td>145, 131</td>
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<td>2598</td>
<td>3687</td>
<td>3221</td>
<td>-13</td>
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</tr>
<tr>
<td>C13A, P</td>
<td>200, 150</td>
<td>50.3</td>
<td>2400</td>
<td>3669</td>
<td>3454</td>
<td>-6</td>
<td>1.00</td>
</tr>
<tr>
<td>C13B, P</td>
<td>330, 170</td>
<td>50.9</td>
<td>2274</td>
<td>3983</td>
<td>3681</td>
<td>-8</td>
<td>1.50</td>
</tr>
<tr>
<td>C4B, P</td>
<td>329, 147</td>
<td>50.0</td>
<td>1735</td>
<td>3855</td>
<td>3317</td>
<td>-14</td>
<td>0.25</td>
</tr>
</tbody>
</table>
Table 3  Geometry and initial conditions of aluminum SLS specimens, measured crack initiation forces, $P_i$, and final failure loads, $P_{Exp}$, per unit width, compared with predicted failure loads, $P_{Pred}$, using a 60 mm subcritical crack length. SF, LF and P stand for small fillet, large fillet and precracked, respectively.

<table>
<thead>
<tr>
<th>Specimen, Initial Condition</th>
<th>$L_1$, $L_2$, $L_3$ (mm)</th>
<th>$\psi$ (°)</th>
<th>$P_i$ (kN/m)</th>
<th>$P_{Exp}$ (kN/m)</th>
<th>$P_{Pred}$ (kN/m)</th>
<th>Error (%), $(P_{Pred} - P_{Exp}) / P_{Exp}$</th>
<th>Displ. Rate (mm/min.)</th>
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<tbody>
<tr>
<td>S1A, SF</td>
<td>197, 99, 120</td>
<td>47.4</td>
<td>2016</td>
<td>2277</td>
<td>2429</td>
<td>7</td>
<td>1.25</td>
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<tr>
<td>S1B, SF</td>
<td>159, 108, 82</td>
<td>47.2</td>
<td>-</td>
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<td>2370</td>
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<td>0.60</td>
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<tr>
<td>S3A, SF</td>
<td>192, 125, 145</td>
<td>48.3</td>
<td>-</td>
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<tr>
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<td>45.9</td>
<td>932</td>
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<td>1943</td>
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<tr>
<td>S5B, SF</td>
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<td>47.8</td>
<td>1279</td>
<td>-</td>
<td>-</td>
<td>-</td>
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</tr>
<tr>
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<td>48.5</td>
<td>-</td>
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<td>2666</td>
<td>2</td>
<td>0.60</td>
</tr>
<tr>
<td>S3B, LF</td>
<td>158, 153, 122</td>
<td>48.9</td>
<td>-</td>
<td>2891</td>
<td>2843</td>
<td>-2</td>
<td>0.20</td>
</tr>
<tr>
<td>S4B, LF</td>
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<td>46.6</td>
<td>1708</td>
<td>2177</td>
<td>2161</td>
<td>-1</td>
<td>0.25</td>
</tr>
<tr>
<td>S5C, LF</td>
<td>182, 115, 133</td>
<td>47.6</td>
<td>1505</td>
<td>2174</td>
<td>2445</td>
<td>12</td>
<td>0.25</td>
</tr>
<tr>
<td>S2A, P</td>
<td>172, 121, 143</td>
<td>48.6</td>
<td>1610</td>
<td>2605</td>
<td>2739</td>
<td>6</td>
<td>0.65</td>
</tr>
<tr>
<td>S5A, P</td>
<td>192, 125, 103</td>
<td>47.2</td>
<td>1924</td>
<td>2392</td>
<td>2311</td>
<td>-3</td>
<td>0.25</td>
</tr>
</tbody>
</table>
Fig. 9  Approximate crack initiation energy release rate, $G_c^i$, (shaded) observed with aluminum CLS and SLS specimens compared with the steady state critical strain energy release rate, $G_c^s$, from the fracture envelope at the same phase angle. CLS and SLS initial conditions: (a) small fillet, (b) large fillet and (c) precracked. Specimen numbers refer to Tables 2 and 3.
Based on the optically measured crack lengths at different times during the SLS and CLS testing, the energy release rate was calculated for some of the specimens during the subcritical crack growth. Figure 11 compares the $G_c$ versus crack length graphs obtained from a CLS joint with the corresponding graph obtained from DCB fracture envelope tests at $\psi=55^\circ$. It should be noted that the phase angle generally decreases with increasing crack length in CLS specimens [15]; however, for the present specimens, this change in phase angle due to the subcritical crack growth was less than 5°. This small change in the phase angle with crack length results from the increase in $L_1$ and the corresponding decrease in $L_2$ (Fig. 1), and may be calculated using the equations in the Appendix for CLS joints. It is observed that the $G_c$ vs. crack length graph obtained from a CLS joint was similar to the corresponding graph from a DCB joint at the same phase angle, and that the $G_c$ for the CLS joint reached the plateau of the DCB graph at a crack length of 50-60 mm.

In summary, the subcritical crack growth length was consistent with the location of the change in the fracture surface of CLS and SLS joints. It also agreed with the optically measured crack length at the onset of fast fracture, and was close to the length of the rising part of a DCB test at $\psi=55^\circ$. Therefore, it is concluded that, as the crack grows and the applied $G$ on the CLS or SLS joints is increased, $G_c$ also increases due to the development of the damage zone. This toughening behavior prevents the joint from fracturing and the joint can bear higher forces. As the $G_c$ reaches the plateau value, $G_c^s$, the toughening ends and catastrophic failure of the joint
occurs. The subcritical crack growth length must therefore be considered when predicting the ultimate failure load using the fracture envelope.

![Graph](image)

Fig. 11 Comparison of \( G_c \) vs. crack length for an aluminum CLS joint and two aluminum DCB fracture envelope tests at \( \psi = 55^\circ \). Point of onset of fast fracture is shown for CLS joint.

Based on the above observations, the failure loads for the aluminum CLS and SLS joints were predicted by calculating the phase angle for the joint at the critical crack length and then using the fracture envelope (Fig. 6) to find the corresponding \( G_c^k \). The ultimate joint strength was then the load that would generate such a strain energy release rate. For all of these SLS and CLS ultimate load predictions, it was assumed that the subcritical crack length was 60 mm. Tables 2 and 3 show that the measured ultimate loads and the predictions agreed well, with an average absolute relative error of 9% and 4%, respectively, for CLS and SLS joints.

In order to assess the sensitivity of the ultimate load prediction on the assumed amount of subcritical crack growth, predictions were also made assuming lengths of 0, 40, 50, 70 and 80 mm. The average relative error and the average absolute relative error between the predictions and experiments are given in Table 4 for CLS and SLS joints. It is seen that errors as large as 29% occurred if subcritical crack growth was not taken into account in the calculation of \( \psi \) and \( G_c \), but that the failure load prediction was relatively insensitive to the amount of subcritical
crack propagation assumed in the analysis; e.g. using a subcritical crack length of 40 mm or 80 mm instead of the actual subcritical crack length of 60 mm increased the average absolute error by less than 7% for both CLS and SLS joints. Therefore, choosing this subcritical crack length based on the length of the rising part of the R-curve in DCB fracture envelope tests for the corresponding phase angle yields good predictions of the final fracture load for CLS and SLS joints.

Table 4  Sensitivity of the failure load predictions to the assumed subcritical crack growth length for aluminum CLS and SLS joints. Errors are between experimental and predicted failure loads. The average absolute error is the average of the absolute value of each error in the failure load predictions.

<table>
<thead>
<tr>
<th>Subcritical Crack Growth Length (mm)</th>
<th>0</th>
<th>40</th>
<th>50</th>
<th>60</th>
<th>70</th>
<th>80</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLS Joints</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Error (%)</td>
<td>23</td>
<td>1.6</td>
<td>-4</td>
<td>-8</td>
<td>-12</td>
<td>-17</td>
</tr>
<tr>
<td>Average Absolute Error (%)</td>
<td>23</td>
<td>5</td>
<td>7</td>
<td>10</td>
<td>13</td>
<td>17</td>
</tr>
<tr>
<td>SLS Joints</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Error (%)</td>
<td>33</td>
<td>11</td>
<td>6</td>
<td>1</td>
<td>-3</td>
<td>-7</td>
</tr>
<tr>
<td>Average Absolute Error (%)</td>
<td>33</td>
<td>11</td>
<td>6</td>
<td>4</td>
<td>5</td>
<td>7</td>
</tr>
</tbody>
</table>

In earlier work with the relatively brittle Cybond 4532GB adhesive [16,17], the rising part of the R-curve was only about 5 mm for phase angles of 45º to 55º, the range for CLS and SLS joints. Therefore, it was not necessary to base the ultimate load calculation of $G_c$ on the joint geometry at the point of final failure. As a result, the ultimate load of SLS and CLS joints could be found by assuming that $G_c$ was reached at the overlap end with no subcritical crack growth.

For most of the CLS and SLS fracture joints, the cross-head displacement was increased continuously until the final failure, but for two CLS and two SLS joints, a start-stop loading was followed, similar to that used with the DCB tests. In this approach, the displacement was increased at a constant slow rate until the crack started to grow. At this point the displacement was held constant until the crack arrested as $G$ fell. This was followed by another increase in displacement until the crack again propagated, and the displacement was again held constant until the crack arrested. This quasi-static procedure was repeated until catastrophic failure occurred. For the chosen range of crack speed in our fracture experiments, no difference was
observed in the amount of subcritical crack growth or the R-curve behavior of the adhesive compared with the continuous loading, and the experimental fracture load was still close to the predictions, with an average absolute error of 6%. Therefore, it was concluded that these forms of continuous and start-stop loading were sufficiently similar to produce the same $G_c$ and length of the rising part of the R-curve.

4.2.2. Steel Adhesive System

Tables 5 and 6 give the geometry of the steel CLS and SLS specimens, which were tested at a constant displacement rate of 0.25 mm/min.

For the steel CLS and SLS joints, the transition between slow stable crack propagation and the fast final fracture occurred at 70±10 mm from the end of the overlap. As with the aluminum joints, this was consistent with the length of the rising part of the R-curve for steel DCB joints tested at the same phase angle. Tables 4 and 5 give the predicted ultimate loads for CLS and SLS tests, respectively, assuming 70 mm of subcritical crack growth. The agreement with the measured ultimate loads was good, having an average absolute error for CLS and SLS joints of 6% and 7%, respectively. As with the aluminum joints, the length of the subcritical crack growth in the CLS and SLS joints could be estimated to be the same as the length of the rising part of the R-curve in DCB fracture envelope tests.

Table 5  Geometry and initial conditions of steel CLS specimens, and comparison between the experimental, $P_{Exp}$, and predicted failure loads, $P_{Pred}$, per unit width. SF, LF and P stand for small fillet, large fillet and precracked, respectively.

<table>
<thead>
<tr>
<th>Specimen, Initial Condition</th>
<th>$L_1$, $L_2$ (mm)</th>
<th>$P_{Exp}$ (kK/m)</th>
<th>$P_{Pred}$ (kN/m)</th>
<th>Error (%), ($P_{Pred}$ - $P_{Exp}$) / $P_{Exp}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLS 1B, SF</td>
<td>274, 183</td>
<td>5328</td>
<td>5540</td>
<td>4</td>
</tr>
<tr>
<td>CLS 2B, SF</td>
<td>349, 142</td>
<td>4471</td>
<td>4704</td>
<td>5</td>
</tr>
<tr>
<td>CLS 3A, SF</td>
<td>319, 170</td>
<td>4651</td>
<td>5228</td>
<td>12</td>
</tr>
<tr>
<td>CLS 1A, LF</td>
<td>311, 141</td>
<td>4132</td>
<td>4720</td>
<td>14</td>
</tr>
<tr>
<td>CLS 2A, LF</td>
<td>227, 129</td>
<td>4540</td>
<td>4587</td>
<td>1</td>
</tr>
<tr>
<td>CLS 3B, P</td>
<td>277, 154</td>
<td>4677</td>
<td>4989</td>
<td>7</td>
</tr>
</tbody>
</table>
Table 6  Geometry and initial conditions of steel SLS specimens, and comparison between the experimental, $P_{Exp}$, and predicted failure loads, $P_{Pred}$, per unit width. SF, LF and P stand for small fillet, large fillet and precracked, respectively.

<table>
<thead>
<tr>
<th>Specimen, Initial Condition</th>
<th>$L_1$, $L_2$, $L_3$ (mm)</th>
<th>$P_{Exp}$ (kK/m)</th>
<th>$P_{Pred}$ (kN/m)</th>
<th>Error (%)</th>
<th>$(P_{Pred} - P_{Exp}) / P_{Exp}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>SLS15A, SF</td>
<td>158, 173, 122</td>
<td>4313</td>
<td>4156</td>
<td>-4</td>
<td></td>
</tr>
<tr>
<td>SLS15C, SF</td>
<td>217, 107, 93</td>
<td>2923</td>
<td>2923</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>SLS14A, LF</td>
<td>159, 128, 82</td>
<td>3436</td>
<td>3223</td>
<td>-6</td>
<td></td>
</tr>
<tr>
<td>SLS14C, LF</td>
<td>138, 173, 102</td>
<td>4273</td>
<td>4097</td>
<td>-4</td>
<td></td>
</tr>
<tr>
<td>SLS14B, P</td>
<td>223, 141, 194</td>
<td>3685</td>
<td>4028</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>SLS15B, P</td>
<td>217, 87, 93</td>
<td>2473</td>
<td>2731</td>
<td>11</td>
<td></td>
</tr>
</tbody>
</table>

A limitation of this approach to ultimate load prediction is that the overlap must be at least as long as the subcritical crack growth length - approximately 60 mm with aluminum CLS and SLS joints and 70 mm for the steel joints. The prediction of the ultimate load for shorter joints would require a more accurate model of subcritical crack growth; in particular, the critical strain energy release rate at initiation, $G_c^i$, and $G_c$ vs. crack length in the rising part of the R-curve. Comparing this crack growth behavior with the calculated strain energy release rate at the particular phase angle of the joint would allow the prediction of the maximum attainable load.

5. Conclusions

A previously established fracture-based, analytical approach for predicting the ultimate loads in common adhesive joints (CLS and SLS) can be applied to an epoxy adhesive that is an order of magnitude tougher than the earlier generation of (un-toughened) epoxy adhesives, provided the joint analysis takes into account the degree of subcritical crack growth. The approach is based on the observation that the strength of an adhesive bondline can be characterized by the fracture envelope for that particular adhesive system (i.e. the critical strain energy release rate, $G_c$, as a function of the mode ratio of loading, $\psi$). The subcritical crack growth length for both CLS and SLS specimens was approximately equal to the length of the rising part of the R-curve from the DCB specimens used to measure the fracture envelope. Using this approach, the average absolute error in the ultimate strength predictions was less than 10% for both aluminum and steel CLS and SLS joints.

The joint strength increased as the elastic modulus of the adherend increased as seen
when comparing the steel and aluminum joints. This was simply due to the relation between load, stiffness and the strain energy release rate. However, no difference was observed in the critical strain energy release rates that could be attributed to the adherend modulus.

Although the approach has been demonstrated for SLS and CLS joints of aluminum and steel, it is also applicable to other elastically deforming two-dimensional joint such as double lap joints. A “sandwich element” is isolated from the overlap end of the joint, and the force and moment reactions acting on the sandwich are used to calculate the strain energy release rate and the phase angle of loading.

During the fracture envelope measurement using aluminum and steel DCB specimens, the toughening behavior of the adhesive produced a relatively long rising part of the R-curve ($G_c$ vs. crack length) prior to reaching a steady-state critical strain energy release rate, $G_c^s$. This extensive, stable subcritical crack growth also occurred during the loading of the CLS and SLS joints, thereby changing the joint geometry considerably and altering the strain energy release rate, $G$, and to a smaller extent, the crack tip mode ratio, $\psi$. It was found that accurate predictions of the ultimate load could be made if $\psi$ and $G$ are calculated using the final geometry of the CLS and SLS joints after subcritical crack growth equal to that observed in the DCB fracture tests. It was shown that the prediction of the ultimate load is relatively insensitive to the assumed amount of subcritical crack growth. As well, because the subcritical crack growth was so large, the ultimate joint strength was independent of the geometry of the end of the joint overlap (e.g. fillet shape). The detailed geometry of the end of the adhesive layer is expected to affect only the crack initiation load.

Due to the R-curve behavior of the adhesive, the method can only be used to predict the ultimate load of joints having an overlap at least as large as the average subcritical crack growth length; otherwise the adhesive crack tip damage zone will not correspond to $G_c^s$. For joints with shorter overlaps, the rising part of the R-curve behavior would need to be modeled to permit a failure criterion based on $G_c$ as a function of the subcritical crack length, rather than the steady-state value $G_c^s$. Another limitation of the present approach is that it is applicable only to elastically deforming adherends since the equations used to calculate the strain energy release rate and the phase angle are based on this assumption, and the fracture envelope ($G_c^s$ as a function of the phase angle) was measured using elastic DCB specimens.

The high toughness and visco-elastic nature of the adhesive made it preferable to identify the critical fracture load during the DCB fracture envelope testing using load drop rather than the earlier optical method of detecting crack advance. These DCB fracture tests were relatively
insensitive to the cross-head speed over the range 0.5 – 5 mm/min.
Appendix – Beam Deflection Analysis for Fracture Specimens

The equations that are used to calculate the energy release rate, $G$, and phase angle, $\psi$, from the calculation of beam deflections are presented in this appendix.

2.A. Beam Deflection Analysis for Fracture Specimens – CLS Joints

Following the approach in ref. [15], a CLS joint is divided into two beams, and for each, the beam deflection is assumed according to Eq. (17). Applying the boundary conditions, the continuity of displacements and slopes for the overlap end, and considering that the difference in bending moments at the right and left side of the overlap end must be the moment due to the tensile force, together with the beam theory relations for bending moments and shear forces, eight equations were derived. Solving these set of equations, yielded the following closed form solution for the constants in the beam deflection solution (Eq. (17)):

\[
C_3 = -\frac{P \Delta \lambda_2}{D_1 \lambda_1^2} \left( \frac{\tanh(-\lambda_1 L_1)}{\lambda_2 \tanh(-\lambda_1 L_1) - \lambda_1 \tanh(-\lambda_2 L_2)} \right) \tag{A.1}
\]

\[
C_4 = -\frac{P \Delta \lambda_2}{D_1 \lambda_1^2} \left( \frac{1}{\lambda_2 \tanh(-\lambda_1 L_1) - \lambda_1 \tanh(-\lambda_2 L_2)} \right) \tag{A.2}
\]

\[
C_7 = -\frac{P \Delta \lambda_2}{D_2 \lambda_2^2} \left( \frac{\tanh(\lambda_2 L_2)}{\lambda_2 \tanh(-\lambda_1 L_1) - \lambda_1 \tanh(-\lambda_2 L_2)} \right) \tag{A.3}
\]

\[
C_8 = -\frac{P \Delta \lambda_2}{D_2 \lambda_2^2} \left( \frac{1}{\lambda_2 \tanh(-\lambda_1 L_1) - \lambda_1 \tanh(-\lambda_2 L_2)} \right) \tag{A.4}
\]

\[
C_2 = \frac{L_1 L_2}{L_1 + L_2} \left( \frac{C_7 - C_3}{L_2} + C_8 \lambda_2 - C_4 \lambda_1 \right) \tag{A.5}
\]

\[
C_1 = \frac{C_2}{L_1} \tag{A.6}
\]

\[
C_6 = C_2 + C_3 - C_7 \tag{A.7}
\]

\[
C_5 = -\frac{C_6}{L_2} \tag{A.8}
\]

where $\Delta=\frac{h}{2}$ ($h$ being the thickness of adherends).

Having the beam deflection, the bending moments at any desired cross-section of $x=x_c$, $x$ was measured from the overlap end in Fig. 1, and could be calculated by:
\[ M = -D \frac{\partial^2 v(x_c)}{\partial x^2} \quad \text{(A.9)} \]

The crack tip is at \( x=0 \). \( G \) was calculated from Eq. (15), and for phase angle, Eqs. (19-21) were used. This will lead to the following equations for equal-adherend CLS joints [15]:

\[ G = Eh^5 \lambda_1^4 \left( \frac{1}{576} + \frac{\tanh^2(\lambda_1 L_1) - \tanh^2(\lambda_2 L_2)}{768\sqrt{1/8 \tanh(\lambda_1 L_1) + \tanh(\lambda_2 L_2)}} \right) \quad \text{(A.10)} \]

\[ \frac{G_H}{G_I} = \frac{4}{3} + \frac{4}{3} \sqrt{2} \left( \frac{\tanh(\lambda_2 L_2)}{\tanh(\lambda_1 L_1)} \right) + \frac{2}{3} \left( \frac{\tanh(\lambda_2 L_2)}{\tanh(\lambda_1 L_1)} \right)^2 \quad \text{(A.11)} \]

\[ |\psi| = \arctan \left( \frac{G_H}{\sqrt{G_I}} \right) \quad \text{(A.12)} \]

**2.B. Beam Deflection Analysis for Fracture Specimens – SLS Joints [15]**

The approach for calculating the beam deflections and, therefore, the bending moments, for SLS joints was similar to the approach explained above for CLS geometry. However, instead of two beam sections for the case of CLS, the SLS joint is divided into three beams. This will lead to twelve equations and twelve unknowns. This set of equations is presented below, and was solved numerically to find the beam deflection constants. For simplicity, in the following equations \( L_1 \) is as illustrated in Fig 1, however, \( L_2 = (L_1 + L_2)_{\text{fig.1}} \) and \( L_3 = (L_1 + L_2 + L_3)_{\text{fig.1}} \).

\[ C_2 = C_3 = 0 \quad \text{(B.1)} \]

\[ C_1 L_1 + C_4 \sinh(\lambda_1 L_1) = C_5 L_1 + C_6 + C_7 \cosh(\lambda_2 L_2) + C_8 \sinh(\lambda_2 L_2) \quad \text{(B.2)} \]

\[ C_1 + C_4 \lambda_1 \cosh(\lambda_1 L_1) = C_5 + C_7 \lambda_2 \sinh(\lambda_2 L_2) + C_8 \lambda_2 \sinh(\lambda_2 L_2) \quad \text{(B.3)} \]

\[-D_1 C_4 \lambda_1^2 \sinh(\lambda_1 L_1) - P\Delta_1 = -D_2 \lambda_2^2 \left[ C_7 \cosh(\lambda_2 L_1) + C_8 \sinh(\lambda_2 L_1) \right] \quad \text{(B.4)} \]

\[-D_1 C_4 \lambda_1^3 \cosh(\lambda_1 L_1) = -D_2 \lambda_2^3 \left[ C_7 \sinh(\lambda_2 L_1) + C_8 \sinh(\lambda_2 L_1) \right] \quad \text{(B.5)} \]

\[ C_5 L_2 + C_6 + C_7 \cosh(\lambda_2 L_2) + C_8 \sinh(\lambda_2 L_2) = C_9 L_2 + C_{10} + \]

\[ C_{11} \cosh(\lambda_3 L_2) + C_{12} \sinh(\lambda_3 L_2) \quad \text{(B.6)} \]

\[ C_5 + C_7 \lambda_2 \sinh(\lambda_2 L_2) + C_8 \lambda_2 \cosh(\lambda_2 L_2) = C_9 + C_{11} \lambda_3 \sinh(\lambda_3 L_2) + C_{12} \lambda_3 \cosh(\lambda_3 L_2) \quad \text{(B.7)} \]

\[-D_2 \lambda_2^2 \left[ C_7 \cosh(\lambda_2 L_2) + C_8 \sinh(\lambda_2 L_2) \right] - P\Delta_2 = -D_3 \lambda_3^2 \left[ C_{11} \cosh(\lambda_3 L_2) + C_{12} \sinh(\lambda_3 L_2) \right] \quad \text{(B.8)} \]

\[-D_2 \lambda_2^3 \left[ C_7 \sinh(\lambda_2 L_2) + C_8 \cosh(\lambda_2 L_2) \right] = -D_3 \lambda_3^3 \left[ C_{11} \sinh(\lambda_3 L_2) + C_{12} \cosh(\lambda_3 L_2) \right] \quad \text{(B.9)} \]

\[ C_9 L_3 + C_{10} + C_{11} \cosh(\lambda_3 L_3) + C_{12} \sinh(\lambda_3 L_3) = 0 \quad \text{(B.10)} \]
\[ C_{11} \cosh(\lambda_1 L_3) + C_{12} \sinh(\lambda_1 L_3) = 0 \]  
(B.11)

where \( \Delta_1 = \Delta_2 = h/2 \). The set of coordinates originated at the left loading pin in Fig.1.

Knowing the bending moments at the crack tip, \( M_1(L^-_1) \) and \( M_2(L^+_1) \), Eq. (15) reduces to:

\[
G = \left[ \frac{M_1^2(L^-_1)}{2D_1} + \frac{P^2}{2(Eh)_1} \right] - \left[ \frac{M_2^2(L^+_1)}{2D_2} + \frac{P^2}{2(Eh)_2} \right] 
\]  
(B.12)

For the SLS joints with equal adherends, which was the case in this study, the phase angle was calculated as follows:

\[
G_I = \frac{M_2(L^-_1)^2}{4D_1} 
\]  
(B.13)

\[
G_{II} = G - G_I 
\]  
(B.14)

\[
|\psi| = \arctan \left( \frac{G_{II}}{G_I} \right) 
\]  
(B.15)
6. References


30. C. Yan, Y.W. Mai, Q. Yuan, L. Ye, J. Sun, Effects of substrate materials on fracture


Chapter 3

The Effect of Mode Ratio and Bond Interface on the Fatigue Behavior

1. Introduction

The fatigue behavior of adhesives has been studied extensively under mode I loading [1-6], and the testing procedure for double cantilever beam (DCB) and tapered double cantilever beam (TDCB) joints is well established. The differences between the fatigue behavior under mixed-mode and mode I loading has also been investigated [7-11]; however, usually only one other mode ratio was compared with the mode I loading condition making the fatigue dependence on mode ratio unclear. Mall and Johnson [7] observed no effect of increasing phase angle when comparing the fatigue crack growth rates of composite DCB and cracked lap shear (CLS) specimens bonded using a ductile adhesive. They also found no effect of mode ratio on the static debonding behavior of the same adhesive system, and hence proposed that mode I DCB testing should be sufficient to characterize the fatigue behavior of relatively tough adhesive joints [7]. A brittle adhesive, on the other hand, showed a higher cyclic debond growth rate under mixed-mode (CLS) loading compared to mode I (DCB) [9]. Pirondi and Nicoletto [11] investigated the effect of mode ratio over a range of phase angles in the linear crack growth rate region and also observed a somewhat higher fatigue crack growth rate as the phase angle increased. This contrasts with the observations of Dessureault and Spelt [10] who found that the fatigue performance improved under mixed-mode loading compared to mode I.

The ultimate fracture strength of toughened epoxy adhesive joints can be predicted using a failure criterion based on the critical strain energy release rate as a function of the loading phase angle [12-15]. One of the objectives of the present study was to investigate whether a similar concept could be applied to the fatigue crack growth rate or threshold.

The engineering design of adhesive joints subject to cyclic loads can be based on the specification of loads that either limit crack growth rates to an acceptably small value or that prevent crack growth altogether by staying below the crack growth threshold. Consequently, in
the general case, it is desirable to measure both the threshold strain energy release rate corresponding to near-zero crack growth and the crack speed as a function of the strain energy release rate. As mentioned above, it was of interest in the present study to determine the dependence of both the threshold energy release rate and the crack speed on the mode ratio of loading. This was done using three different joint geometries to achieve four loading phase angles. A single highly-toughened epoxy adhesive was used to make these joints with either steel or aluminum adherends pretreated in several ways to achieve various interfacial bond strengths.

2. Experimental Approach

2.1. Specimen Preparation

Mode I loading was achieved using a DCB specimen while two geometries of asymmetric DCB (ADCB) joints, one with an adherend thickness ratio of two and the other with a ratio of four, gave phase angles, $\psi$, of $18^\circ$ and $24^\circ$, respectively [16]. The phase angle is a measure of the mode ratio of loading defined as $|\psi|=\arctan\left(\sqrt{G_{II}/G_I}\right)$, where $G_I$ and $G_{II}$ are the mode I and II components of the strain energy release rate. Cracked lap shear (CLS) specimens were used to generate a loading phase angle of $50^\circ$ (Fig. 1). An adhesive bondline thickness of 0.4 mm was established by using spacing wires in the bondline. The cure cycle was monitored using an embedded thermocouple to ensure that the rubber toughened adhesive remained at $180^\circ$C for 30 min as recommended by the manufacturer. A cohesive precrack was made in the adhesive by placing a 20 $\mu$m thick folded aluminum foil in the bondline. The precrack length was 40 mm from the loading pins for DCB and ADCB joints, and 10 mm for the CLS specimens. The sides of DCB joints manufactured for quasi-static fracture testing were milled using a very sharp cutter to avoid edge damage and overheating. To remove the excessive adhesive after curing the fatigue specimens, the sides of the joints were sanded using a disc sander with water as a coolant, followed by gentle sanding using a belt sander with 120 grit paper. To improve the visibility of the crack and remove any surface damage, the bondlines were sanded with 600 grit sandpaper, and a thin coating of diluted white correction fluid was applied to provide a high-contrast image of bondline cracking. The pretreatment used for the aluminum and steel adherends are described in the following sections. The prepared specimens were then stored in desiccant boxes ready to be tested.
Fig. 1  Geometry of (a) DCB, (b) ADCB2 with thickness ratio of 2, (c) ADCB4 with thickness ratio of 4, and (d) CLS (not to scale). The mounted clip gauge on the ADCB joint is schematically shown in (b). All dimensions in mm unless stated.

2.1.1. Aluminum System

The aluminum joints were fabricated from AA6061-T651 flat bars. The fracture specimens and some of the fatigue joints were abraded using a silicon-carbide nylon-mesh abrasive pad which gave an average roughness of $R_a=0.77$ µm with standard deviation of 0.02 µm over 4 measurements. The majority of the fatigue specimens were abraded using a coarser aluminum oxide abrasive pad producing an $R_a=1.33$ µm with standard deviation of 0.16 µm over 4 measurements. The aluminum bars were then pretreated using the P2 etching process [17].

2.1.2. Degreased Steel System

Degreased steel joints were manufactured from AISI 1018 steel bars by first abrading the bonding surfaces using the aluminum oxide abrasive pad to produce an $R_a=1.44\pm0.15$ µm. The bars were then wiped using cheesecloth and acetone, degreased for 5 min in acetone, and finally rinsed with ethanol.

2.1.3. Zn-Phosphated Steel System

After abrasion with the aluminum oxide abrasive pad to produce an $R_a=1.44\pm0.15$ µm, the AISI 1018 steel bars were given a standard Zn-phosphate pretreatment as follows:

1. Pacocleaner® 319 (Henkel Corp.) immersion for 2 min at 57°C. 2. Rinse with tap
water for 30 s. 3. Fixodine® ZL (Henkel Corp.) immersion for 30 s. 4. Bonderite® 958 (Henkel Corp.) immersion for 2 min at 49°C. 5. Rinse with tap water for 30 s. 6. Parcolene® 99x (Henkel Corp.) immersion for 30 s. 7. Rinse with tap water for 30 s. 8. Dry in oven for 10 min at 121°C.

2.2. Fatigue Testing

All fatigue experiments were carried out at a cyclic frequency of 20 Hz, under force control, with a constant load ratio, $R=P_{\text{min}}/P_{\text{max}}=0.1$. A dry condition (11% - 15% relative humidity) was achieved by performing the experiments in a desiccant chamber.

The fatigue testing was divided into two parts: the threshold strain energy release rate, $G_{th}$, was identified first, followed by the measurement of the crack speed, $da/dN$, versus the applied strain energy release rate, $G$. The test started with a force value which gave a $G$ that was relatively large compared to $G_{th}$, and the crack growth rate was progressively slowed by decreasing the applied load in small steps of less than 10% of the maximum force until the fatigue threshold was established at a crack speed of $10^{-6}$ mm/cycle [18]. Prior to the start of the step-wise decrease in applied force, the crack had propagated about 3 mm from the foil precrack, thereby ensuring that $G_{th}$ corresponded to a fully developed fatigue crack-tip damage zone. After measuring $G_{th}$, the applied $G$ was increased by 10% and the corresponding load was kept constant throughout the remainder of the test. As the crack grew, this constant load caused a gradual increase in $G$ and the crack speed. The methodology for monitoring of the crack length is described in Section 2.3.2.

2.3. Crack Length Measurement and Data Reduction

2.3.1. Fracture Experiments

The quasi-static fracture testing approach was explained in detail in Chapter 2; therefore, the procedures are summarized only briefly. The load jig of ref. [13] was used to measure the critical strain energy release rate, $G_{cs}$, as a function of the phase angle using a DCB specimen. The load was increased with a constant crosshead speed of 1 mm/min. The crack length was measured using a microscope mounted on a micrometer stage. Crack growth was stable in this system so that many crack extension events could be recorded with a single DCB specimen. The critical load that caused the macro-crack to extend and the corresponding crack length were measured and used to calculate the critical strain energy release rate and phase angle with a beam-on-elastic-foundation model [16].
2.3.2. Fatigue Experiments

The unloading joint compliance approach [19] was used to measure the fatigue crack length. The joint compliance was measured during the unloading portion of the cycle using the load cell output and a clip gauge attached to the end of the specimen. A CCD camera (2 mm field of view) on a motorized linear stage was used to measure the crack length and relate it to the measured joint compliance for a given specimen type using the approach of ref. [18]. A least squares regression was used to fit a third-order polynomial to the normalized crack length, $a/w$, versus the normalized specimen compliance, $CEB$, for fatigue joints:

$$a/w = c_1 \times (CEB)^3 + c_2 \times (CEB)^2 + c_3 \times (CEB) + c_4$$  \hspace{1cm} (1)$$

where $a$ is the crack length, $w$ is the specimen length from the loading pins, $C$ is the compliance, $E$ is the tensile modulus of the adherends, and $B$ is the specimen width. $c_1 - c_4$ are the regression coefficients, calculated from the curve fitting. For CLS joints, $w$ was considered to be the initial bond length.

For the DCB and ADCB joints, the clip gauge was mounted at the end of the specimen (Fig. 1(b)), while for CLS specimens, a block was bonded close to the overlap end and the clip gauge was mounted between the block and the overlap end (Fig. 1(d)). Fatigue set-up for DCB and ADCB testing is shown in Fig. 2.

The strain energy release rate for DCB and ADCB joints was calculated from the measured force and crack length using an analytical beam-on-elastic-foundation model. The analytical model of Fernlund and Spelt for DCB joints [16] was modified to allow $G$ calculation for ADCB specimens as well; which gives

$$G = 12(Pa)^2(A + B)$$  \hspace{1cm} (2)$$

where

$$A = \frac{1}{2E_a h_u^3} \left[ 1 - \frac{1}{1-t_i/h_u} \right]^3 \left[ 1 + 0.667 \left( \frac{1-t_i/h_u}{1+t_i/h_u} \right) \left( \frac{2E_a/E_u - 1}{h_u/a} \right) \right]^{0.25} \frac{h_u}{a} \right]^2$$  \hspace{1cm} (3)$$

and

$$B = \frac{1}{2E_i h_i^3} \left[ 1 - \frac{1}{1-t_u/h_i} \right]^3 \left[ 1 + 0.667 \left( \frac{1-t_u/h_i}{1+t_u/h_i} \right) \left( \frac{2E_i/E_u - 1}{h_i/a} \right) \right]^{0.25} \frac{h_i}{a} \right]^2$$  \hspace{1cm} (4)$$
and $P$ is the force per unit width, $E$ is the elastic modulus, $t$ is the adhesive thickness and $h$ is the adherend thickness. The subscripts $a$, $u$ and $l$ refer to the adhesive, and the upper and the lower substrates, respectively. By setting $h_l=h_u$, these equations allow for the calculation of $G$ for DCB joints.

![Experimental set up for DCB and ADCB fatigue testing.](image)

The accuracy of the beam-on-elastic-foundation model and the contribution of the adhesive compliance to the calculated $G$ were investigated by comparing with three different $G$ calculation approaches as explained in Appendix 3.A. It was observed that as the phase angle increased, the crack path at the threshold became closer to the more highly strained adherend (ADCB or CLS). This shift in the crack path from the mid-plane of the adhesive layer changed the ratio between the residual adhesive thicknesses bonded to the upper and the lower adherends, and thus the elastic foundation modulus for the adherends in Eq. (3), which resulted in a slight increase, about 1-2%, in the calculated $G$. The finite element model of Appendix 3.A was used to calculate the change in the phase angle at the crack tip due to the shift in the crack path from the mid-plane. However, this change was found to be small; for instance it decreased the phase angle at the crack tip for an ADCB joint by less than $1^\circ$.

To calculate the energy release rate for CLS joint, an “adhesive sandwich” was isolated.
as shown in Fig. 3, and the J-integral for this sandwich was calculated as [20]:

\[
G = \left[ \frac{F_i^2}{2 (Eh)} + \frac{M_i^2}{2 D_i} + \frac{1.2 V_i^2}{2 (G_s h_i)} \right] + \left[ \frac{F_2^2}{2 (Eh)_2} + \frac{M_2^2}{2 D_2} + \frac{1.2 V_2^2}{2 (G_s h)_2} \right] - \left[ \frac{F_0^2}{2 (Eh)_0} + \frac{M_0^2}{2 D_0} + \frac{1.2 V_0^2}{2 (G_s h)_0} \right]
\]  

where \( F, M \) and \( V \) are, respectively, the tensile force, the bending moment and the shear force per unit width in the adherends at the crack tip, while \( D \) is the flexural rigidity (defined below), \( h \) is the total adherend thickness at the cross-sections 0, 1 and 2 shown in Fig. 3, and \( G_s \) is the shear modulus at each cross-section. The tensile forces and bending moments were found from the equation for beam deflection, \( v_i \), at section \( i \) [21]:

\[
\frac{\partial^4 v_i}{\partial x^4} - \frac{P}{D_i} \frac{\partial^2 v_i}{\partial x^2} = 0 
\]

which has the general solution

\[
v_i = C_{i1} x + C_{i2} + C_{i3} \cosh(\lambda_i x) + C_{i4} \sinh(\lambda_i x)
\]

\( C_{i1}-C_{i4} \) are constants in the solution, with

\[
\lambda_i = \sqrt{\frac{P}{D_i}}
\]

where \( D_i \) is the flexural rigidity per unit width of the beam under plane stress at section \( i \)

\[
D_i = \frac{Eh^3}{12}
\]

A CLS joint was divided into three beam sections with lengths \( L_1, L_2 \) and \( L_3 \), as can be seen in Fig. 1(d). For each section, denoted by the subscript \( i \), the beam deflection was assumed according to Eq. (6). Applying the boundary conditions, continuity of displacements and slopes for the overlap end, and considering that the difference in bending moments at both ends of the overlap must be the moment due to the tensile force, leads to twelve equations in twelve unknowns. Appendix 3.B lists these equations which were solved to find the beam deflections and the resulting bending moments and shear forces for the calculation of \( G \) and the phase angle as explained in [21] for a simpler CLS joint. The calculated bending moments and shear forces were verified with a finite element analysis. The results for the \( G \) calculation using Eq. (4) were compared with both an adhesive sandwich model that considers the contribution of the adhesive
to the total $G$ [22], and also a finite element analysis with the adhesive layer present. The difference was less than 2% compared to the adhesive sandwich model, and less than 5% compared to the FEA. Therefore, it was concluded that the effect of the adhesive on the calculated $G$ for CLS joints could be ignored. This is consistent with the observation that for DCB and ADCB joints, as the phase angle increased the error of ignoring the presence of adhesive in the calculated $G$ decreased. As an example, for a crack length of 60 mm, the adherend thickness ratio, $h_l/h_u$, was varied from 1 to 8, resulting in an increase in phase angle from 0° to 26°. The error due to ignoring the adhesive was 23%, 19%, 13% and 8% for adherend thickness ratios of 1, 2, 4 and 8, respectively. In this example, the adherend thicknesses were chosen such that the total adherend stiffness of the joints, and thus the contribution of the adhesive layer compliance to the total joint compliance, remained constant. This ensured that the changes seen in the calculated errors were due only to the increase in phase angle.

![Fig. 3 Cracked adhesive sandwich model at the end of the overlap of CLS joints.](image)

**3. Experimental Results and Discussions**

**3.1. Effect of Loading Phase Angle on Fatigue and Fracture Behavior**

**3.1.1. Aluminum Adhesive System**

*Effect of phase angle on fracture $G_c$*

To understand the effect of loading phase angle on the quasi-static fracture of the aluminum adhesive system, the approach explained in Section 2.3.1 Chapter 2 was followed. After crack initiation at $G_c$, the first several crack growth sequences occurred at an increasing critical energy release rate, $G_c$, as the damage zone at the crack tip developed to its steady-state
form (Fig. 4) [13,14]. The steady state critical strain energy release rate, $G_c^s$, at each phase angle was considered to be the average value over the “plateau” (steady-state) region.

![Graph](image)

**Fig. 4** Typical R-curve behavior of an aluminum DCB joint tested at a phase angle $\psi=27^\circ$.

The fracture envelope for the aluminum adhesive system (Fig. 5) showed a weak dependence of $G_c^s$ on $\psi$ in the range $\psi = 0^\circ-30^\circ$, becoming more significant at higher phase angles, similar to what was previously reported for more brittle adhesive systems [14,21].

With mode I loading, the crack propagated cohesively in the mid-plane of the bondline. Under mixed-mode loading, increasing the phase angle resulted in a crack path that was closer to the more highly strained arm of the DCB joint, producing a cohesive fracture surface with more shear hackles in accordance with expectations [23,24]. FE analysis also showed that increasing the loading phase angle shifted the mode I plane in the bondline towards the more strained arm, which was consistent with ref. [23]. Crack growth was cohesive at all phase angles so that it is expected that these fracture results were independent of the aluminum roughness.
Fig. 5 Fracture envelope for aluminum adhesive system ($R_a=0.77 \, \mu m$) (Chapter 2). Given values are average $G_{cs}$ ($\pm 1$ SD). Number of specimens tested is shown above each data point. Total number of data points at each phase angle: $0^\circ$-$155$, $16^\circ$-$163$, $27^\circ$-$44$, $48^\circ$-$25$, $54^\circ$-$14$, $55^\circ$-$29$ and $65^\circ$-$46$.

**Effect of phase angle on fatigue – rougher aluminum**

Figure 6 shows the measured $G_{th}$ for the rougher aluminum adhesive system ($R_a=1.33 \, \mu m$), at four phase angles, $\psi$, corresponding to DCB, two different ADCB’s and CLS joints. It is observed that the effect of phase angle on $G_{th}$ was similar to that observed for $G_{cs}$ (Fig. 5); i.e. a negligible effect was observed at $\psi<25^\circ$, and a very significant effect was observed at higher phase angles ($G_{th}$ increased by about 80% from $\psi=24^\circ$ to $\psi=50^\circ$). T-tests showed that, with 95% confidence, neither the difference in the measured $G_{th}$ between mode I and $\psi=18^\circ$, nor between $\psi=18^\circ$ and $\psi=24^\circ$ were statistically significant.

$G_{th}$ was found to be between 4 and 5% of $G_{cs}$ regardless of the mode ratio, showing the destructive effect of cyclic loading. The $G_{th}/G_{cs}$ ratio for the current highly toughened epoxy adhesive was compared with similar values in the literature (Table 1). In general, it seems as if the toughening mechanisms used to improve the quasi-static toughness have not resulted in an improved fatigue behavior. In fact, the $G_{th}/G_{cs}$ ratios for the low to medium toughness adhesives are higher than those for the highly toughened adhesives.
Fig. 6  Effect of loading phase angle, $\psi$, on $G_{th}$ for the aluminum adhesive system with average roughness of $R_a=1.33$ µm. Given values are average $G_c^s$ (±1 SD). Number of tested specimens is shown above each data point.

Table 1  Comparison of the fatigue and fracture behavior of different adhesives.

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>$G_c$ (J/m$^2$)</th>
<th>$G_{th}$ (J/m$^2$)</th>
<th>$G_{th}/G_c$</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hysol (EA9309)</td>
<td>5700</td>
<td>150</td>
<td>0.03</td>
<td>25</td>
</tr>
<tr>
<td>Toughened Epoxy</td>
<td>3860</td>
<td>195</td>
<td>0.05</td>
<td>-</td>
</tr>
<tr>
<td>Ciba (XD4600)</td>
<td>3500</td>
<td>355</td>
<td>0.10</td>
<td>25</td>
</tr>
<tr>
<td>Cyanamid (FM73M)</td>
<td>2930</td>
<td>285</td>
<td>0.10</td>
<td>26</td>
</tr>
<tr>
<td>3 M (AF-I 63-2M)</td>
<td>1720</td>
<td>560</td>
<td>0.33</td>
<td>3</td>
</tr>
<tr>
<td>Hysol (EA9628)</td>
<td>1700</td>
<td>215</td>
<td>0.13</td>
<td>25</td>
</tr>
<tr>
<td>Epoxy film adhesive</td>
<td>1690</td>
<td>250</td>
<td>0.15</td>
<td>27</td>
</tr>
<tr>
<td>Teroson (Terokal4520-34)</td>
<td>740</td>
<td>240</td>
<td>0.32</td>
<td>25</td>
</tr>
<tr>
<td>Ciba (AV119)</td>
<td>450</td>
<td>85</td>
<td>0.19</td>
<td>28</td>
</tr>
<tr>
<td>Cybond (4523GB)</td>
<td>212</td>
<td>50</td>
<td>0.24</td>
<td>10</td>
</tr>
</tbody>
</table>

As in the fracture tests, the fatigue crack grew in the mid-plane of the bondline under mode-I loading and moved closer to the more highly strained adherend as the phase angle increased. For example, the residual adhesive thickness remaining on the more highly strained adherend at the fatigue threshold at phase angles of 0°, 18° and 24° was measured as 200 µm, 80 µm and 20 µm, respectively. In addition, it was observed that the crack tended to move progressively closer to the more highly strained adhered as the crack speed decreased. Both the phase angle and crack speed effects are illustrated in Fig. 7 which shows that, although the fatigue crack path was cohesive in all cases, the crack was observed to propagate much closer to
the more highly strained adherend at higher phase angles (i.e. in the CLS joints), and when the crack speed was low (i.e. near $G_{th}$). For CLS joints, the residual adhesive on the more highly strained adherend formed an extremely thin layer with some small scattered pieces of adhesive attached. The layer between the scattered pieces of the adhesive appeared as a dull surface, as opposed to the shiny surface of the aluminum. An XPS (x-ray photoelectron spectroscopy) surface analysis showed that the percentage of carbon and oxygen on this dull portion of the failure surface matched closely to that of the adhesive, confirming that the crack path was fully in the adhesive, and not in the interface of aluminum and adhesive.

Figure 8 shows the fatigue crack speed as a function of $G_{max}$ for three aluminum DCB joints (mode I) with adherends having $R_a=1.33$ µm. The threshold and the linear (Paris law) regions are evident in the figure.

Figure 9 shows how $\psi$ affected the crack growth rate for the adhesive system ($R_a=1.33$ µm), using one representative test for each of the DCB and ADCB. Because of the large cyclic applied forces necessary for CLS joints, the repeated premature failure of the aluminum adherend prevented a single specimen from being used over a wide enough range of $G_{max}$. Therefore, the CLS results are a combination of data from three specimens. The maximum crack length of the CLS joints before aluminum failure was approximately 50 mm.
Fig. 7 Fatigue failure surfaces on the more highly strained adherend near the threshold. Rougher aluminum specimens with $R_a=1.33$ µm: (a) DCB, (b) ADCB with thickness ratio of 2, (c) ADCB with thickness ratio of 4, and (d) CLS. Crack speed increased from left to right beyond the threshold region.
Fig. 8  Fatigue crack growth rate for three aluminum DCB joints with $R_a=1.33$ µm tested under mode I.

Fig. 9  Effect of loading phase angle, $\psi$, on fatigue crack growth rate behavior of the rougher aluminum adhesive system with $R_a=1.33$ µm.  $m$ and $b$ are as per Eq. (9), SD is the standard deviation, $N$ is the number of specimens.
It is again observed that $\psi$ had little effect on the fatigue behavior in the range of $0^\circ \leq \psi \leq 25^\circ$, but caused a significant decrease in the crack growth rates at a given $G$ at $\psi=50^\circ$ (CLS specimen). The experimental scatter, both for the measurement of $G_{th}$ and the crack growth rates, was higher for the CLS than the DCB and ADCB specimens. This was partly because the CLS data were from multiple specimens, each providing data over a relatively narrow range of $G_{\text{max}}$. Another reason for the increased scatter might be related to the manner in which these specimens were loaded. The change in the applied $G$ with crack length at a constant force is not as large in CLS joints as it is in DCB or ADCB joints. Therefore, the $G$ in CLS joints was increased by manually increasing the applied load in small 5% increments, whereas for the DCB and ADCB specimens, the load was kept constant and the $G$ increased gradually as the crack grew. The non-gradual increase in the applied $G$ for the CLS specimens might have resulted in a non-smooth increase in the crack speed with the applied $G$, leading to the increased scatter. A final reason for the increased scatter seen in CLS joints could be the proximity of the crack path to the interface. Any inconsistency in the interface condition, such as a change in the local surface roughness, could affect the fatigue behavior, increasing the experimental scatter.

In the Paris law region there is a linear relation between the log $(da/dN)$ and log $(G_{\text{max}})$:

$$\log (da/dN) = m \times \log (G_{\text{max}}) + b \quad (9)$$

The average values for $m$ and $b$ at different phase angles are given in Fig. 9. The slope, $m$, for fully cohesive failures was in the range of 4-5, which is typically seen for adhesives [1,4,7,11,29,30]; however, for CLS joints the slope decreased to 2.5. The slope $m$ can be an indication of the destructive effect of cyclic loading, i.e. the lower the magnitude of $m$ the higher the difference between the strain energy release rate at the low and the high crack speeds. Therefore, it is expected that the slope $m$ for CLS joints should decrease from the range 4-5, measured for the fully cohesive failure. This is due to the observation that for CLS joints, high phase angle, the cyclic loading makes the failure become less cohesive and more interfacial as the crack speed decreased. This worsening of the fatigue behavior due to the failure mode change is depicted in the drop of $m$.

**Effect of surface roughness**

The fatigue experiments with the aluminum substrates were initially performed using the same adhesive system as used for the DCB fracture specimens with a surface roughness $R_a=0.77$
µm. This system fractured cohesively under quasi-static loading at all the tested phase angles; however, under cyclic loading at very low crack speeds near the threshold the failure surfaces for DCB and ADCB joints exhibited great variability, being at times fully cohesive, or fully interfacial, or sometimes a combination of the two failure modes. Interestingly, for DCB and ADCB joints at relatively high fatigue crack speeds, the failure was always fully cohesive as seen in Fig. 10 (a)-(b). When the surface roughness was increased to $R_a=1.33$ µm, the failure became fully cohesive for all the tested DCB and ADCB joints at all crack speeds in fatigue. Therefore, it appears as if the interfacial bond strength was insufficient on the smoother aluminum substrates ($R_a=0.77$ µm), for very small crack speeds, with the roughness effect disappearing in the fracture tests and at the higher fatigue crack growth rates of DCB and ADCB joints.

Although the smoother aluminum system ($R_a=0.77$ µm) produced greater variability in the failure mode of DCB and ADCB joints near $G_{th}$, three CLS joints with $R_a=0.77$ µm were also tested. They exhibited a fully interfacial failure (Figure 10 (c)) at low crack speeds near threshold, giving an average $G_{th}$ of 168 J/m² with a standard deviation of 47 J/m². This was significantly lower than the $G_{th}$ of 305 J/m² obtained for the $R_a=1.33$ µm for the CLS joints (Fig. 6) because of the difference in crack path (interfacial for smooth, as opposed to cohesive for rough aluminum).

To confirm that the interfacial failure of the $\psi=50^\circ$ CLS specimen seen in the smoother aluminum system (Fig. 10) was due only to the increase in the phase angle, a mode I DCB specimen with $R_a=0.77$ µm was manufactured and tested under cyclic loading. As expected, a fully cohesive failure was observed, even at threshold. After reaching the threshold, the same specimen was modified to be a CLS joint, and tested again under cyclic loading. Figure 11 shows that immediately at the start of the CLS mixed-mode loading, when the $\psi$ increased from $0^\circ$ to $50^\circ$, the crack path changed from cohesive to interfacial. The crack path was interfacial over the tested range of crack speeds ranging from $10^{-6}$ mm/cycle to $5\times10^{-4}$ mm/cycle. At the end of the CLS fatigue testing, the specimen was opened with a chisel, and the crack path again became cohesive. This confirmed that the observed change in the fatigue crack path was due to the change of the loading phase angle, and not as a result of any inconsistency in specimen preparation.

Since all of the smooth aluminum specimens failed cohesively in the fracture tests at phase angles up to $65^\circ$, it is clear that fatigue testing can induce interfacial failure not seen in quasi-static tests. This illustrates the importance of evaluating the integrity of an adhesive
system based on both quasi-static and fatigue loading.

Fig. 10  Fatigue failure surfaces of smoother aluminum joints (R_a=0.77 µm): (a) ADCB2 (ψ=18°), (b) ADCB4 (ψ=24°), (c) magnified surface of CLS (ψ=50°), showing mainly bare aluminum.
Comparison of Figs. 7 and 10 demonstrates how an increase in adherend surface roughness affected the residual adhesive thickness on the more highly strained adherend under mixed-mode loading. The rougher specimens had more residual adhesive at the threshold, but the surface roughness did not have a noticeable effect on the cohesive $G_{th}$ since the crack path was still cohesive in the ADCB specimens regardless of roughness. The effect of increasing surface roughness was not noticeable at higher crack speeds, with the amount of residual adhesive being the same for both aluminum roughnesses. As well, the aluminum roughness had no effect on the crack path under mode I loading since the crack path was in the mid-plane of the adhesive layer.

3.1.2. Degreased Steel Adhesive System

The fracture envelope, $G_c(\psi)$, of the degreased steel adhesive system was measured previously in Chapter 2 and expressed as:

$$G_c^s = 2741.1 + 2.3884\psi + 0.1423\psi^2 + 0.025\psi^3$$

(10)

Under quasi-static loading the crack path was mostly cohesive, with 10%-20% of the failure surface being interfacial. These small interfacial patches in the crack path are believed to
be the reason for the lower $G_c^s$ of the steel system when compared to the aluminum system. Figure 12, which shows the failure surfaces for both fatigue and fracture tests performed on the same ADCB specimen demonstrates that there was a fully interfacial fatigue failure for the steel system at all the tested crack speeds ($10^{-6}$ - $10^{-2}$ mm/cycle range). This was the case for all the phase angles, including mode I loading. Figure 13 shows that $G_{th}$ for the degreased steel adhesive system varied with $\psi$ in a manner that was quite similar to that seen with the aluminum system (Fig. 6) even though the crack paths were quite different. As with the aluminum system, $\psi$ had little influence at low phase angles, but had a large effect at higher $\psi$ (comparing CLS to ADCB results). The interfacial crack path in the degreased steel system decreased $G_{th}$ to between one-third to one-fifth that of the aluminum system.

![Fig. 12 Failure surface of a degreased steel ADCB joint.](image)

Figure 14 shows that, as was the case for the aluminum system (Fig. 9), an increase in $\psi$ reduced the fatigue crack growth rates for the degreased steel system. The slope of the linear part of the crack speed graphs was approximately 2 for all $\psi$ (Fig. 14), which was comparable to that for the aluminum CLS joints where fatigue cracking was also very near to the interface. Therefore, the adherend stiffness had no discernible effect on this aspect of fatigue crack propagation.

These experiments further highlighted the great sensitivity of fatigue experiments, particularly at low crack speeds, to the surface treatment and hence the interfacial bond strength. The purely interfacial fatigue failures contrasted with the cohesive crack paths observed in the quasi-static fracture tests with the same adhesive system, reported in Chapter 2 and ref. [15].
Fig. 13  Effect of loading phase angle, $\psi$, on $G_{th}$ of the degreased steel adhesive system. Given values are average $G_c$ ($\pm$ 1 SD). Four specimens were tested at each phase angle.

Fig. 14  Effect of loading phase angle, $\psi$, on fatigue crack growth rate behavior of the degreased steel adhesive system. Data for four CLS specimens and a single ADCB specimen are shown. $m$ and $b$ are as per Eq. (9), SD is the standard deviation, $N$ is the number of specimens.

3.1.3. Zn-phosphated Steel Adhesive System

Figure 15 shows that the effect of $\psi$ on $G_{th}$ was similar to the previous two systems.
Figure 16 shows a sample of the failure surfaces of the Zn-phosphated steel joints. Fully cohesive fatigue cracks were observed for the lower phase angle DCB and ADCB specimens, and close to interfacial cracks (i.e. with small pieces of adhesive on the strap adherend) for the high phase angle CLS joints. The more cohesive crack path resulted in $G_{th}$ values which were 4-5 times higher than those of the degreased steel system (Fig. 13). Similar to the aluminum system (Figs. 7 and 10), as the phase angle increased the crack path became closer to the interface of the more highly strained substrate.

The scatter in the measured $G_{th}$ for adhesive systems which resulted in cohesive crack paths (i.e. the DCB and ADCB joints for the rougher aluminum system and the Zn-phosphated steel system) was much lower than that seen in the degreased steel specimens which resulted in interfacial failure. The standard deviation divided by the average $G_{th}$ for the two cohesive systems was in the range of 2% to 10%, while it was 19% to 29% for the degreased steel system. The CLS joints of the rougher aluminum ($R_d=1.33 \, \mu m$) and the Zn-phosphated steel systems, which had crack paths that were very close to the interface, resulted in a higher scatter in measured $G_{th}$ and crack growth rates than those seen in the DCB and ADCB of those systems, as explained in Section 3.1.1. It thus appears as if experimental scatter increases as the crack path approaches the adhesive/adherend interface.
Fig. 16 Fatigue failure surfaces of Zn-phosphate treated steel adhesive systems: (a) DCB, (b) ADCB, (c) CLS at the threshold.
Figure 17 shows how $\psi$ affected the fatigue crack growth behavior of the Zn-phosphated steel joints. Because of the good repeatability of the fatigue results and for clarity of the figure, only one representative test is given for each DCB and ADCB case, while the data points for CLS are from 4 specimens in order to be able to cover a broad range of crack speeds. As with the aluminum system, the CLS data (corresponding to the highest $\psi$) show a significantly lower crack growth rate with a large amount of scatter. The Paris law slopes in Fig. 17 for the fully cohesive failures of the DCB and ADCB are in the range of 4-5, while the slope is 2.7 for the CLS joints. These values are close to the measured slopes for the aluminum system. In all of the present experiments, the slope tended to decrease to about 2 when the fatigue crack path became interfacial or near-interfacial, and near 4 when it was fully cohesive. The Paris law slope can be an indication of the destructive effect of cyclic loading. The lower the slope the larger the difference between the applied $G$ at the high and the low crack growth rates. DCB and ADCB joints produced a fully cohesive failure over the full range of tested $da/dN$. CLS joints also produced a fully cohesive failure under fracture loading and high crack growth rates. However, as the crack growth rate decreased the crack path became less cohesive. As a result, while in the case of DCB and ADCB tests, the Paris law slope was only an outcome of fatigue loading, in CLS joints further decrease in the slope was resulted from the destructive effect of the change in the failure mode from cohesive in the adhesive to interfacial.

Figure 18 compares the crack speed of the degreased and Zn-phosphated steel ADCB joints. Clearly, the surface treatment had a significant effect on the fatigue performance at all crack speeds because of the change in the crack path.
Fig. 17 Effect of loading phase angle, $\psi$, on fatigue crack growth of the Zn-phosphated steel adhesive system. Data for four CLS specimens and a single DCB and ADCB specimens are shown. $m$ and $b$ are as per Eq. (9), SD is the standard deviation, $N$ is the number of specimens.

<table>
<thead>
<tr>
<th>$\psi$ (°)</th>
<th>$m$ ± SD</th>
<th>$b$ ± SD</th>
<th>$N$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>4.42 ± 0.24</td>
<td>-15.64 ± 0.52</td>
<td>3</td>
</tr>
<tr>
<td>18</td>
<td>4.82 ± 0.31</td>
<td>-16.87 ± 0.81</td>
<td>3</td>
</tr>
<tr>
<td>50</td>
<td>2.71</td>
<td>-12.34</td>
<td>4</td>
</tr>
</tbody>
</table>

Fig. 18 Effect of surface treatment on the fatigue crack growth rate behavior of steel adhesive systems.
3.2. Effect of Substrate Stiffness on Fatigue Behavior

Several studies in the literature have noted an effect of substrate stiffness on the critical strain energy release rate at quasi-static fracture. For example, Yan et al. [31] observed that the $G_c^s$ for an adhesive system with steel adherends was less than that of the same adhesive with aluminum adherends. This was attributed to elevated stress levels in the crack tip region for the stiffer steel joint. On the other hand, Bell and Kinloch [32] compared the $G_c^s$ of aluminum, steel and CFRP joints and found that $G_c^s$ increased with adherend stiffness. This was attributed to the shape and the size of the plastic zone ahead of the tip and within the adhesive layer which was believed to be affected by the transverse elastic modulus of the adherends. Later however, it was found that by repeating the experiments using a similar adhesive, the difference between the aluminum and steel joints was less pronounced [33]. The difference between the CFRP and the metallic adherend specimens was attributed to water absorbed by the CFRP substrates, and a much less pronounced effect was observed when the composite substrates were fully dried before bonding [33].

There are other possible explanations for differences in adhesive fracture behavior when steel and aluminum adherends are used. The greater mismatch between the coefficients of thermal expansion between the adherend and the adhesive for the steel system is expected to result in larger residual stresses after curing [34] than the aluminum system. Moreover, the difference in stiffness between the aluminum and steel adherends also produces a difference in crack tip strain rate. Finally, the constraint induced by the substrate on the adhesive layer is larger in the steel adhesive system, which may affect the state of stress at the crack tip. It was shown in Chapter 2 that degreased steel adherends produced a lower $G_c^s$ than aluminum adherends for the same adhesive used in this study. However, the lower $G_c^s$ was attributed to a change in the crack path which was fully cohesive at all phase angles with aluminum, but had small scattered interfacial patches on the degreased steel adherends.

Statistical analysis of $G_{th}$ on the rough aluminum and Zn-phosphated steel (Figs. 6 and 15) showed that the difference between the two systems at $\psi=18^\circ$ (ADCB) was statistically insignificant with 95% confidence. Similarly, no effect of adherend stiffness on the crack growth rate of ADCB joints was observed. Under mode-I loading, the effect was very small, but statistically significant (t-test, 95% confidence). Considering a three-fold increase in the stiffness of the substrates from aluminum to steel, it is thus likely that neither of the aforementioned stiffness effects had a very significant effect on the measured fatigue behavior. The statistically significant (95% confidence interval) higher $G_{th}$ seen in steel versus aluminum...
CLS joints could either be due to the large scatter or due to a slightly higher interfacial strength in the steel joints. This last possibility is supported by the fact that a larger amount of residual adhesive was found on the steel adherends in the region where the threshold was obtained, than the aluminum ones (compare Figs. 7(d) and 16(c)).

Therefore, it is observed that the change in the substrate material does not significantly affect the fatigue behavior of the current adhesive system, unless the crack path is affected due to a poor bonding and surface treatment. It should be noted that more significant substrate stiffness effects may have been observed if the changes in the substrate modulus were larger than those in the present systems, or if the studied adhesive was more viscoelastic.

4. Conclusions

The fatigue behavior of different adhesive systems under mixed-mode loading was investigated in order to examine the possibility of characterizing the fatigue behavior of an adhesive system as a fatigue envelope, similar to fracture envelope which was previously examined for quasi-static loading [12-15]. A broad range of phase angles, 0° to 50°, was achieved by utilizing DCB, two types of ADCBs, and CLS joints. Various adhesive systems and fatigue failure modes were tested by changing the substrates, the surface roughness and the adherend surface pretreatment. The conclusions can be summarized as follows:

Under quasi-static loading, the loading phase angle had a small effect on $G_c^s$ for $\psi < 30^\circ$, and strongly affected the $G_c^s$ at higher phase angles.

Under cyclic loading, the effect of $\psi$ on the $G_{th}$ and the fatigue crack growth rate was similar to that seen in quasi-static loading, i.e. $\psi$ affected the fatigue behavior only at $\psi > 25^\circ$. The fatigue performance was found very sensitive to the failure pattern. The more cohesive the failure at higher phase angles, the higher the $G_{th}$ and the better the fatigue performance.

The threshold strain energy release rate, $G_{th}$, was 4-5% of the steady-state critical strain energy release rate of quasi-static fracture, $G_c^s$, for phase angles between 0° and 50°. Comparing this with the literature suggests that the increased fracture toughness of the present adhesive has not resulted in a commensurate improvement in the fatigue behavior.

Surface roughness was found to improve the adhesive bonding under cyclic loading. For the aluminum adhesive system at $\psi < 25^\circ$, an increase in the surface roughness increased the residual adhesive thickness on the more highly strained arm and resulted in a fully cohesive failure. This resulted in an improvement in the fatigue behavior at higher phase angles.
Compared to static loading, the fatigue testing was found to be more sensitive to the characteristics of an adhesive system, such as surface roughness and surface treatment, especially at lower crack speeds. This was consistent with the observation that the residual adhesive thickness on the more highly strained adherend decreased with decreasing \( G \); i.e. it was maximum for quasi-static loading and was progressively smaller as the crack speed fell during fatigue crack propagation, becoming nearly interfacial near the fatigue threshold. Consequently, the strain energy release rate at the fatigue threshold was more sensitive to the interfacial bond strength and the interface condition than at higher crack speeds and under quasi-static fracture.

An adhesive system which performs well under static loading does not necessarily perform well under cyclic loading, and it is thus important to choose the proper surface preparation technique for joints that will undergo cyclic loading. Due to this high sensitivity, the characterization of the fatigue behavior of an adhesive system using the concept of the fatigue envelope should be performed on the adhesive system that is intended for the design and service application.

Ignoring the presence of the adhesive will significantly underestimate the strain energy release rate, \( G \), calculations at low loading phase angles. The beam on elastic foundation model resulted in a very good agreement with the finite element calculations for \( G \). The analysis showed that ignoring the deflection of the crack path from the mid-plane of the adhesive layer towards one interface, which is caused by the increase in the loading phase angle, will only slightly underestimate the calculated \( G \) and overestimates the phase angle at the crack tip.

Comparing the rougher aluminum adhesive system (\( R_a = 1.33 \mu m \)) and the Zn-phosphated steel systems, which produce similar fatigue failure modes, showed a very small effect of the substrate material on the fatigue threshold and fatigue crack growth rates. The change in the substrate material can affect the fatigue performance if it affects the bonding because of the changes in the surface preparation of the new system.
Appendix 3.A. Accuracy of Beam-on-Elastic-Foundation Model

The first comparison was with a simple beam theory model for a homogenous, isotropic layer, which ignores the contribution of the adhesive layer compliance to the total strain energy release rate [24]. Assuming the adherends to be in plane stress, the $G$ for the DCB and ADCB joints was given by:

$$G = \frac{6M^2}{E} \left[ \frac{1}{h^3} + \frac{1}{H^3} \right]$$  \hspace{1cm} (A.1)

where $h$ and $H$ are the upper and lower adherend thicknesses, respectively, and $M = P \times a / B$, is the applied bending moment at the crack tip per unit width, where $P$ is the applied load at the loading pins.

A second comparison was with a two-dimensional elasto-plastic finite element analysis (FEA) carried out in ANSYS® 11 (Ansys Inc., Canonsburg, PA). The structural model used plane182 elements with plane stress assumptions for the substrate and plane strain for the adhesive. The adhesive was modeled with a bilinear stress-strain curve derived from tensile tests. To account for plasticity effects, bilinear isotropic hardening using a von Mises yield criterion was implemented in the FEA. The material properties used in the model are given in Table A1. The energy release rate was calculated using a virtual crack extension approach.

The third comparison used the equation [35]:

$$G = \frac{P^2}{2B} \frac{\partial C}{\partial a}$$  \hspace{1cm} (A.2)

where the rate of change of compliance, $C$, with crack length, $a$, $\partial C/\partial a$ was either calculated using FEA or determined experimentally from the clip gauge readout. The advantage of the experimental approach was that no information was needed concerning the material properties of the substrate and the adhesive, since it required only the measured force and optically measured crack length.

Figure A1 compares the calculated $G$ values using the four different approaches. The analyses were performed at a constant force, while the crack length was increasing, for an aluminum ADCB joint ($\psi=18^\circ$). It is seen the contribution of the adhesive to the total joint compliance, and hence the total $G$, decreases as the crack grows. Since this contribution
increases as the stiffness of the substrates increases, the error due to ignoring the adhesive will be larger for the steel system compared to the results shown in Fig. A1 for the aluminum system. Apart from the simple beam theory, the other approaches resulted in very similar values of $G$; e.g. an average difference of 3% between the beam-on-elastic-foundation model and the FEA. Similarly good agreement was found when comparing the beam-on-elastic-foundation model with the $G$ values from the finite element $\partial C/\partial a$ approach for the aluminum DCB, steel DCB and steel ADCB; giving average errors of 1.5%, 2.3% and 0.7%, respectively. The beam theory predictions became closer to those of the beam-on-elastic-foundation model as the phase angle increased.

Table A1  Mechanical properties of adherends and the adhesive used in the FE model. Yield stress, $\sigma_y$, tensile elastic modulus, $E$, and tensile tangent modulus, $E_t$.

<table>
<thead>
<tr>
<th>Material</th>
<th>$\sigma_y$ (MPa)</th>
<th>$E$ (GPa)</th>
<th>$E_t$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum, AA6061-T6</td>
<td>270</td>
<td>68.9</td>
<td>0</td>
</tr>
<tr>
<td>Steel, AISI 1018</td>
<td>370</td>
<td>206</td>
<td>0</td>
</tr>
<tr>
<td>Adhesive</td>
<td>37</td>
<td>1.96</td>
<td>0.140</td>
</tr>
</tbody>
</table>

Fig. A1  Comparison of the $G$ calculation from the finite element model (FE), beam-on-elastic-foundation (BOEF) model, beam theory (BT), finite element compliance curve (FE Curve), and experimental compliance curve (Exp Curve) for an aluminum ADCB joint.
Appendix 3.B. Beam Deflection Analysis for CLS Joints

The CLS joints were divided into three beam sections of different stiffness (Fig. 1(d)), each of which had a deflection described by Eq. (6). As a result, twelve constants, $C_1$-$C_{12}$, four for each section, must be determined using the boundary and continuity conditions. The following set of equations were solved numerically to find the constants, $C_1$-$C_{12}$, required to calculate the deflections of CLS joints, Eq. (6), and the resulting $G$ and phase angle:

\[- C_1 L_1 + C_2 + C_3 \cosh(\lambda_1 L_1) - C_4 \sinh(\lambda_1 L_1) = 0 \]
\[C_5 \cosh(\lambda_1 L_1) - C_4 \sinh(\lambda_1 L_1) = 0 \]
\[C_2 + C_3 - C_6 - C_7 = 0 \]
\[C_1 + C_4 \lambda_1 - C_5 - C_8 \lambda_2 = 0 \]
\[C_3 (-D_1 \lambda_1^2) + C_7 D_2 \lambda_2^2 = -P \Delta \]
\[C_4 (D_1 \lambda_1^3) + C_8 D_2 \lambda_2^3 = 0 \]
\[C_5 L_2 + C_6 + C_7 \cosh(\lambda_2 L_2) + C_8 \sinh(\lambda_2 L_2) - C_9 L_2 - C_{10} - C_{11} \cosh(\lambda_3 L_2) - C_{12} \sinh(\lambda_3 L_2) = 0 \]
\[C_5 + C_7 \lambda_2 \sinh(\lambda_2 L_2) + C_8 \lambda_2 \cosh(\lambda_2 L_2) - C_9 - C_{11} \lambda_2 \sinh(\lambda_3 L_2) - C_{12} \lambda_2 \cosh(\lambda_3 L_2) = 0 \]
\[C_9 (D_2 \lambda_2^3) + C_8 D_2 \lambda_2^2 \sinh(\lambda_2 L_2) - C_{11} D_3 \lambda_2^3 \cosh(\lambda_3 L_2) - C_{12} D_3 \lambda_2^2 \sinh(\lambda_3 L_2) = -P \Delta \]
\[C_7 D_2 \lambda_2^3 \sinh(\lambda_2 L_2) + C_8 D_2 \lambda_2^3 \cosh(\lambda_2 L_2) - C_{11} D_3 \lambda_2^3 \sinh(\lambda_3 L_2) - C_{12} D_3 \lambda_2^3 \cosh(\lambda_3 L_2) = 0 \]
\[C_9 (L_2 + L_3) + C_{10} + C_{11} \cosh(\lambda_3 (L_2 + L_3)) + C_{12} \sinh(\lambda_3 (L_2 + L_3)) = 0 \]
\[C_{11} \cosh(\lambda_3 (L_2 + L_3)) + C_{11} \sinh(\lambda_3 (L_2 + L_3)) = 0 \]

where $\Delta = h/2$ ($h$ being the thickness of adherends), and $L_1$, $L_2$ and $L_3$ are as shown in Fig. 1(d).
5. References

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Chapter 4

Fatigue Threshold Behavior of Adhesive Joints

1. Introduction

Cyclic loads can produce fatigue failure in adhesive joints at stress levels much lower than they can withstand under monotonic loading. Consequently, there has been extensive research on the fatigue crack growth of adhesive joints under mode I loading [1-4] and mixed-mode loading [5-7]. Investigators have also examined the effect of testing parameters such as frequency [4,8], load ratio (mean load) [4] and environmental conditions [2,3,9-11] on the fatigue performance of adhesive joints. A reliable approach for the design of joints that will experience cyclic loading is to keep service loads below those corresponding to the threshold energy release rate, $G_{th}$, so that little or no crack propagation occurs. However, if this design criterion is considered too conservative, allowable joint loads may also be based on a crack growth rate that is sufficiently small to ensure the joint will fulfill its service life. In general, a complete characterization of fatigue behavior covers both the threshold and higher crack growth rate regimes. The present work focuses on several factors affecting the fatigue testing of adhesive joints at very low crack growth rates in the vicinity of the threshold: (i) the adhesive layer starting condition (fillet or crack) and its role in initiation, (ii) differences due to the use of either load or displacement controlled testing, and (iii) the effects of crack speed on the crack path and failure mode.

Effect of fillet on $G_{th}$

The majority of the literature on fatigue threshold measurements pertains to crack growth from an existing fatigue crack (e.g. refs. [2,3]). This is also the approach recommended by ASTM E647 [12]; however, it is of practical interest to ascertain whether the threshold strain energy release rate, $G_{th}$, is different when the starting condition is an intact fillet. In this case, $G_{th}$ is interpreted as the value below which cracks will not initiate and extend from an undamaged fillet. In practice, this limiting value of the strain energy release rate is usually defined as that which causes crack initiation and growth that is less than some small distance in a fixed number
of cycles. Ashcroft and Shaw [3] suggested that a threshold defined as the maximum $G$ that causes no initiation in a million cycles could be different from one measured as the arrest of an established fatigue crack; however, no data was provided to support this hypothesis.

Dessureault and Spelt [6] measured the same number of cycles to initiation for aluminum CLS joints having an intact spew fillet, a mode I fast precrack and a fatigue precrack, and thus concluded that the starting condition had a negligible effect on fatigue crack initiation for that particular adhesive system. Johnson and Mall [13] found that the minimum cyclic load that initiated debonding after a million cycles in composite CLS joints made with two different adhesives resulted in a $G_{th}$ which was very close to that associated with a crack propagation rate of $10^{-6}$ mm/cycle. Lefebvre and Dillard [14] reported that fatigue crack initiation was more sensitive to adherend pretreatment than fatigue crack propagation for epoxy-aluminum wedge specimens.

The contribution of crack initiation to the total fatigue life of adhesive joints is an important part of the development of fatigue life models; however, there is uncertainty about the relative durations of initiation and propagation. Some authors have found that the crack initiation phase is much shorter than the crack propagation phase prior to the failure of single lap-shear (SLS) joints [15-17], and thus can be safely ignored. However, Imanaka et al. [18] and Harris and Fay [19] found the opposite; i.e. that the fatigue life of SLS joints was initiation dominated. Indeed, Crocombe et al. [20] and Crocombe and Richardson [21] found that, even at loads as high as 50% of the static failure load, crack initiation accounted for at least half of the total fatigue life of SLS joints. For CLS joints, the crack initiation phase has been reported to account for between 20% and 80% of the total fatigue life, depending on the loading level [21]. However, removing the adhesive fillet in SLS joints eliminated the initiation phase [20]. As might be expected, as the force level decreased, the contribution of crack initiation to the total fatigue life of the joint increased [18,21]. The initiation fraction of the fatigue life of a joint seems to be a function of the adhesive system, the joint configuration and the loading level. The present work examined crack initiation and propagation at load levels near the threshold strain energy release rate for different starting conditions.

**Effect of testing approach – force vs. displacement control**

In a force controlled test, the maximum and minimum applied loads are kept constant as the crack grows. In a double cantilever beam (DCB) or cracked lap-shear (CLS) specimen this causes the applied strain energy release rate, $G$, at the crack tip, and thus the crack speed, to
increase over time. As a result, in force controlled testing, the crack growth is typically initiated in the threshold region and progressively accelerated to the maximum desired crack speed. This is opposite to the procedure often used in a displacement controlled test where the applied $G$ and crack speed decreases as the crack grows in DCB and CLS specimens. In these cases, the test begins with the maximum desired crack growth rate and terminates as the crack reaches the threshold. Displacement controlled fatigue tests are usually faster and easier to control than load controlled tests.

Mall et al. [22] observed no effect of testing approach on debond growth rates as a function of $\Delta G$, the cyclic range of the strain energy release rate ($\Delta G = G_{\text{max}} - G_{\text{min}}$), for a composite DCB joint bonded using a thermosetting rubber-toughened epoxy adhesive. The same DCB joint was tested under both force and displacement control at load and displacement ratios of $R=0.1$ (ratio of minimum to maximum load or displacement), which resulted in the same crack growth rates. However, using the same adhesive system, Mall and Johnson [23] found a higher debonding rate as a function of $G_{\text{max}}$ at $R=0.1$ under displacement control compared to force control, and suggested that the process may have been influenced by $dG/da$, the rate of change of $G$ with the crack length, although this was not explained. Nevertheless, considering the scatter in the data, the difference between the measured crack growth rates from the two approaches was relatively small [23]. Mangalgiri et al. [24] tested the same adhesive system as in [22,23] at load and displacement ratios of 0.1 and found that the increased crack growth rate associated with displacement control became more significant as the composite adherend thickness was increased from 8-ply to 24-ply. At the same $\Delta G$, a higher crack growth rate was measured under displacement control compared to force control. The authors considered an explanation in terms of the plastic zone size at the crack tip, but found this to be inadequate to explain the observed trends [24].

The crack tip damage zone and crack face roughness can prevent complete closure of the crack in the unloading part of the cycle when the applied load goes to zero. This can result in residual tensile stress in the adhesive ahead of the propagating crack [25] that is evident when the minimum load is close to zero; i.e. when $R$ is sufficiently small [26]. Consequently, the correlation of fatigue crack growth rates with $\Delta G$, rather than $G_{\text{max}}$, may be a better approach since it can include the effect of crack closure on the actual $G_{\text{min}}$ in the cycle. For example, fatigue crack growth rates were found to be independent of the load ratio, $R$, when plotted against $\Delta G$ or $\Delta G_{\text{eff}}$, but a significant dependence on $R$ was observed when the graphs were presented versus $G_{\text{max}}$ [4,22]. In these cases, $\Delta G_{\text{eff}}$ was defined as the difference in the energy
release rate between the maximum force and the force at which crack closure occurred, $P_{cl}$, which is found by plotting force versus displacement in a cycle, and finding the load in which the curve deviated from linearity [12]. Differences between load and displacement controlled testing may be related to crack closure effects. For example, in [22] crack growth was plotted against $\Delta G$, and no effect of testing approach was found; however plotting the crack growth against $G_{\text{max}}$ [23] resulted in a higher observed crack growth rate for the displacement controlled testing.

In a displacement controlled test, the force ratio achieved throughout the test under a displacement ratio of $R$ will not necessarily remain the same as in a force controlled test at a load ratio of $R$. As will be discussed in Section 3.2, this can happen if the specimen compliance changes during a fatigue cycle. The question of whether displacement control produces fatigue results that are equivalent to those under load control was examined in the experiments described below.

_Sensitivity of fatigue crack growth to interfacial bond strength_

The relationship between fatigue crack growth and interfacial bond strength has not been studied explicitly. The related topic of fatigue crack growth as a function of bond durability has been examined in a liquid water environment as an accelerated test of the relative durability and aging of adhesive systems [2,10,27]. By comparing the fatigue threshold and the locus of failure in wet tests with similar experiments performed in a dry environment, durable systems were identified. Experiments performed at crack speeds as low as the fatigue threshold ($10^{-7}$ mm/cycle) and as high as $10^{-2}$ mm/cycle indicated that the more durable systems had lower crack growth rates for a given load. Similar studies have also been performed at elevated temperatures and relative humidities to assess the durability of adhesive joints prepared using different surface treatments [28-30].

This chapter presents the results of fatigue experiments performed on a highly-toughened epoxy adhesive bonded to aluminum adherends at the fatigue threshold and at higher crack growth rates under dry conditions. The results were examined as a function of: (i) adhesive starting geometry (fillet or fatigue precrack), (ii) testing approach (load and displacement control), and (iii) interfacial bond strength. The fatigue data were compared with fracture measurements on identical specimens.
2. Experimental Approach

2.1 Effect of Fillet on $G_{th}$

Specimen preparation

Experiments were performed on cracked lap shear (CLS) joints manufactured from 12.7×19.05 mm (1/2"×3/4") AA6061-T651 flat bars (Fig. 1(a)). The aluminum bars were abraded using two types of silicon-carbide nylon-mesh abrasive pads which gave an average roughness of $R_a=0.77±0.02\ \mu m$ or $R_a=1.33±0.16\ \mu m$ ($±$ gives the standard deviation over four measurements on two bars). The aluminum bars were then pretreated using the P2 etching process [31]. In order to prevent the failure in the aluminum bars and at the loading pins, three ¼”-20 bolts were used to reinforce the specimen near the loading pins. The three vertical holes in Fig. 1(a) are for this purpose.

An adhesive bondline thickness of 0.4 mm was established using spacing wires in the bondline. The cure cycle was monitored using an embedded thermocouple to ensure that the adhesive remained at 180°C for 30 min as recommended by the manufacturer. Excess adhesive that spewed from the sides of the specimens during curing was removed by sanding with a succession of abrasive papers (60, 120 and 600 grit) using water as a coolant, before applying a thin coating of diluted white correction fluid to provide a high-contrast image of bondline cracking. Some CLS specimens were prepared with a precrack made by placing a 20 $\mu m$ folded aluminum foil in the bondline. To form the fillet in the other CLS joints, after assembling the joint and before curing it, the specimen was left in the room environment for about 30 min to allow any excess adhesive to flow out of the joint. The excess adhesive was then removed from the edge of the overlap end using a spatula before curing. Examples of the resulting fillets are shown in Fig. 2.

Fatigue testing

The threshold for an existing fatigue crack in the bondline was measured under force control by decreasing the applied load in steps of less than 10% of the force magnitude (keeping $R$ constant) until the fatigue threshold was established at a crack speed of $10^{-6}$ mm/cycle [12]. Prior to the start of the force drop, the crack had propagated 2-3 mm from the foil precrack, thereby ensuring that $G_{th}$ corresponded to a fully developed fatigue crack tip damage zone.

For the intact adhesive spew fillets, $G_{th}$ was defined as the energy release rate at which a crack would form in the fillet and propagate less than 1 mm after one million cycles. If no crack
initiation was observed after one million cycles, the force was increased by 10% and cycling continued for another million cycles. In the present experiments, crack initiation and growth to less than 1 mm in the fillet occurred from between 2 and 7 million cycles; i.e. in some cases, the load needed to be increased 6 times to finally observe initiation and growth of less than 1 mm in 1 million cycles.

All fatigue experiments in this paper were carried out at a sinusoidal cyclic frequency of 20 Hz, and under a dry condition (11% - 15% relative humidity), which was achieved by performing the experiments in a desiccant chamber.

---

Fig. 1  Geometry of (a) CLS and (b) ADCB and (c) DCB joints. All dimensions in mm, unless stated. CLS joints made with intact fillet or with folded foil as a crack starter.
Measurement of crack initiation and data reduction

Crack initiation and damage development in adhesive joints has been detected and defined in various ways: optically [11,19], via changes in joint stiffness [17,19], and using strain gauges [14,18,20,32,33]. However, changes in joint stiffness and strain fields are usually very small for the minute crack lengths that might typically define initiation, and the success of the strain measurement technique is highly dependent on the location and the size of the strain gauges [20]. Therefore, in the present experiments crack lengths were measured using a CCD camera with a 2 mm field of view mounted on a motorized linear stage. The resolution of this system was sufficient to permit fatigue initiation to be defined accurately as the creation of a 50 µm long crack.

Fig. 2 Crack initiation and early stage propagation in three aluminum CLS joints with intact fillet at the overlap end. Bondline thickness was 0.4 mm.
The strain energy release rate, $G$, and phase angle, $\psi = \arctan \left( \sqrt{\frac{G_{II}}{G_{I}}} \right)$, for the CLS joints were calculated following the analytical approach of [34] in which an “adhesive sandwich” was isolated at the crack tip region and the J-integral was calculated around this sandwich. The tensile and bending reactions acting at the ends of the sandwich element are given by a system of twelve equations described in Chapter 3 and ref. [35] for the current CLS geometry.

### 2.2 Effect of Testing Approach – Force vs. Displacement Control

**Specimen preparation**

Asymmetric double cantilever beam (ADCB) joints with an adherend thickness ratio of 2 were made from 12.7×19.05 mm (1/2”×3/4”) AA6061-T651 flat bars, roughened to $R_a=1.33$ µm (standard deviation of 0.16 µm over four measurements on two bars), using a silicon-carbide nylon-mesh abrasive pad (Fig. 1(b)). A 40 mm long cohesive precrack was formed by embedding a 20 µm folded aluminum foil in the bondline.

**Fatigue testing**

In the force controlled experiments, the threshold strain energy release rate was identified first, followed by the measurement of the crack speed, $da/dN$, versus the applied strain energy release rate, $G$. The test procedure was identical to that described for the specimens that had an existing crack in Section 2.1 [12]. After measuring $G_{th}$, the applied $G$ was increased by 10% and the corresponding load was kept constant throughout the remainder of the test, causing a gradual increase in $G$ and the crack speed as the crack grew.

The displacement controlled fatigue tests began with the highest $G$ of interest and the crack slowed to the threshold value ($10^{-6}$ mm/cycle) as the displacement was held constant and the joint compliance increased with crack length. Figure 3 shows the change in the force during typical force and displacement controlled tests on an ADCB joint.

**Crack length measurement and data reduction**

The unloading joint compliance approach [36] was used to measure the fatigue crack length during these experiments. The compliance was measured during the unloading part of the cycle using the load cell output and a clip gauge attached to the end of the specimen (Fig. 1). The CCD camera described above was used to measure the crack length and relate it to the measured joint compliance for a given specimen type using the approach of ref. [12]. A least
squares regression was used to fit a third-order polynomial to the normalized crack length, $a/w$, versus the normalized compliance, $CEB$, for fatigue joints:

$$a/w = c_1 \times (CEB)^3 + c_2 \times (CEB)^2 + c_3 \times (CEB) + c_4$$

where $a$ is the crack length, $w$ is the specimen length from the loading pins, $C$ is the compliance, $E$ is the tensile modulus of the adherends, and $B$ is the specimen width.

Fig. 3 Change in the applied maximum force in the cycle, $P_{\text{max}}$, during fatigue threshold testing of aluminum ADCB under (a) force control and (b) displacement control.
The strain energy release rate for the ADCB joint was calculated from the measured force and crack length using an analytical beam-on-elastic-foundation model, described in Chapter 3. The model predicted $G$ values for aluminum and steel DCB and ADCB joints that were within 2% of those from a two-dimensional elasto-plastic finite element model for crack lengths of 40-120 mm.

2.3 Sensitivity of Fatigue Crack Growth to Interfacial Bond Strength

Specimen preparation

Specimens of varying interfacial bond strength were produced by varying the surface roughness, adhesive batch and adherend pretreatment. The aluminum CLS joints described in Section 2.1 with $R_a=0.77 \ \mu m$ and $R_a=1.33 \ \mu m$, and aluminum ADCB joints with $R_a=1.33 \ \mu m$ and $R_a=1.87 \ \mu m$ were found to have different interfacial bond strengths depending on their roughness. Significant variations in the interfacial bond strength were also observed to result from the batch-to-batch variability in the adhesive used to make the aluminum ADCB joints described in Section 2.2. Finally, two different pretreatments were used with AISI 1018 steel bars to make DCB, ADCB and CLS joints (Fig. 1) of differing interfacial bond strength. In the first steel pretreatment, the bars were abraded using an aluminum oxide abrasive pad producing an $R_a=1.44 \ \mu m$ (standard deviation of 0.15 $\mu m$ over five measurements on two bars). The bars were then wiped using cheesecloth and acetone, degreased for 5 min in acetone, and then rinsed with ethanol. In the second pretreatment, the steel bars were abraded as before, and were then given a Zn-phosphate pretreatment as follows: 1. Pacocleaner® 319 (Henkel Corp.) immersion for 2 min at 57°C. 2. Rinse with tap water for 30 s. 3. Fixodine® ZL (Henkel Corp.) immersion for 30 s. 4. Bonderite® 958 (Henkel Corp.) immersion for 2 min at 49°C. 5. Rinse with tap water for 30 s. 6. Parcolene® 99x (Henkel Corp.) immersion for 30 s. 7. Rinse with tap water for 30 s. 8. Dry in oven for 10 min at 121°C.

Crack length measurement and data reduction

The procedures used with CLS joints were explained in Section 2.1, while those of Section 2.2 were used for the DCB and ADCB joints.

Fatigue and fracture testing

Fatigue experiments were carried out under force control at a load ratio of 0.1 as per the procedures explained in Sections 2.1 and 2.2.
The fracture tests were performed on aluminum ADCB joints. As with the fatigue experiments, the same load was applied on both arms of the ADCB to produce a mixed-mode loading condition \( (\psi = 18^\circ) \) at the crack tip. ADCB specimens were loaded with a constant cross-head speed of 1.0 mm/min. The crack length was measured from the center of the loading pins on the ADCB specimens using a microscope mounted on a micrometer stage with an accuracy of 0.01 mm. Crack growth was stable in this system so that many crack extension events could be recorded with a single ADCB fracture specimen. To measure the critical load at each crack length, the cross-head displacement was started and stopped repeatedly in the vicinity of the expected fracture load (each time at a constant crosshead speed of 1.0 mm/min) until a drop in the applied load was observed. This maximum load prior to the drop was taken as the critical fracture load for the measured crack length if visual inspection through the microscope confirmed that the macro-crack had propagated. After measuring the new macro-crack length, the ADCB was unloaded and the same procedure was followed again beginning at the new crack length. These procedures were similar to those of Chapter 2 and ref. [37].

In order to define the crack path accurately, the residual adhesive thickness that remained on the more highly-strained aluminum adherend was measured on some of the tested specimens. At intervals along the length of the adherend, the residual adhesive was removed from a small region about 2 mm wide on opposing sides of the joint using a solvent (mixture of methylene chloride and methyl alcohol; Glue Buster, Kosmic Surf-Pro Inc., Saint Amable, Quebec) to provide an elevation datum corresponding to the bare aluminum. An optical profilometer (Nanovea ST 400, Microphotonics Inc., Irvine, CA, USA) was then used to make a line scan across the width of the joint from one datum region to the other, thereby giving the average thickness of the residual adhesive.

3. Results and Discussion

3.1 Effect of Fillet on \( G_{th} \)

It was observed that fatigue cracks initiated at various locations within the fillets of the aluminum CLS joints and propagated at a 45° angle toward the strap adherend (the more highly strained member in the joint) where they then grew close to the interface (Fig. 2). In the joints containing a foil precrack, the crack initiated from the precrack tip and again grew toward the strap adherend at approximately 45° before propagating along the interface (Fig. 4). In these cases, \( G_{th} \) was measured after 2-3 mm of growth from the end of the foil.
Table 1 compares $G_{th}$ from the spew fillet and the foil precrack for the two aluminum adherend surface roughnesses, where $G_{th}$ was the maximum strain energy release rate applied in the cycle. As mentioned previously, literature values of the fatigue threshold have also been based on the maximum force that results in no crack initiation in one million cycles; therefore, this value is provided in the first row of Table 1, while the second row shows the very similar values of $G_{th}$ as defined in Section 2.1. For $R_a=1.33$ µm, $G_{th}$ was statistically indistinguishable (t-test, 95% confidence) for the fillet and the fatigue crack (grown from foil precrack), but with $R_a=0.77$ µm $G_{th}$ was smaller for fatigue cracks (t-test, 95% confidence). With the smoother adherends ($R_a=0.77$ µm), cracks grew interfacially and relatively quickly along the strap adherend leaving very little residual adhesive (Fig. 5(a)). Therefore, $G_{th}$ for these fatigue precracked specimens was smaller since it was related to a mostly interfacial failure along the relatively smooth adherend. However, crack initiation from a fillet was higher since it was controlled mostly by the length of the cohesive crack growth prior to reaching the interface. Increasing the surface roughness made the failure more cohesive adjacent to the strap adherend (Fig. 5(b)), thereby making $G_{th}$ the same for both starting conditions with the specimens with $R_a=1.33$ µm. As expected, the surface roughness had no effect on the measured $G_{th}$ from the fillet (Table 1) since the initiation and early stage crack growth was cohesive within the adhesive fillet.
Table 1 Comparison of $G_{th}$ for aluminum CLS joints of two roughnesses from an intact fillet and from a fatigue crack grown from the foil precrack under force control. $G_{th}$ for intact fillets according to two definitions: the maximum load prior to initiation, and the minimum load that produced initiation and crack growth of 1 mm in 1 million cycles. $N$ is the number of experiments, ± the standard deviation.

<table>
<thead>
<tr>
<th>Condition</th>
<th>$R_a=0.77 \mu m$</th>
<th>$R_a=1.33 \mu m$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$G_{th}$ (J/m²)</td>
<td>$N$</td>
</tr>
<tr>
<td>Intact Fillet - greatest load without initiation</td>
<td>250 ± 32</td>
<td>3</td>
</tr>
<tr>
<td>Intact Fillet – smallest load producing initiation and propagation</td>
<td>270 ± 28</td>
<td>3</td>
</tr>
<tr>
<td>Fatigue precrack (ASTM)</td>
<td>168 ± 47</td>
<td>3</td>
</tr>
</tbody>
</table>
Fig. 5  Fatigue failure surfaces of CLS joints at the threshold with (a) $R_a=0.77 \, \mu m$ and (b) $R_a=1.33 \, \mu m$, and (c) fracture failure surfaces of two aluminum SLS joints. Fatigue experiments were done under force control.

As explained previously, the procedure to measure $G_{th}$ involved increasing the load by steps of 10% if no initiation was observed in 1 million cycles. To investigate whether this history of prior cyclic loading affected the measured $G_{th}$, one of the specimens from the rougher system was cycled with a $G_{max}$ of 246 J/m$^2$, close to the average of previously measured $G_{th}$
values from the fillet. It was observed that the crack initiated after 540,000 cycles, and that the
 crack growth was less than 1 mm in 1 million cycles, suggesting that pre-cycling had no effect
 on the measured $G_{th}$ from the fillet. This cyclically formed crack was then used as a precrack for
 which $G_{th}$ was found to be 248 J/m$^2$, showing again that there was no significant difference
 between $G_{th}$ from a fillet and a fatigue precrack, provided that the crack path was cohesive and
 not interfacial. As mentioned before, this is consistent with Johnson and Mall [13] who also
 observed no difference between the minimum cyclic load that initiated debonding after 1 million
 cycles and the load corresponding to a crack propagation rate of $10^{-6}$ mm/cycle from a crack in a
 composite CLS joint.

The good repeatability of $G_{th}$ from the spew fillet (Table 1) shows the insensitivity of
 fatigue crack initiation to the detailed shape of the fillet. This is consistent with the observations
 of [6], made with a much weaker epoxy adhesive, that fatigue crack initiation in a CLS joint was
 independent of the starting conditions. It is also consistent with the results of [38] where, for the
 same adhesive system as was used in the present experiments, crack initiation under mixed-mode
 quasi-static fracture was found to be only weakly dependent on the starting geometry.

**Crack Initiation**

The number of cycles to initiation from an intact fillet at the applied $G_{th}$ for the three CLS
 specimens tested at each of the two roughnesses is given in Table 2. Consistent with the results
 of Table 1, there was no statistically significant difference between the results at $R_a=0.77$ µm and
 $R_a=1.33$ µm ($t$-test, 95% confidence level), implying again that the surface roughness had no
 effect on the fatigue crack initiation behavior from a fillet.

<table>
<thead>
<tr>
<th>$R_a$ (µm)</th>
<th>Specimen</th>
<th>Average $N_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>#1</td>
<td>#2</td>
</tr>
<tr>
<td>0.77</td>
<td>320,400</td>
<td>460,400</td>
</tr>
<tr>
<td>1.33</td>
<td>536,600</td>
<td>520,300</td>
</tr>
</tbody>
</table>

As was mentioned previously, it has been a common assumption in life prediction
 modeling [15,16,39] that the number of cycles leading to crack initiation can be neglected
 relative to those required for crack propagation in practical joints. If it is assumed that a constant
crack growth rate of $10^{-6}$ mm/cycle was sustained in a CLS joint after initiation at $G_{th}$ until the joint effectively failed after 20 mm of growth (under force control, the increase in $G$ with crack length growth is relatively small), the ratio between the number of cycles to initiation, $N_i$, and the number of cycles to failure, $N_f$, would be approximately 2%. This small $N_i/N_f$ ratio suggests that, for the purpose of predicting the number of cycles to failure at small crack speeds and close to the fatigue threshold region, it is acceptable to neglect the initiation phase for the presently considered highly toughened epoxy adhesive system. It is expected that this would also be true of single lap shear (SLS) joints since the phase angle is approximately the same as in CLS joints, and both the phase angle and $G$ would change a relatively small amount over 20 mm of crack length [37]. The focus of the current work was the fatigue threshold region, and thus relatively small loads were used. The fraction of the total fatigue life attributable to initiation at higher crack growth rates with the current highly-toughened epoxy adhesive cannot be predicted and would need to be examined separately.

3.2. Effect of Testing Approach - Force Control vs. Displacement Control

In the force controlled experiments the load ratio, $R_p=P_{\text{min}}/P_{\text{max}}$, was kept constant, while the displacement controlled experiments were carried out at a constant displacement ratio, $R_\delta=\delta_{\text{min}}/\delta_{\text{max}}$. Since force, $P$, and displacement, $\delta$, are related through the compliance, $C=\delta/P$, the relation between the load ratio, $R_p$, and displacement ratio, $R_\delta$, will be:

$$R_p = \frac{C_{\text{max}}}{C_{\text{min}}} \times R_\delta$$

(2)

where, $C_{\text{min}}$ and $C_{\text{max}}$ are the compliance at the maximum and the minimum force in the cycle, respectively. Therefore, anything that causes $C_{\text{min}}$ and $C_{\text{max}}$ to change and become different during a displacement controlled cycle will affect the resulting load ratio. Possible factors that may do this are:

**Geometric change due to fatigue crack growth**

In principle, the amount of crack growth occurring in a single cycle will change the compliance; however this effect will be very small at the crack growth rates used in the present study. For example, at $10^{-2}$ mm/cycle with a crack length of 40 mm, the ratio of $C_{\text{max}}/C_{\text{min}}$ will change by only 0.03% in one cycle.
Plasticity effects on joint compliance

Plastic deformation at the crack tip in the adhesive layer will cause the joint compliance to be dependent on the applied force and thus \( C_{\text{min}} \) and \( C_{\text{max}} \) may be different.

The magnitude of the effect was estimated using a two-dimensional elasto-plastic finite element analysis (ANSYS® 11, Ansys Inc., Canonsburg, PA). The structural model used plane182 elements with plane stress assumptions for the substrate and plane strain for the adhesive. The adhesive was modeled with a bilinear stress-strain curve derived from tensile tests. The properties of adherend and adhesive are given in Table 3. At a constant crack length of 80 mm, the force on a 20 mm wide aluminum DCB joint was increased from 50 N to 1,000 N (resulting in \( G_{\text{min}} \) and \( G_{\text{max}} \) of 4 J/m\(^2\) and 1,670 J/m\(^2\)). It was seen that the calculated joint compliance was independent of the applied load up to \( G \approx 100 \text{ J/m}^2 \) and then increased linearly with the applied \( G \). However, the calculated joint compliance at \( G=1,670 \text{ J/m}^2 \) was only about 1\% higher than that at \( G=100 \text{ J/m}^2 \).

Table 3. Mechanical properties of the adherend and the adhesive used in the FE model. Yield stress, \( \sigma_y \), tensile elastic modulus, \( E \), and tensile tangent modulus, \( E_t \).

<table>
<thead>
<tr>
<th>Material</th>
<th>( \sigma_y ) (MPa)</th>
<th>( E ) (GPa)</th>
<th>( E_t ) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum, AA6061-T6</td>
<td>270</td>
<td>68.9</td>
<td>0</td>
</tr>
<tr>
<td>Adhesive</td>
<td>37</td>
<td>1.96</td>
<td>0.140</td>
</tr>
</tbody>
</table>

Roughness and plasticity induced crack closure

Roughness and plasticity induced crack closure causes the apparent crack length at \( \delta_{\text{min}} \) to be smaller than the fully opened crack length, and thus \( C_{\text{min}} \) becomes smaller than \( C_{\text{max}} \) and \( R_P \) increases with respect to the applied \( R_\delta \) (Eq. (2)). For example, at a crack length of 60 mm, as a result of 1 mm, 2 mm and 3 mm crack closure, a 5\%, 9\% and 15\% increase in the load ratio will be achieved. Roughness induced crack closure becomes more important as the load or displacement ratio decreases [26]. As a result of permanent tensile plastic deformation left in the wake of a fatigue crack, plasticity induced crack closure can also occur [25]. Therefore, plasticity induced crack closure becomes important at higher values of \( G_{\text{max}} \).

To compare the two testing approaches, ratios of 0.1 and 0.5 were chosen for the present experiments. For the six experiments performed at a displacement ratio \( R_\delta =0.1 \), the force ratio, \( R_P \), varied from about 0 to 0.10, and for the three experiments conducted at \( R_\delta =0.5 \), \( R_P \) varied
from 0.42 to 0.5. Table 4 gives $\Delta G_{th}$ for the two test methods along with the Paris law crack growth parameters (discussed below). As mentioned in Section 2.2, all specimens had $R_a=1.33$ $\mu$m. At $R_\delta$ and $R_P$ of 0.1 ($t$-test, 95% confidence), $\Delta G_{th}$ was indistinguishable among the two test approaches, but there was a small statistically significant difference of 10% at $R_\delta$ and $R_P$ of 0.5. This small effect is believed to be an artifact of the experimental scatter and would probably disappear with a larger sample size. This view is supported by the crack growth rates (discussed below) which were independent of the testing approach at both values of $R$.

Table 4 $\Delta G_{th}$ for aluminum ADCB joints measured using force control and displacement control at load and displacement ratios, $R$, of 0.1 and 0.5. $m$ and $b$ are as per Eq. (3) and correspond to the data of Fig. 6, $N$ is the number of fatigue threshold experiments. ± the standard deviation. $R_a=1.33$ $\mu$m.

<table>
<thead>
<tr>
<th>Test</th>
<th>$R=0.1$</th>
<th>$R=0.5$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\Delta G_{th}$</td>
<td>$m$</td>
</tr>
<tr>
<td>Force Control</td>
<td>125±6 3.6±0.4</td>
<td>-13.4±1.0</td>
</tr>
<tr>
<td>Displacement Control</td>
<td>121±12 3.4±0.8</td>
<td>-12.9±1.7</td>
</tr>
</tbody>
</table>

The crack growth rates for two representative specimens using each testing approach are shown in Fig. 6. Overall, there was no clear difference between the force and displacement control approaches. This was confirmed using a linear regression fit to the data points above the threshold region:

$$\log (da/dN) = m \times \log (\Delta G) + b \quad (3)$$

The differences in the values of $m$ and $b$ for the various combinations of test method and load/displacement ratio (Table 4) were statistically insignificant at the 95% confidence level. Therefore, it is concluded that the fatigue behavior of this highly-toughened epoxy adhesive was unaffected by the testing approach. This supports the use of displacement controlled fatigue testing, which is faster and more convenient than force controlled testing.
Fig. 6 Measured crack growth rate behavior of aluminum ADCB using force control and displacement control at load and displacement ratios of (a) 0.1 and (b) 0.5. Two representative experiments are shown for each case. $R_s=1.33 \mu m$.

3.2.1 Effect of $dG/da$

As mentioned in the Introduction, Mall and Johnson [23] suggested that adhesive fatigue
crack growth rates can be different under force and displacement control because of changes in 
\(dG/da\), the \(G\) gradient. This can be examined using \([40]\):

\[
G = \frac{P^2}{2b} \frac{dC}{da}
\]  
(4)

where \(P\) is the applied force, \(b\) is the width of the specimens, \(C\) is the joint compliance and \(a\) is the crack length. With \(C = \delta/P\), where \(\delta\) is the load-line displacement, it is seen that for:

**Force control:**

\[
\frac{dG}{da} = \frac{P^2}{2b} \frac{d^2 C}{da^2}
\]  
(5)

**Displacement control:**

\[
\frac{dG}{da} = \frac{\delta^2}{2b} C^2 \left[ \left( -\frac{2}{C} \right) \left( \frac{dC}{da} \right)^2 + \frac{d^2 C}{da^2} \right]
\]  
(6)

To find the compliance of DCB and ADCB joints, Krenk’s beam-on-elastic-foundation model \([41]\) was modified as follows:

\[
C = \frac{4 \beta_l}{k} \left( 1 + 2 \beta_l a + 2 \beta_l^2 a^2 + \frac{2}{3} \beta_l^3 a^3 \right) + \frac{4 \beta_u}{k} \left( 1 + 2 \beta_u a + 2 \beta_u^2 a^2 + \frac{2}{3} \beta_u^3 a^3 \right)
\]  
(7)

where,

\[
E_a' = \frac{E_a}{1 - \nu_a^2}
\]  
(8)

\[
k = \frac{E_a' b}{2t}
\]  
(9)

\[
\beta^4 = \frac{k}{4EI}
\]  
(10)

Here \(E\) and \(E_a\) are the tensile moduli of the adherend and adhesive, respectively, \(\nu_a\) and \(t\) are the Poisson ratio and thickness of the adhesive, and \(I\) is the moment of inertia of the adherends defined by subscripts \(l\) and \(u\) referring to the upper and lower adherends, respectively. The compliance for an aluminum ADCB joint calculated from Eq. (7) agreed well with the experimentally measured compliance and an elasto-plastic two-dimensional finite element model as shown in Fig. 7.

The relation between the crack speed and the energy release rate was taken from the experimentally measured coefficients for Eq. (3) as given in Table 4 for the ADCB joints and
Fig. 16 for DCB joints (Section 3.3). The corresponding $dG_{\text{max}}/da$ versus crack speed for DCB and ADCB specimens tested under force and displacement control are compared in Fig. 8. The crack propagation started from a 40 mm precrack with a constant maximum force per unit width of 31.5 (DCB) and 33.5 (ADCB) kN/m under force control, and constant maximum displacements of 4.2 mm (DCB) and 4.8 mm (ADCB) under the displacement control. These force and displacement values were chosen so that at the precrack they would result in $da/dN=10^{-6}$ mm/cycle (the lowest desired crack speed) for force control, and $da/dN=10^{-2}$ mm/cycle (the highest desired crack speed) for displacement control. It is seen (Fig. 8) that $dG_{\text{max}}/da$ (or $dG/da$ at other points in the loading cycle) at crack speeds above the threshold region is significantly higher under displacement control than in load control and that the difference becomes very small at the threshold ($10^{-6}$ mm/cycle). However, no statistically significant difference between the fatigue crack growth rates for the two test methods was measured (Table 4 and Fig. 6), indicating that $dG/da$ did not influence $da/dN$ in the present experiments, contrary to the suggestion of [23].

![Graph](image_url)

Fig. 7 Comparison of the compliance of aluminum ADCB joints measured experimentally from three specimens and calculated using Eq. (7) and the FEA. The vertical axis is the product of $C$ and $B$, the compliance and the width of the joint, respectively.
Fig. 8 Analytical calculations of $dG_{\text{max}}/da$ versus crack speed under force and displacement control testing of aluminum (a) DCB and (b) ADCB specimens.
It is noted that the higher $dG/da$ for a displacement controlled test, implies that to achieve the same range of crack speeds a shorter period of testing and a significantly shorter crack extension is required than under force control. As an example, to measure the crack growth rate between $10^{-6}$ to $10^{-2}$ mm/cycle, from an initial crack length of 40 mm, a 50 mm crack extension would be needed under displacement control, compared with 120 mm under force control.

### 3.2.2 Effect of Adhesive Stiffness

As was mentioned in the Introduction, Mangalgiri et al. [24] observed no significant difference between force and displacement controlled testing when testing composite DCBs with 8-ply adherends, but a difference between the two testing approaches became apparent when the adherend thickness was increased to 24-plies. It is noted that the presence of the adhesive layer was ignored in the calculation of $G$ for the DCB joints of [24] and it is hypothesized that this may be the cause of the reported difference between the force and displacement controlled data. Based on the fitted line to the crack growth rate graphs of DCBs with 24-ply adherends in [24], it seems that the difference became more noticeable as the crack speed increased. Figure 9(a) shows the measured fatigue curves for two of the present DCB joints with 12.7 mm adherends, one tested under force and the other displacement control, with $G$ calculated using the simple beam theory [35] which ignores the presence of the adhesive. The difference in the graphs was not noticeable. Graphing the crack growth rates when $G$ was calculated using the beam on-elastic-foundation (BOEF) model also showed no difference between the testing approaches in this case. The effect of the relative stiffness of the adherends and adhesive in the $G$ calculation was then investigated by increasing the adherend thickness three times and assuming that the adherend thickness would have no effect on the crack growth rate behavior. The data of Fig. 9(a) were thus converted to this hypothetical joint by adjusting the joint loads to give the same $G$, as per the BOEF model. This synthetic data set of load versus crack length was then used to produce, the crack growth rate graphs for the hypothetical thick specimens when the adhesive is neglected in the $G$ calculation as was done in [24]. The result is shown in Fig. 9(b) where a difference between displacement and force control becomes increasingly evident as the crack speed increases. Therefore, neglecting the adhesive compliance has resulted in an apparent dependence on the test approach which is roughly of the same size as seen in [24]. As the adherend thickness increases and the adherends become stiffer, the contribution of the adhesive layer to the total joint compliance increases. Conversely, as the crack length grows the joint becomes more flexible and the effect of neglecting the adhesive layer decreases. The high crack
growth rate points in a displacement controlled test are measured at the start of the test, while those points are collected towards the end of a force controlled test. This is the reason why the two curves of Fig. 9(b) diverge at high crack growth rates.

Fig. 9 Effect of neglecting the presence of the adhesive layer in the $G$ calculation for force and displacement control with DCB adherend thicknesses of (a) 12.7 mm and (b) a stiffer hypothetical joint with 38.1 mm thick adherends.
It is concluded that $dG/da$, should not have a significant effect on fatigue crack growth rates. Furthermore, it is suspected that the reported difference in the fatigue results measured from force and displacement controlled testing in ref. [24] come mainly from ignoring the presence of the adhesive in the calculation of $G$.

### 3.3. Sensitivity of Fatigue Crack Growth to Interfacial Bond Strength

#### 3.3.1 Bond Strength Influenced by Surface Roughness

The quasi-static fracture tests on two aluminum ADCB specimens with surface roughness of $R_a=1.33$ µm resulted in an average steady-state critical strain energy release rate $G_c^s=3,360\pm153$ J/m² (± standard deviation, 53 data points). Tests with a third aluminum ADCB specimen, this one with $R_a=1.87$ µm, gave $G_c^s=3,275\pm104$ J/m² (29 data points), showing no statistically significant difference due to roughness (t-test, 95% confidence). This was expected since the crack paths were fully cohesive for all of these specimens. However, under cyclic loading, increasing the roughness caused an increase in $G_{th}$ from $121\pm12$ J/m² (6 experiments, $R_a=1.33$ µm) to $174\pm12$ J/m² (2 experiments, $R_a=1.87$ µm).

The reason for the greater sensitivity of $G_{th}$ to surface roughness compared to the steady-state critical strain energy release rate, $G_c^s$, was the difference in the crack paths. It was observed that the crack path consistently moved closer to the interface of the more highly strained adherend (becoming less cohesive) as the fatigue crack growth rate decreased, as shown in Fig. 10. This is consistent with the observations reported for a similar adhesive system in Chapter 3 and ref. [35]. However, even for the highest tested $G$ under cyclic loading, which was about 1/4 to 1/3 of $G_c^s$, the crack path did not become as cohesive as that observed in the fracture test (Fig. 10). As an example, a quasi-static fracture test was performed after fatigue testing on two aluminum ADCB specimens (adherend thickness ratio of 4:1) from adhesive batch #1. The average residual adhesive thickness at the threshold was 10 µm, while it was 80 µm where the fatigue crack growth rate had been $10^{-3}$ mm/cycle. On the quasi-static fracture surface, the remaining adhesive was 120 µm thick.

#### 3.3.2 Bond Strength Influenced by Batch-to-Batch Variability

As mentioned previously, the present experiments were performed using five different adhesive batches having the same nominal formulation, but manufactured at different times and in different manufacturing facilities. This was found to produce significant differences in the
interfacial bond strength that were only apparent in fatigue testing at very low crack growth rates under mixed-mode loading.

Fig. 10 Failure surface of an aluminum ADCB specimen tested in both fatigue (displacement control) and quasi-static loading. Specimen is from batch #5. $R_a=1.33$ µm.

**Fatigue tests**

Figure 11 shows that there was a significant difference between the average fatigue threshold of the first batch and the other four batches using aluminum ADCB joints. However, Fig. 12 shows that the fatigue crack growth rate behavior was similar for the different adhesive batches at higher crack speeds, and that batch #1 deviated from the others mainly at lower crack speeds. These observations are consistent with those made above in the context of surface roughness; i.e. that the crack path tended to move closer to the interface of the more highly-strained adherend as the crack speed decreased. Thus slow, mixed-mode fatigue crack growth tends to highlight differences in interfacial bond strength. Both mixed-mode loading and the low crack speed cause the crack to move very close to the interface.

Figure 13 shows that batch #1, which had the greatest $G_{th}$, produced a fatigue crack path that was more cohesive, although none of the batches produced completely interfacial failure – there was always a thin layer of residual adhesive on the thinner ADCB adherend. This had been verified in Chapter 3 using x-ray photoelectron spectroscopy in earlier fatigue threshold testing with the same adhesive system at $R_a=1.3$ µm. In all specimens, the crack path became more cohesive as the crack speed increased. This illustrates why the effect of interfacial bond strength was most pronounced near the threshold of the ADCB joints, where cracks grew very close to the interface.
Fig. 11  $G_{th}$ of aluminum ADCB specimens bonded using different batches of the adhesive, $R_s=1.33$ µm, tested under force control. Six specimens were tested with the first batch, three with batch #5, and one for each of the other three batches. Error bars represent ±1 standard deviation.

Fig. 12  Comparison of the fatigue crack growth behavior of ADCB joints from different adhesive batches. Experiments were carried out under force control at $R_s=1.33$ µm. Results for
one representative experiment are shown for each adhesive batch.

Fracture tests

Fracture tests using ADCB specimens with these same five adhesive batches produced a typical R-curve as shown in Fig. 14. After crack initiation at $G_c^i$, the first several crack growth sequences occurred at an increasing critical strain energy release rate, $G_c$, as the damage zone at
the crack tip developed to its steady-state form [34,42]. One specimen was tested from each of adhesive batches 1-4 and two were tested from the fifth batch. This resulted in 38, 26, 36, 33 and 53 data points on the R-curve plateau for the first to the fifth batches, respectively. The crack paths for all ADCB joints tested under quasi-static loading were fully cohesive. When comparing $G_c^s$ for the different batches (Fig. 15), it is observed that the amount of scatter was comparable to the specimen-to-specimen experimental scatter previously seen for a similar adhesive in Chapter 2 and does not indicate anything unusual about the quasi-static fracture properties of batch #1, even though this batch behaved differently from the others in fatigue (Figs. 11 and 12). This suggests that quasi-static ultimate strength tests of lap shear joints, for example, would not detect the batch-to-batch differences in the adhesive that were evident in $G_{th}$. These differences were only evident in fatigue tests at low crack growth rates under mixed-mode loading.

![Fig. 14 Typical R-curve behavior of an aluminum ADCB joint (batch #1). $R_a=1.33 \, \mu m$.](image)

To further evaluate the effect of crack path on $G_{th}$, three aluminum DCB joints from both batches #1 and #5 were tested with a mode-I fatigue loading so that the crack propagated in the mid-plane of the bondline rather than close to the aluminum-adhesive interface of the thinner adherend as in an ADCB specimen. Experiments on batch #1 (tested under force control at $R_p=0.1$) resulted in $G_{th}=197 \pm 12 \, J/m^2$, while fatigue tests on batch #5 (tested under displacement...
control, at \( R_\delta = 0.1 \) gave almost the same value, \( G_{th} = 195 \pm 3 \, \text{J/m}^2 \). The crack growth rate curves of the DCB specimens from these two batches are compared in Fig. 16. For the sake of clarity, two representative experiments are shown for each case. The Paris law coefficients, related to the linear part of the crack growth rate graphs, are also given in Fig. 16. The fact that both the fatigue threshold and the fatigue crack growth rates were essentially identical confirms the hypothesis that the batch-to-batch differences were apparent only in mixed-mode fatigue tests, and mainly at low crack growth speeds, where both factors tend to move the crack path closer to the interface.

![Graph showing Gc of aluminum ADCB joints bonded using 5 different adhesive batches. Error bars represent ± 1 standard deviation of the data points on the plateau of the R-curve. \( R_\delta = 1.33 \, \mu\text{m}. \)](image)

**Fig. 15** \( G_c \) of aluminum ADCB joints bonded using 5 different adhesive batches. Error bars represent ± 1 standard deviation of the data points on the plateau of the R-curve. \( R_\delta = 1.33 \, \mu\text{m}. \)

### 3.3.3 Bond Strength Influenced by Surface Treatment

Previous quasi-static fracture tests with the present adhesive and steel bars degreased as described in Section 2.3 resulted in a crack path that was mostly cohesive, with only 10%-20% of the failure surface being interfacial at phase angles \( 0^\circ \leq \psi \leq 55^\circ \), Chapter 2 and ref.[37]. In the present experiments, DCB, ADCB and CLS specimens were fabricated from steel using this same surface preparation and fatigue tested under force control at \( R_\sigma = 0.1 \). The failure mode in all the tested specimens and at all the crack speeds was fully interfacial, showing a poor bonding
under cyclic loading. This is illustrated in Fig. 17 which compares the failure surfaces of a DCB joint tested under quasi-static loading at $\psi=16^\circ$ using the loading jig of ref. [42] with an ADCB joint tested under cyclic loading at a phase angle of $\psi=18^\circ$. Close to the end of the ADCB specimen, where a large static load was applied, the failure became cohesive once again. The use of the Zn-phosphate pretreatment on the steel bars improved the bond strength markedly, causing the crack path to become fully cohesive under cyclic loading (Chapter 3). The effect of surface treatment on the fracture of the Zn-phosphate steel adhesive system was not investigated; however, due to the mostly cohesive fracture seen with the degreased steel system [38], it was expected that Zn-phosphating would not cause a significant change in the fracture strength. The change in the failure mode, from interfacial to cohesive, improved the fatigue behavior of the steel adhesive system at all crack speeds. For example, the $G_{th}$ values of Zn-phosphate treated DCB and ADCB joints were almost 4-5 times greater than those of the degreased joints (see Table 5).

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**Fig. 16** Fatigue crack growth behavior of DCB joints from batches #1 and #5. $m$ and $b$ are as per Eq. (3), $N$ is the number of specimens, ± the standard deviation. Results for two representative experiments are shown for each adhesive batch. Batch #1 was tested under force control, while batch #5 was tested under displacement control. $R_s=1.33 \, \mu m$. 

---

Table 5

<table>
<thead>
<tr>
<th>Batch #</th>
<th>m ± SD</th>
<th>b ± SD</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.7±0.3</td>
<td>-16.7±0.5</td>
<td>3</td>
</tr>
<tr>
<td>5</td>
<td>4.4±0.2</td>
<td>-15.7±0.6</td>
<td>3</td>
</tr>
</tbody>
</table>
Fig. 17 Failure surfaces of (a) degreased steel DCB tested under quasi-static loading at $\psi=16^\circ$ and (b) degreased steel ADCB tested under cyclic loading at $\psi=18^\circ$ under force control. Specimens were 19 mm wide.

Table 5 Effect of surface treatment on fatigue threshold of steel adhesive system, $R_a=1.44 \ \mu m$, tested under force control. ± the standard deviation, $N$ is the number of experiments.

<table>
<thead>
<tr>
<th>Specimen, Phase Angle</th>
<th>Degreased</th>
<th>Zn-phosphated</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$G_{th}$ (J/m$^2$)</td>
<td>$N$</td>
</tr>
<tr>
<td>DCB, 0°</td>
<td>35 ± 10</td>
<td>4</td>
</tr>
<tr>
<td>ADCB, 18°</td>
<td>36 ± 7</td>
<td>4</td>
</tr>
<tr>
<td>CLS, 50°</td>
<td>106 ± 31</td>
<td>4</td>
</tr>
</tbody>
</table>

Such a difference in the fatigue and fracture failure surfaces was also observed for aluminum joints. CLS and SLS joints were fabricated from AA6061-T651 flat bars and were tested under quasi-static loading to measure the ultimate joint strengths, Chapter 2 and ref. [37]. Prior to bonding, the aluminum bars were abraded to an average $R_a=0.77 \ \mu m$. A satisfactory bonding with a fully cohesive failure was achieved in quasi-static fracture (see Fig. 5(c)). However, when the same CLS joints were tested under cyclic loading, an interfacial failure was
observed, Chapter 3 (see Fig. 5(a)).

3.3.4 Discussion

As observed from all the case studies in this section, mixed-mode low-speed fatigue cracks tend to propagate very close to the interface on the more highly-strained adherend and are thus highly sensitive to the interfacial bond strength. Fracture and fatigue tests at higher crack growth rates and/or mode I tend to generate crack paths that are further from the interface and are therefore less sensitive to the interface condition.

The effect of the phase angle is well known, with the crack path moving from the mid-plane towards the more highly-strained adherend as the phase angle increases [35,37,23].

The relation between the cyclic crack speed and the crack path has not been established, although several hypotheses have been advanced in Appendix A. One possible reason is an increase in the local phase angle at the crack tip resulting from an increase in the adhesive stiffness due to the cycling. This is supported by the present data which showed that the CLS joint stiffness increased with the number of cycles in the threshold region and the period prior to crack initiation in the fillet. This implies that as the crack speed decreases the local phase angle at the crack tip increases, which moves the crack closer to the interface and causes the fatigue performance to become more sensitive to the bond strength. Other possible reasons for the increase in the local phase angle with decreasing crack speed are changes in adhesive temperature and stiffness with crack speed, either due to hysteretic heating or friction between the fracture surfaces. Plastic deformations in the damage zone or local toughening at the crack tip [6] were also suggested as possible mechanisms for the effect of cyclic crack speed on crack path. Based on the local toughening hypothesis the crack is deflected around the zone that is toughening as the cyclic loading continues for long periods near the threshold.

4. Conclusions

The fatigue threshold measured at the tip of a fully developed fatigue crack was the same as that for an uncracked fillet provided that the crack path after initiation was cohesive. When the crack path became interfacial after beginning cohesively, a lower $G_{th}$ was measured from a fatigue pre-crack. As a result, it is concluded that measuring $G_{th}$ from an established fatigue crack provides a better assessment of an adhesive system for design purposes. Surface roughness had no effect on $G_{th}$ and the average number of cycles to crack initiation when loading
at $G_{th}$ from an intact fillet.

Cyclic loading at low loads in the threshold region caused crack initiation in a relatively small number of cycles compared to the expected total fatigue life of the joint. This suggests that for the current adhesive system the crack initiation phase can be safely ignored in life prediction modeling under low load conditions.

Fatigue tests conducted under force control yielded the same threshold and crack growth rate as those made with displacement control using load and displacement ratios of 0.1 and 0.5. This result supports the use of displacement control, which is usually a shorter and more convenient testing method than load control. The fatigue test results were independent of $dG/da$, and it was discovered that artificial differences between force and displacement controlled tests can result if the adhesive layer compliance is neglected in the calculation of the strain energy release rate when plotting the data.

The crack path in fatigue was observed to be a function of both the phase angle and the crack speed. Mixed-mode fatigue testing at low crack speeds near the threshold tended to drive cracks close to the interface of the more highly strained adherend and was highly sensitive to the bond strength of this interface. In contrast, typical quasi-static mixed-mode fracture tests, such as DCB, cracked lap shear or single lap shear joints, could not distinguish between adhesive systems having subtle differences in bond strength because the crack paths tended to be further from the interface. This was also true of mode I fatigue testing since the crack path tended to be in the middle of the adhesive layer rather than being driven toward the more highly strained adherend, as happens under mixed-mode loading. Higher crack growth rates caused the crack path to move away from the interface and the fatigue behavior also became less sensitive to the interfacial bond strength. This effect was due to the mechanics of fatigue crack growth, and was not a function of environmental attack since the present experiments were conducted in dry air. It is therefore concluded that mixed-mode fatigue testing in the vicinity of the threshold is a highly effective way of assessing the interfacial bond strength of an adhesive system. Subtle changes in the adhesive, substrates, pretreatment or surface roughness were found to affect the mixed-mode, low speed fatigue results, but had very little influence on the either the mixed-mode fracture behavior or higher speed crack growth. This conclusion is of particular importance in applications experiencing high cycle mixed-mode fatigue loading near the threshold region. In such cases, the cohesive failure surfaces associated with good bonding can become unexpectedly interfacial if prior testing has not included fatigue at low crack growth rates.
Appendix 4. Effect of Displacement and Load Ratio on Fatigue Threshold

A.1. Introduction

Fatigue threshold experiments are performed typically using either a constant load or displacement ratio, $R$. For load control, $R=P_{\text{min}}/P_{\text{max}}$, where $P_{\text{min}}$ and $P_{\text{max}}$ are the minimum force and the maximum force in a cycle, respectively. For a displacement control test, $R=d_{\text{min}}/d_{\text{max}}$, where $d_{\text{min}}$ and $d_{\text{max}}$ are the minimum displacement and the maximum displacement in a cycle, respectively. It is necessary for a threshold-based design to understand how the fatigue threshold may change with the load or displacement ratio.

Mall et al. [22] investigated the influence of the load ratio on the crack propagation rate in DCB and cracked lap shear (CLS) joints. They found that $da/dN$ decreased as $R$ increased for a given $G_{\text{max}}$. This is reasonable, because increasing $R$ while keeping the maximum load constant, $G_{\text{max}}$, reduces the magnitude of the range of the applied cyclic load, in the limit approaching the quasi-static case. However, this apparent improvement was found to disappear when the $da/dN$ curves were plotted against $\Delta G=G_{\text{max}}-G_{\text{min}}$ rather than $G_{\text{max}}$ [22,44]. In fact, Pirondi and Nicoletto [4] found a higher crack growth rate under constant $\Delta G$ for $R=0.4$ in comparison with $R=0.1$. In other words, they found that increasing load ratio, $R$, while keeping $\Delta G$ constant led to an increase in $da/dN$. This may simply have been due to the increased average $G$ applied during the load cycle. After counting for the crack closure phenomenon, by considering $\Delta G_{\text{eff}}$ rather than $\Delta G$, the crack growth rate graphs became much closer to each other.

The idea of modifying the Paris law equation, which represents the linear part of the crack growth rate versus the applied strain energy release rate graph, to incorporate load ratio effects has been done in various ways [45-51]. For instance, Jia and Davalos [52] studied the mode I fatigue behavior of a wood-FRP composite bonded interface using contoured DCB (CDCB) specimens. They found that a modified form of the Paris law equation could account for the load ratio effect; i.e. a single curve could represent the fatigue crack growth rate graphs at $R=0.1$, 0.3 and 0.5. The modified equation was based on $\Delta G$ and $G_{\text{min}}$, rather than only $G_{\text{max}}$ or $\Delta G$ in the classic form. $\Delta G$ at the threshold was found to decrease as $R$ increased; however, $G_{\text{max}}$ at threshold was almost unchanged [52]. Therefore, it was concluded that the fatigue threshold is controlled by $G_{\text{max}}$.

This appendix describes the effect of load and displacement ratio on the mixed-mode
fatigue threshold behavior of an aluminum-epoxy adhesive joint. The specimen preparation, fatigue testing approach and the experimental results have already been presented in Chapter 4, and thus only the final threshold measurements are discussed here.

A.2. Results and Discussion

Experiments were conducted under both displacement control and load control. The load and displacement ratios of 0.1 and 0.5 were chosen for this study. Table A.1 gives the measured fatigue threshold, $G_{th}$, at load ratios of 0.1 and 0.5, while Table A.2 lists the results for the displacement controlled tests. $G_{max}$ is the maximum strain energy release rate in a cycle. $\Delta G = G_{max} - G_{min}$, where $G_{min}$ is the minimum strain energy release rate in a cycle. $G_{AP}$ is an effective strain energy release rate over the loading range calculated using a single load equal to $\Delta P = P_{max} - P_{min}$, where $P_{max}$ and $P_{min}$ are the maximum and the minimum forces in a cycle, respectively. It is seen that the effect of load and displacement ratio was significant when the threshold was expressed in terms of either $G_{max}$ or $\Delta G$. Increasing the load and displacement ratio from 0.1 to 0.5 increased the $G_{max}$ and $\Delta G$ at threshold by about three times; however, the effect became insignificant when the strain energy at the threshold was calculated in terms of $\Delta P$.

As can be seen from the beam-on-elastic foundation model presented in Chapter 3, $G$ is proportional to $P^2$, therefore, $G_{max}$ is proportional to $P_{max}^2$; $\Delta G$ is proportional to $\Delta(P_{max}^2 - P_{min}^2)$, and $G_{AP}$ is proportional to $(\Delta P)^2$.

The stress intensity factor, $K$, is widely used in fracture mechanics studies of metallic materials, with the following relation between $G$ and $K$ [53]:

$$G = \frac{K^2}{E}$$

where $E$ is the elastic modulus. The strain energy release rate is popular in the analysis of adhesive joints, since it can be calculated without knowing the exact stress state at the crack tip. Stress analysis at the crack tip in adhesive joints is more complex than in standard metallic specimens, due to the layered system and the damage zone at the crack tip.

In the fatigue analysis of metallic materials using the fracture mechanics approach, results are usually presented as a function of $\Delta K = K_{max} - K_{min}$, with $K_{max}$ and $K_{min}$ being the maximum and the minimum stress intensity factor in a cycle, respectively. Since the stress intensity factor is proportional to the force, $\Delta K$ is proportional to $\Delta P$. Therefore, when $G$ is calculated based on $\Delta P$, $G_{AP}$, the strain energy calculation becomes analogous to the use of $\Delta K$ as an independent variable in fatigue analysis. As can be seen from Tables A.1 and A.2, there was...
no significant difference between the values of $G_{\text{max}}$, $\Delta G$ and $G_{\Delta P}$ at $R=0.1$ ($t$-test, 95% confidence). The reason is that at such a small load ratio, $G_{\text{min}}$ and $P_{\text{min}}$ are so small that they have little effect on these three calculated values. This result, that the fatigue behavior can be better illustrated with a stress-based approach, $K$, rather than an energy-based approach, $G$, at crack growth rates as low as the fatigue threshold is consistent with the approach usually followed in the analysis of high-cycle fatigue [54]. This observation is also supported by the fact that the damage zone size, and thus the energy dissipation at the crack tip region, is relatively small, and therefore an energy-based approach may not be as suitable as a stress-based technique for the characterization of the threshold behavior.

This conclusion is of particular significance in the design of adhesive joints for automotive applications, where a reliable design approach should be based on either the fatigue threshold or a very small crack growth rate. A fatigue threshold test at a single load or displacement ratio should be enough for adhesive joint design purposes.

Table A.1 Fatigue threshold measurements at load ratios of 0.1 and 0.5. $N$ is the number of threshold measurements.

<table>
<thead>
<tr>
<th>Load ratio</th>
<th>$N$</th>
<th>$G_{\text{max}}$ (J/m$^2$)</th>
<th>$\Delta G$ (J/m$^2$)</th>
<th>$G_{\Delta P}$ (J/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>3</td>
<td>124 ± 6</td>
<td>125 ± 6</td>
<td>102 ± 5</td>
</tr>
<tr>
<td>0.5</td>
<td>4</td>
<td>454 ± 22</td>
<td>340 ± 17</td>
<td>114 ± 6</td>
</tr>
</tbody>
</table>

Table A.2 Fatigue threshold measurements at displacement ratios of 0.1 and 0.5. $N$ is the number of threshold measurements.

<table>
<thead>
<tr>
<th>Displacement ratio</th>
<th>$N$</th>
<th>$G_{\text{max}}$ (J/m$^2$)</th>
<th>$\Delta G$ (J/m$^2$)</th>
<th>$G_{\Delta P}$ (J/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>6</td>
<td>121 ± 12</td>
<td>121 ± 11</td>
<td>121 ± 12</td>
</tr>
<tr>
<td>0.5</td>
<td>3</td>
<td>385 ± 11</td>
<td>307 ± 7</td>
<td>117 ± 3</td>
</tr>
</tbody>
</table>
5. References

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Chapter 5

Hypotheses for the Effect of Crack Speed on
Crack Path Selection

1. Introduction

As was mentioned in Chapters 3 and 4, as the crack speed increased under mixed-mode loading (due to increasing $G_{max}$), more residual adhesive remained on the more highly strained adherend. This is shown for two typical aluminum ADCB joints ($\psi=18^\circ$) in Fig. 1. Figure 1(a) was fatigue tested under force control, while Fig. 1(b) was fatigue tested under displacement control followed by a quasi-static fracture test. The crack path at high fatigue crack growth rates (up to $10^{-2}$ mm/cycle) approached that seen in fracture tests. Figure 2 also shows the measured residual adhesive thickness on the more strained arm of a force control tested aluminum ADCB joint ($\psi=25^\circ$) versus the crack growth rate. Similar to the lower phase angle ADCBs, Fig. 1, the crack path became more cohesive as the crack growth rate increased. No such a crack growth rate dependency has been reported in the literature. This appendix seeks different hypotheses to explain this behavior.

2. Change in the Local Phase Angle

The mode-I criterion, or sometimes called “the criterion of local symmetry”, states that a crack in a brittle homogeneous isotropic material selects a mode I path, i.e. for which $K_{II} = 0$ [1,2]. Therefore, any parameter that affects the local phase angle in the crack tip region can affect the crack path in the adhesive layer. The elastic mismatch between the adhesive and adherends causes the local phase angle at the crack tip to decrease from that of a homogenous material, or a multilayer with the same Young’s modulus for each layer [3,4]. Increases in the Young’s modulus of polymers and adhesives due to cyclic loading have been reported in the literature [5-7], and attributed to polymer chain alignment in the load direction [5]. Therefore, one possible reason for the dependency of crack path on crack speed could be a change in the local phase angle at the crack tip resulting from an increase in the adhesive stiffness due to the cycling. To test this hypothesis, aluminum ADCB joints were tested under displacement control,
for which \( G \) decreases as the crack grows. After reaching the threshold \( 10^{-6} \) mm/cycle crack speed, the cyclic loading was continued so that the crack slowed even further. The measured joint stiffness increased 1-2% as the adhesive was subject to a large number of cycles (about 200,000) at these small crack speeds (\( 10^{-6} \) to \( 10^{-7} \) mm/cycle). Since the contribution of the adhesive stiffness to the total joint stiffness is small, and the change in the adhesive properties is limited to a small area around the crack tip, this relatively small increase in the joint stiffness indicates a much larger stiffening of the adhesive in the crack tip region. The increase in the adhesive Young’s modulus decreases the elastic mismatch between the adhesive and the adherend, thereby increasing the local phase angle at the crack tip [3,4]. Such an increase would cause the crack path to be closer to the interface; however, it is not known if this stiffening effect would be greater at high or low crack growth rates, and hence whether it is consistent with the experimental observations. Furthermore, using the FE model described in Chapter 3 it was estimated that this effect would change the local phase angle by only \( 1^\circ - 2^\circ \). This was also verified following the analytical model proposed by Fleck et al. [3] for the phase angle shift due to the elastic mismatch in a multilayer. It is likely that such a small phase angle change was not solely responsible for the observed change in the crack path with crack speed.

---

Fig. 1 Effect of crack speed on the mixed-mode crack path for an aluminum ADCB specimen tested in (a) fatigue (force control) and (b) fatigue (displacement control) and quasi-static loading.
An increase in temperature at the crack tip would decrease the elastic modulus of the adhesive as the crack speed increases [8,9]. This might result from hysteretic heating of the viscoelastic adhesive or friction between the fracture surfaces [10-14]. Figure 3 shows how the measured area of the hysteresis loop increases with an increase in the crack growth rate in an aluminum ADCB joint. The measurements were conducted using the recorded clip gauge force-displacement curves during fatigue testing. Such an increase in the released energy may increase the temperature of the adhesive at the crack tip, which can consequently decrease $E$ of the adhesive in that area. As discussed above, the decrease in $E$ decreases the local phase angle and thus makes the crack path more cohesive, consistent with the experimental results.

A change in the adhesive layer compliance can also result from damage evolution and accumulation in the adhesive layer under cyclic loading [15-17], thereby causing a decrease in the elastic modulus of the adhesive as microcracks nucleate and grow [15]. According to this mechanism, the larger the $G$ (or equivalently, the higher the crack speed) the greater the density of the microcracks and thus the lower the elastic modulus of the adhesive. A lower adhesive modulus would decrease the phase angle at the crack tip and cause the crack to move further from the interface as the crack speed increases, consistent with what was observed.
Fig. 3 Relation between the fatigue crack growth rate and the hysteresis loop area in an aluminum ADCB joint.

An increase in the crack growth rate due to increasing applied $G$ will increase the strain rate at the crack tip. As an example, using the 2D finite element model explained in Chapter 3, it was found that increasing $G$ from 200 J/m$^2$ (close to $G_{th}$ of aluminum ADCBs from batch #1) to $G=1,000$ J/m$^2$ increased the strain rate at the crack tip by 2.5 times. The simulated situation was based on a force-controlled ADCB joint, in which the threshold was reached at a crack length of ~60 mm, and $G=1,000$ J/m$^2$ was reached at a crack length of ~150 mm. The adhesive was a viscoelastic material and its elastic modulus usually increases with strain rate [18,19]. However, as with the previous mechanisms that could affect the local phase angle, the typical changes in the elastic modulus with strain rate will make a small change in the local phase angle at the crack tip. In addition, even if the change was significant, the trend is opposite to the observed crack path dependency pattern; i.e. as the crack speed increases the elastic modulus of the adhesive increases, which raises the local phase angle. Such a change in the local phase angle should move the crack path closer to the interface as the crack speed increases.

Therefore, any mechanism that increases the compliance of the adhesive layer with increasing crack growth rate may result in a crack path selection consistent with the experimental observations. As discussed above, these mechanisms include stiffening of the adhesive due to cycling, raise in the crack tip temperature as a result of the energy released at the crack tip, and
damage evolution and accumulation in the adhesive layer due to the cyclic loading.

3. Local Toughening and the Effect of Damage Zone

Another possible explanation for the change in the crack path with crack speed is local toughening at the crack tip due to either prolonged cyclic loading or sustained constant loading of an adhesive, similar to what has been reported in refs. [20,21]. It is hypothesized that this might cause the crack to deflect to follow the weaker path around the locally toughening material. There seems to be some evidences for the presence of such a mechanism in the studied adhesive system. As described in Chapters 3 and 4, in the force controlled testing approach the force was decreased gradually until reaching to the threshold, followed by an increase in the applied load to start measuring the crack growth rate behavior. In the case of about 1/3 of the tested specimens, after measuring the threshold and increasing the force, the crack did not start propagating with the same speed at which it was propagating prior to measuring $G_{th}$. The applied force had to be increased several times to be able to measure the crack growth rate graph. This behavior could be due to the toughening of the adhesive in the threshold crack tip region, which was under the cyclic loading for over a million cycles.

In other work currently ongoing to understand the degradation behavior of a similar adhesive system under quasi-static loading by another PhD student, it was observed that as the joint’s exposure to the degradation condition increases, and thus $G_{c}$ decreases, the crack path under mixed-mode loading becomes closer to the interface. This suggests that the mechanism behind crack path selection is not related solely to cyclic loading.

Different criteria have been proposed for crack path selection in brittle materials. As mentioned above, one is the mode-I criterion [1,2], which states that the crack follows a pure mode I path. Based on this criterion, Hutchinson and Suo [4] analytically derived the crack path as a function of remote loading phase angle and for different elastic mismatch in a multi-layer. For the tested adhesive system in the current work, the analysis of ref. [4] showed that a mode-I crack path exists in the adhesive layer only for remote phase angles below ~9°. For phase angles above this value, there will be no pure mode-I path in the bondline, and thus the crack should propagate in the interface. Hence, this criterion cannot explain the dependence of crack path on speed.

Using the finite element analysis, the $K_{II}=0$ path was calculated as a function of adherend thickness ratio in an aluminum ADCB. Figure 4 shows that there is no path corresponding to
$K_{II} = 0$ for adherend thickness ratios above 1.35. This geometry represents a remote loading phase angle of $9^\circ$, which supports the analytical model of Hutchinson and Suo [4]. Therefore, based on the mode I crack path criterion, the crack in the aluminum ADCB joints with thickness ratio of 2 should propagate in the aluminum-adhesive interface.

![Graph](image)

Fig. 4 $K_{II} = 0$ path in aluminum ADCB joints as a function of adherend thickness ratio. $c$ is the distance between the upper interface and the crack tip, and $t$ is the adhesive thickness.

Another criterion widely used to determine the crack path in brittle materials is the maximum opening stress by Erdogan and Sih [22]. This criterion proposes that the crack will follow the path in which the maximum opening stress occurs. The finite element model was used to investigate the crack path in aluminum ADCB joints with thickness ratio of 2. Similar to the mode-I criterion, the crack was found to keep propagating towards the more strained adherend, the thinner arm, prior to reaching the interface and propagating in the interface.

These analyses suggest that there must be a mechanism that prevents the crack in ADCB joints to select the path at the interface between the adhesive and the adherend. It is hypothesized that plastic deformation in the damage zone causes the adhesive to become tougher, thereby preventing the crack from following its growth path at the interface. This is similar to the crack tip toughening mechanism discussed in the previous section, but in this context its effect is to keep the crack away from the interface. The effect of $G$ on the damage zone thickness and size will be illustrated in Chapter 7. At threshold, for $t=380 \ \mu$m, the damage
zone is small, and thus the crack can move closer to the interface. As $G$ increases, and as a result
the crack growth rate, the damage zone becomes larger and pushes the crack farther away from
the interface. Therefore, the higher the $G$, the more cohesive the failure becomes.

As was shown in Chapter 4, Table 4, increasing the load ratio (minimum force divided by
the maximum force in a cycle) from $R=0.1$ to $R=0.5$ significantly increased the $G_{\text{max}}$ and $\Delta G =
G_{\text{max}} - G_{\text{min}}$ for aluminum ADCB joints. Consistent with the above hypothesis, the crack path at
the same $G$ for $R=0.5$ was significantly farther from the interface compared to that of $R=0.1$. To
prove the validity of this hypothesis, further studies and analyses are required.
4. References


Chapter 6

Effect of Bondline Thickness – Part I: Experiments

1. Introduction

An understanding of the effect of bondline thickness on the quasi-static and cyclic loading behavior of adhesive joints can guide the selection of an optimal adhesive thickness and indicate the sensitivity to variations in thickness that may be caused by processing limitations.

Most studies of the effect of adhesive bondline thickness have examined quasi-static fracture under either mode-I [1-6] or mixed-mode loading [7-11]. Some investigators have observed a significant strength improvement in adhesive joints with increasing adhesive thickness [5,6,12], while others have reported no significant effect [1,7], or even a decrease [10,11] in strength. It has also been reported that the $G_s^c$, the quasi-static steady-state critical strain energy release rate, initially increases with bondline thickness and then decreases, eventually reaching a constant value [1,2].

Fatigue crack growth rates were found to decrease with increasing adhesive thickness under mode I loading [13-15]. However, Mall and Ramamurthy [13] found that the fatigue crack growth rates in mode I tests did not change when the bondline thickness was increased from 0.102 mm to 0.254 mm, and improved at 0.508 mm only at higher crack growth rates. At lower crack growth rates, the fatigue resistance was the same for all three bondline thicknesses. A more significant effect of bondline thickness was found when the adhesive was filled and toughened, compared to only a filled adhesive [15]. This was attributed to the larger plastic zone size of the tougher, more ductile adhesive. In general, the effect of bondline thickness on the fatigue and fracture behavior of adhesive joints has been attributed mainly to the relative size of the bondline thickness and the plastic zone ahead of the crack, with the plastic zone size being affected by the adherend restraint [1,2,15].

There have been relatively few papers dealing with the effect of bondline thickness under mixed-mode cyclic loading. Since most practical joints are loaded under mixed-mode conditions, this represents a significant gap in the literature. Schmueser [16] measured an increase in the cyclic debonding rate of mixed-mode steel cracked lap shear joints with
increasing bondline thickness. The crack path in these joints was interfacial.

As mentioned above, there have been very few papers dealing with the effect of bondline thickness under mixed-mode cyclic loading, and very little work exists in the fatigue threshold region \((da/dN < 10^{-5} \text{ mm/cycle})\) for any mode ratio. The present chapter describes experiments on the quasi-static and cyclic behavior of a highly-toughened epoxy adhesive as a function of bondline thickness under both mode I and mixed-mode loading. The effect of substrate material on the mode-I fatigue performance was also studied by comparing aluminum and steel adherends. The next chapter examines these data in terms of the effect of bondline thickness and adherend material on the stress distribution and plastic zone size using finite element modeling. Results are also presented in ref. [17].

2. Experimental Approach

2.1 Specimen Preparation

Aluminum double cantilever beam (DCB) and asymmetric double cantilever beam (ADCB) specimens (Fig. 1) were used for testing under mode I \((\psi = 0^\circ)\) and mixed-mode \((\psi = 18^\circ)\) conditions, respectively. The phase angle is a measure of the mode ratio of loading, and is defined as \(|\psi| = \arctan \left( \sqrt{G_{II}/G_I} \right)\), where \(G_I\) and \(G_{II}\) are the mode I and II components of the strain energy release rate. Aluminum specimens were fabricated from 12.7 mm×19.05 mm (1/2”×3/4”) and 25.4 mm×19.05 mm (1/2”×3/4”) AA6061-T651 flat bars. Prior to bonding, the substrates were abraded using a silicon carbide nylon mesh abrasive pad which produced an \(R_a=1.33 \mu m\) (standard deviation of 0.16 \(\mu m\) over 4 measurements on 4 different samples and for a scan length of 15 mm). The aluminum bars were then pretreated using the P2 etching process [18]. To remove the excessive adhesive after curing, the sides of the joints were sanded using a disc sander with water as a coolant, followed by a light sanding using a belt sander with 120 grit paper. To further improve the visibility of the crack and minimize any surface damage, the bondlines were finished by hand with 600 grit sandpaper, and a thin coating of diluted white correction fluid was applied to provide a high-contrast image.
Specimens with three different bondline thicknesses were manufactured using spacing wires with diameters of 0.13 mm (0.005”), 0.38 mm (0.015”) and 0.79 mm (0.031”). In all experiments, a single-part heat-cured highly-toughened epoxy adhesive was used.

2.2 Fatigue Testing

All fatigue experiments were carried out at a cyclic frequency of 20 Hz in a desiccant chamber to achieve a dry condition (11% - 15% relative humidity). The experiments were conducted under displacement control, with a constant displacement ratio, $R=\delta_{\text{min}}/\delta_{\text{max}}=0.1$, measured at the loading pins. The tests began with application of the highest strain energy release rate, $G$, which then decreased as the crack grew under constant loading pin displacement until the threshold crack growth rate ($10^{-6}$ mm/cycle) was reached at the threshold strain energy release rate, $G_{\text{th}}$. It was previously shown in Chapter 4 that testing under force or displacement control does not significantly affect the fatigue behavior [19].

The unloading joint compliance approach [20] was used to measure the fatigue crack length. The joint compliance was measured during the unloading portion of the cycle using the load cell output and a clip gauge attached to the end of the specimen. A CCD camera (2 mm
field of view) on a motorized linear stage was used to measure the crack length and relate it to the measured joint compliance for a given specimen type, using the approach of ref. [21]. A least squares regression was used to fit a third-order polynomial, with $c_1$ to $c_4$ as the constants, to the normalized crack length, $a/w$, versus the normalized specimen compliance, $CEB$, for fatigue joints:

$$
a/w = c_1 \times (CEB)^3 + c_2 \times (CEB)^2 + c_3 \times (CEB) + c_4
$$

where $a$ is the crack length, $w$ is the specimen length from the loading pins, $C$ is the compliance, $E$ is the tensile modulus of the adherends, and $B$ is the specimen width.

### 2.3 Fracture Testing

The fracture tests were performed on aluminum ADCB joints. The same load was applied on the both arms of the ADCB at a constant cross-head speed of 1 mm/min to produce a mixed-mode loading condition ($\psi = 18^\circ$) at the crack tip. The crack length was measured from the center of the loading pins to the tip of the macro-crack on the ADCB specimens using a microscope mounted on a micrometer stage. Crack growth was stable in this system so that many crack extension events could be recorded with a single ADCB specimen. To measure the critical load at each crack length, the cross-head displacement was started and stopped repeatedly in the vicinity of the expected fracture load (each time at a constant cross-head speed of 1 mm/min) until a drop in the applied load was observed. This maximum load prior to the drop was taken as the critical fracture load for the measured crack length if visual inspection through the microscope confirmed that the macro-crack had propagated. After measuring the new macro-crack length, the ADCB was unloaded and the same procedure was followed again beginning at the new crack length.

The effect of bondline thickness on the mode-I and mixed-mode crack initiation strain energy release rate, $G_{ci}$, was also tested. These quasi-static fracture measurements were made after the fatigue threshold tests. Crack initiation was defined as the first crack extension of approximately 50 µm from the fatigue precrack corresponding to $G_{th}$.

### 2.4 Strain Energy Release Rate Calculation

The strain energy release rate, $G$, for DCB and ADCB joint was calculated from the measured force and crack length using an analytical beam-on-elastic-foundation model
These predicted $G$ values for aluminum and steel DCB and ADCB joints were within 2% of those predicted from a two-dimensional elasto-plastic finite element model for crack lengths of 40-120 mm.

3. Experimental Results and Discussion

3.1. Fatigue Tests

Figure 2 shows that increasing the bondline thickness from 130 µm to 380 µm doubled $G_{th}$ under mode-I loading. However, no further statistically significant improvement in $G_{th}$ was observed by increasing the adhesive thickness to 790 µm (t-test, 95% confidence). The threshold under mixed-mode loading depended only slightly on the bondline thickness in the studied range; the only statistically significant difference was between $G_{th}$ at the largest (790 µm) and smallest (130 µm) bondline thickness (t-test, 95% confidence).

Figures 3 and 4 show the measured fatigue crack growth rates versus $G_{max}$, the maximum energy release rate in a cycle, for all the tested DCB and ADCB specimens. For the sake of clarity, only three of the six experiments are shown for the mixed-mode ADCB joints with $t=380$ µm. The fatigue results were very repeatable for both the DCB and ADCB joints, showing two
distinct regions, a linear region (Paris law region) at higher crack growth rates, and a threshold region. To show the effect of bondline thickness on the crack growth rate behavior, a least squares regression line (Eq. 2) was fitted to all the data in the linear region at each mode ratio and bondline thickness.,

\[
\log (\frac{da}{dN}) = m \times \log (G_{\text{max}}) + b
\]  

(2)

where \(a\) and \(N\) are the crack length and the cycle number, respectively, with \(m\) and \(b\) being the constants calculated from the regression fit.
Fig. 3 Fatigue crack growth rates for mode-I DCB specimens with bondline thicknesses of (a) 130 µm, (b) 380 µm and (c) 790 µm.
(a)

Log (da/dN), mm/cycle

Log (G max), J/m^2

Test #1
Test #2
Test #3
Test #4

(b)

Log (da/dN), mm/cycle

Log (G max), J/m^2

Test #1
Test #2
Test #3
Fig. 4 Fatigue crack growth rates for mixed-mode ADCB specimens with bondline thicknesses of (a) 130 µm, (b) 380 µm and (c) 790 µm.

Figure 5(a) shows that the crack growth rates for \( t=380 \) µm and 790 µm were very similar when the crack was close to the threshold region under mode-I loading, but there was a small decrease in crack growth rate with increasing bondline thickness as \( G_{\text{max}} \) increased. The crack growth rate at \( t=130 \) µm was significantly higher than at \( t=380 \) and 790 µm (t-test, 95% confidence). Overall, as the crack speed increased, the effect of bondline thickness on the fatigue performance became slightly more pronounced (Fig. 5(a)).

For mixed-mode loading, Fig. 5(b), the crack growth rates were similar for the three bondline thicknesses when the crack speed was in the threshold region. As \( G_{\text{max}} \) increased, however, the crack growth rate for the thinnest adhesive layer, \( t=130 \) µm, increased significantly. The crack growth rates for \( t=380 \) µm and 790 µm were indistinguishable (t-test, 95% confidence). As with the DCB joints, the effect of bondline thickness on the fatigue performance became more pronounced as the crack growth rate (or \( G \)) increased. For example, at \( G=400, 800 \) and 1200 J/m\(^2\), the fatigue crack in the thinnest joint (\( t=130 \) µm) propagated 3, 8 and 11 times faster than that in the thicker joint (\( t=380 \) µm).
Fig. 5  Effect of bondline thickness on fatigue crack growth rate at different strain energy release rates for (a) mode-I loading and (b) mixed-mode loading. The lines are the best fits to the Paris law (linear) region in each of Figs. 3 and 4.
The effect of bondline thickness on fatigue and fracture performance is usually explained in terms of the effect of adherend constraint on the damage zone in the adhesive layer. As the damage zone becomes smaller, the effect of the substrate on the damage zone shape and size is reduced, and at some point, the damage zone becomes too small to be affected by the adherend constraint. The damage zone size depends on the applied load and the mechanical properties of the adhesive. As the applied force, and thus the applied $G$ decreases, the damage zone becomes smaller. This may explain the experimentally observed trend that the effect of bondline thickness becomes less pronounced at lower crack growth rates, when the applied $G_{max}$ is also lower. The effect of adherend constraint on the stress distribution and the damage zone ahead of the crack tip in the adhesive layer under both mode-I and mixed-mode loading will be considered in detail using finite element modeling in the next chapter.

As shown in Figs. 2 and 5(b), the mixed-mode $G_{th}$ did not change appreciably with bondline thickness. Since the majority of practical joints are loaded under mixed-mode conditions, the relatively low sensitivity of the mixed-mode $G_{th}$ to the bondline thickness has important implications for adhesive joint design based on the fatigue threshold. For instance, in many industrial applications, accurate control over the adhesive thickness is not feasible; however, the present results indicate that such control may not be necessary under mixed-mode loading conditions. Furthermore, the measurement of $G_{th}$ at a single bondline thickness for the desired range of phase angles is probably sufficient.

3.2. Fracture Tests

Quasi-static fracture tests using ADCB specimens produced a typical R-curve behavior [22]. After crack initiation at $G_c^i$, the first several crack growth sequences occurred at an increasing critical energy release rate, $G_c$, as the damage zone at the crack tip developed to its steady-state form [23] (Fig 6). The steady-state critical strain energy release rate, $G_c^s$, was considered to be the average value over this “plateau” (steady-state) region.
The effect of bondline thickness on $G_c^i$ measured from the fatigue threshold precrack is shown in Figs. 7 and 8 for DCB and ADCB joints, respectively. $G_c^i$ increased with bondline thickness under mode-I loading (Fig. 7), but the decreasing slope suggested an approach to a plateau. A similar trend was observed under mixed-mode loading (Fig. 8), although a plateau value of $G_c^i$ may already have been reached since the values at 380 µm and 790 µm were indistinguishable (t-test, 95% confidence). As is typical of crack initiation measurements, there was a relatively large scatter in the experimental results due to microscopic differences in the local geometry and adhesive composition and uncertainties in the detection of crack initiation [24]. The latter is compounded by the inability to observe initiation at locations other than the single side viewed by the microscope.

Comparing Figs. 7 and 2, shows that both $G_c^i$ and $G_{th}$ exhibited a somewhat similar trend with bondline thickness under mode I loading. The same can be said under mixed-mode conditions (Figs. 8 and 2). These observations are not too surprising since fatigue crack propagation near the threshold and crack initiation both involve minute amounts of crack extension from a relatively small damage zone (low $G$). They do indicate that strain rate effects were small with this adhesive over this range of loading conditions, since the fatigue tests were conducted at 20 Hz while the fracture initiation was quasi-static.
Fig. 7  Effect of bondline thickness on the average mode-I quasi-static initiation strain energy release rate, $G_{ci}$. Five initiation measurements were performed at $t=380$ µm, with three measurements at the other two bondline thicknesses. Black dots show each $G_{ci}$ measurement, with the average at each adhesive thickness given with the hollow diamond.

Fig. 8  Effect of bondline thickness on the average mixed-mode quasi-static initiation strain energy release rate, $G_{ci}$. Four initiation measurements were performed at $t=380$ µm, and three measurements at the other two bondline thicknesses. Black dots show each $G_{ci}$ measurement, with the average at each adhesive thickness given with the hollow diamond.
Bondline thickness had a large effect on the quasi-static critical strain energy release rate, $G_c^s$, as shown in Fig. 9. The standard deviations were based on 24, 87 and 46 data points on the plateaus of the R-curves at $t=130$ µm, 380 µm and 790 µm, respectively. Three specimens were tested at $t=380$ µm, and one at each of the other two thicknesses. Previous experience with fracture testing of this adhesive in Chapter 2 showed a very good repeatability. The strong dependence of $G_c^s$ on bondline thickness was due to the large plastic zone size at the tip of the fully developed crack in this highly toughened epoxy adhesive. This is discussed further in the next chapter.

![Fig. 9](image-url)  
**Fig. 9** Effect of bondline thickness on the quasi-static steady state critical strain energy release rate, $G_c^s$. Error bars represent ±1 standard deviation. The black line is the least-squares fit.

### 4. Conclusions

The effect of bondline thickness on fatigue behavior became more pronounced as the applied $G$ and the crack growth rate increased. The crack growth rate was more sensitive than the fatigue threshold to changes in the adhesive thickness.

Under mixed-mode loading, a very small effect of bondline thickness on the mixed-mode fatigue threshold was observed. This observation may simplify the design of adhesive joints for fatigue loading.
The critical strain energy release rate for quasi-static fracture increased linearly with bondline thickness from 130-780 µm.
5. References
Chapter 7

Effect of Bondline Thickness –
Part II: Analysis and Finite Element Modeling

1. Introduction

An understanding of the effect of bondline thickness on the fracture and fatigue of adhesive joints can guide the selection of an optimal bondline thickness and indicate the sensitivity to variations in thickness that may arise during production. Chapter 6 presented fatigue and fracture data as a function of bondline thickness for a highly-toughened epoxy adhesive bonding aluminum adherends. The present paper analyzes these data and proposes several explanations for the observed trends.

The trends observed in the literature for the effect of bondline thickness on fatigue and fracture of adhesive joints were explained in Chapter 6 and ref. [1] and are only summarized here. Under quasi-static loading, some investigators have observed a significant strength improvement in adhesive joints with increasing adhesive thickness [6,12], while others have reported no significant effect [4,7], or even a decrease [6,11] in strength. It has also been reported that $G_c^s$, the quasi-static steady-state critical strain energy release rate, initially increases with bondline thickness and then decreases, eventually reaching a constant value [4,8,9]. In general, these trends have been attributed mainly to the relative size of the plastic zone ahead of the crack and the bondline thickness, with the stress and plastic zone size being affected by the adherend restraint [4,8-11]. For example, Hunston et al. [12] related the fracture energy of a toughened epoxy adhesive to the formation and development of the damage zone in the adhesive layer. They explained the increase in fracture toughness with increasing bondline thickness in terms of the expansion of the stress whitened zone ahead of the crack tip, indicating growth in the fracture damage zone. Stress whitening or crazing is a typical deformation mechanism that can occur when the polymer is stretched near its yield point [13]. Daghiyani et al. [14] showed that the increase of the mode-I fracture toughness of compact tension specimens with increasing bondline thickness corresponded to a decrease in the constraint at the crack tip and a corresponding increase in the plastic deformation around the crack tip. This caused the fracture
energy to increase, approaching the value of the bulk adhesive [14].

The majority of the literature concerning the effect of bondline thickness has dealt with the quasi-static fracture of adhesive joints. Fatigue crack growth rates have usually been measured only under mode-I loading and were found to decrease with increasing adhesive thickness [6,10,15,16]. Xu et al. [10] proposed a mechanism similar to that suggested for fracture loading [8,12]; i.e. that the improvement in the mode-I fatigue behavior was due to the effect of reduced adherend constraint causing plastic zone elongation. This was consistent with their observation of a larger effect of bondline thickness when the adhesive was toughened. However, Mall and Ramamurthy [15] found that fatigue crack growth rates in mode I tests did not change when the bondline thickness was increased from 0.102 mm to 0.254 mm, and decreased with a bondline thickness of 0.508 mm only at higher crack growth rates. At lower crack growth rates, near the threshold, the fatigue resistance was the same for all three bondline thicknesses.

There have been relatively few papers dealing with the effect of bondline thickness under mixed-mode cyclic loading. Since most practical joints are loaded under mixed-mode conditions, this represents a significant gap in the literature. Moreover, mixed-mode crack paths tend toward the more highly-strained adherend, and so the interaction of a plastic zone and the adherends will be different than in mode I where the crack lies in the middle of the adhesive layer. In contrast to most of the published mode I fatigue test data, Schmueser [17] measured an increase in the cyclic debonding rate of mixed-mode steel cracked lap shear joints with increasing bondline thickness. The crack path in these joints was interfacial.

Chapter 6 presented data concerning the effect of bondline thickness on the fatigue (fatigue threshold and crack growth rates) and fracture (crack initiation and steady-state critical strain energy release rates) of a single-part highly toughened epoxy adhesive. Under mode-I loading, it was found that the fatigue threshold energy release rate, $G_{th}$, decreased for very thin bondlines, while under mixed-mode loading, $G_{th}$ changed very little with bondline thickness. In both cases, the effect of bondline thickness was more pronounced at higher crack growth rates. For quasi-static fracture, the effect of adhesive thickness on the crack initiation strain energy release rate, $G_c^i$, was similar to that found for the fatigue crack growth rates; however, the steady state energy release rate, $G_c^s$, increased linearly with increasing bondline thickness. The present paper explains these observations using finite element analyses of the stresses in the vicinity of the crack tip.
2. Finite Element Modeling

The DCB and asymmetric DCB (ADCB) joints of Fig. 1 were modeled using finite elements. The adhesive was modeled with a multi-linear stress-strain curve derived from tensile tests (Fig. 2, $E_a=1.5$ MPa), while the AA6061-T6 adherends were assumed to behave as an elastic-perfectly-plastic material with tensile elastic modulus and yield stress values of: $E=68.9$ GPa, $\sigma_y=270$ MPa. The two-dimensional (2D) structural model used plane182 elements (ANSYS® 12, Ansys Inc., Canonsburg, PA) with plane stress assumptions for the substrate and plane strain for the adhesive (Fig. 3). Solid185 brick elements were used for the three-dimensional (3D) FE model, for both the adhesive and adherend. In both the 2D and 3D models, singular elements were used to model the crack tip. In locating the crack in the adhesive layer, the experimentally observed crack path was used; i.e. the mid-plane of the adhesive for DCB joints (mode-I loading), and nearer to the more highly-strained adherend for ADCB joints, due to the mixed-mode loading condition. For ADCB joints, the residual adhesive thickness was measured to identify the crack path as described in Section 3.2.

![Diagram](image1.png)

Fig. 1 Geometry of (a) DCB (mode I) and (b) ADCB (mixed-mode) specimens. All dimensions in mm. Specimens are 240 mm long and 19 mm wide. Not to scale.
Fig. 2  Multi-linear model for the stress-strain behavior of the tested adhesive.

Fig. 3  Two-dimensional finite element model of a DCB specimen with a magnification of the adhesive elements in the crack tip region.
3. Effect of Bondline Thickness on Fatigue Behavior

3.1. Effect of Bondline Thickness on Mode-I Fatigue Behavior

The measured $G_{th}$ for DCB joints at bondline thicknesses $t=380 \, \mu m$ and $790 \, \mu m$ showed no statistically significant difference ($t$-test, 95% confidence; Table 1). However, at $t=130 \, \mu m$, $G_{th}$ decreased significantly and the fatigue crack growth rates were significantly higher compared with the two thicker bondlines, particularly at larger applied strain energy release rates (Fig. 4(a)). This section compares these experimental observations with the corresponding crack tip stresses and damage zone sizes as predicted by the 2D and 3D FE models.

Table 1  Effect of bondline thickness on the measured fatigue threshold, $G_{th}$, quasi-static crack initiation, $G_c^i$, and quasi-static steady-state critical strain energy release rate, $G_c^s$, for aluminum DCB and ADCB joints. $N$ is the number of repetitions. All strain energy release rates are in J/m$^2$. “SD” is the standard deviation.

<table>
<thead>
<tr>
<th>$t$ (µm)</th>
<th>Fatigue DCB (0°)</th>
<th>ADCB (18°)</th>
<th>Fracture DCB (0°)</th>
<th>ADCB (18°)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$G_{th} \pm SD$</td>
<td>$G_{th} \pm SD$</td>
<td>$G_c^i \pm SD$</td>
<td>$G_c^i \pm SD$</td>
</tr>
<tr>
<td>130</td>
<td>92±11</td>
<td>3</td>
<td>114±6</td>
<td>3</td>
</tr>
<tr>
<td>380</td>
<td>195±3</td>
<td>3</td>
<td>121±12</td>
<td>6</td>
</tr>
<tr>
<td>790</td>
<td>194±16</td>
<td>3</td>
<td>132±8</td>
<td>3</td>
</tr>
</tbody>
</table>

Using the 2D model, Fig. 5 shows the opening stress distribution ahead of the crack in the mid-plane of the bondline for the mode-I DCB joint. To compare the finite element results with those available in the literature, the opening stress was non-dimensionalized by the proportional limit of the adhesive, $\sigma_p=30$ MPa, and plotted against log ($x$), where $x$ is the distance from the crack tip. The piecewise linear approximation of Fig. 2 makes the proportional limit a better defined choice than the 0.2% yield stress which was approximately 32 MPa. As expected, the opening stress ($\sigma_{yy}$) exhibited a $x^{-1/2}$ dependence, with the stress growing rapidly at points closer to the crack tip [14,18]. Beyond approximately 1 mm from the crack tip, the opening stress was very similar for the three bondline thicknesses, becoming compressive at about $x=5$ mm (Fig. 5). Figure 5 shows that at the same $G$ in mode I the opening stress near the crack tip increased markedly as the adhesive thickness became smaller, similar to the trend reported by Daghiyani et al. [14].
Fig. 4 Experimental data for the effect of bondline thickness on fatigue crack growth rate at different crack speeds for (a) mode-I loading and (b) mixed-mode loading. The linear curve at each bondline thickness is the average of the curve fit of the Paris law (linear) region of each tested specimen.
Fig. 5 Effect of bondline thickness on the opening stress distribution a distance \( x \) ahead of a crack in a DCB joint at \( G=100 \) J/m\(^2\).

The dependence of the von Mises stress on the bondline thickness was similar to that of the opening stress, as seen in Fig. 6 (2D FE model). The values of \( G \) in these calculations were chosen to be close to \( G_{th} \) at \( t=130 \) \( \mu \)m (100 J/m\(^2\)), close to \( G_{th} \) at \( t=380 \) and 790 \( \mu \)m (200 J/m\(^2\)) and approximately the highest tested \( G \) in the fatigue crack growth rate measurements (1,000 J/m\(^2\)). The maximum von Mises and opening stress did not always occur at the crack tip and a local maximum stress could be observed ahead of the crack tip [19], particularly with the thinner bondlines and at higher loads. At \( G=100 \) and 200 J/m\(^2\), the stress distributions for \( t=380 \) and 790 \( \mu \)m were relatively similar compared with that for \( t=130 \) \( \mu \)m where the von Mises stress was much larger. At \( G=1,000 \) J/m\(^2\), the differences between the stress distributions in the two thicker bondlines became larger (Fig. 6(c)). This may explain why the mode-I \( G_{th} \) was the same at \( t=380 \) and 790 \( \mu \)m while it was approximately half as large at \( t=130 \) \( \mu \)m (Table 1). It is also consistent with the trends of Fig. 4(a), where \( da/dN \) was always much higher for \( t=130 \) \( \mu \)m, and a difference between the crack growth rates for \( t=380 \) and 790 \( \mu \)m was apparent at \( G=1,000 \) J/m\(^2\), but not at \( G=200 \) J/m\(^2\).

The values of the maximum von Mises stress, at or just ahead of the crack tip in the mid-plane of the bondline obtained using the 3D FE model at \( G=200 \) J/m\(^2\) are shown in Fig. 7. In
Fig. 7, the maximum stress was located ahead of the crack tip for $t \leq 380 \, \mu m$. It is seen that this stress increased markedly for bondline thicknesses less than approximately 380 $\mu m$, suggesting that the crack propagation rate would increase as $t$ decreases for a given $G$, which is consistent with the experimental data for $G_{th}$ in Table 1 and $da/dN$ in Fig. 4(a).

![Graph showing stress vs. distance from crack tip](image)
Fig. 6  Effect of bondline thickness on the von Mises stress distribution ahead of a crack in a mode-I DCB joint at (a) $G=100 \text{ J/m}^2$, (b) $G=200 \text{ J/m}^2$ and (c) $G=1,000 \text{ J/m}^2$. 2D FE model.

Fig. 7  Effect of bondline thickness on the maximum von Mises stress at or just ahead of the crack.
crack tip in the mid-plane of a DCB joint at $G=200 \text{ J/m}^2$. 3D FE model.

The stress elevation due to the decrease in the adhesive thickness is due to the constraint imposed by the adherends [14]. The 3D FE model was used to calculate the triaxiality factor (mean stress divided by the von Mises stress [20]) as a measure of constraint at the crack-tip singular nodes across the 10 mm half-width of the joint for applied $G$ values of 200 and 400 J/m$^2$ and the three bondline thicknesses of interest (Fig. 8). The triaxiality factor changed only very slightly with the location of the selected node around the crack tip. It is seen from Fig. 8 that the triaxiality factor, and thus the lateral constraint on the crack tip, increased from the edge of the joint, becoming constant at approximately 20% of the half-width from the edge (2 mm). As expected, the triaxiality factor increased as the bondline became thinner, implying higher crack tip stresses, particularly for $t=130 \ \mu\text{m}$. Comparing Figs. 8(a) and 8(b) illustrates that the effects of bondline thickness were more pronounced as $G$ increased, although in both cases the two thicker bondlines behaved similarly when compared with $t=130 \ \mu\text{m}$. This suggests that the size of the plastic region ahead of the crack (damage zone) at $G=200 \ \text{J/m}^2$ was too small to be affected by the adherend constraint when $t \geq 380 \ \mu\text{m}$; however, when the bondline thickness was 130 \mu m, the damage zone became constrained by the adherends and the triaxiality increased markedly. At $G=400 \ \text{J/m}^2$ (Fig. 8(b)), the damage zone was large enough to begin to be influenced by the adherend constraint at $t=380 \ \mu\text{m}$, as seen by the growing difference between the triaxiality factor at this thickness and at $t=790 \ \mu\text{m}$. As mentioned previously, it is postulated that the elevated crack tip stresses resulting from this increased constraint were responsible for the decreased fatigue performance seen with the thinnest adhesive layer when loaded in mode I.

The previous observations indicate that as $G$ increases the damage zone becomes larger so that adherend constraint becomes more pronounced at the crack tip and the adhesive stress triaxiality increases. Therefore, it is expected that the stress levels in the crack tip region will increase more significantly with applied $G$ once the damage zone thickness approaches the adhesive bondline thickness. This hypothesis is consistent with the trend of Fig. 7.
Fig. 8 Effect of bondline thickness on the triaxiality factor along the width of a DCB joint at (a) $G=200$ J/m$^2$ and (b) $G=400$ J/m$^2$. $Z$ is the distance from the edge of the 20 mm wide joint.
During fracture, the damage zone is typically defined in terms of the plastic zone radius, \( r_p \), usually calculated neglecting the influence of adherend constraint as [21]:

\[
r_p = \frac{1}{2\pi} \frac{E_a G}{\sigma_y^2} \quad \text{for plane stress (1)}
\]

\[
r_p = \frac{1}{6\pi} \frac{E_a G}{\sigma_y^2} \left( \frac{1}{1 - \nu^2} \right) \quad \text{for plane strain (2)}
\]

where \( E_a \), \( \sigma_y \) and \( \nu \) are elastic modulus, yield stress and Poisson ratio of the adhesive, respectively, and \( G \) is the applied strain energy release rate. As mentioned previously, in most of the cases in the present study, the damage zone was affected by the adherend constraint; therefore, these equations were not appropriate and so the damage zone size was calculated using the 2D and 3D finite element models from \( G = 200 \text{ J/m}^2 \) to \( G = 1,000 \text{ J/m}^2 \) in 100 \text{ J/m}^2 steps. The crack tip damage zone was defined as the adhesive volume in which the von Mises stress was larger than some assumed level. Such a damage zone definition, which ignores micro-crack formation, has been used previously to model the fracture behavior of toughened epoxies [8]. The stress limit for the damage zone was considered equal to the proportional limit of the adhesive, \( \sigma_p = 30 \text{ MPa} \) (Fig. 2). The prediction of the 3D model in the middle of the joint width was the best single representation, since that level of triaxiality existed across approximately 80% of the joint width for all bondline thicknesses (Fig. 8). The damage zone area at the free edges of the joints was approximately 1/3 larger than in the middle due to the reduced constraint. The damage zones predicted by the 2D plane strain model were on average only 12% smaller than those in the middle of the 3D joint width, but the 2D plane stress zone sizes were on average 40 times larger.

The damage zone shapes predicted in the middle of the 3D FE model for \( G = 200 \text{ J/m}^2 \) under mode-I and mixed-mode loading are shown in Figs. 9(a) and 9(b). In these cases, where the bondline thickness was large enough to not influence the relatively small damage zones, the shape, but not the size, was independent of the applied \( G \) or the assumed stress limit defining the damage zone. In contrast, Fig. 9(c) illustrates the shape when \( G \) was increased to 1,000 \text{ J/m}^2 to create a damage zone that was influenced by the adherends. Figure 9(d) illustrates that the stress at the aluminum-adhesive interface can reach the stress limit of the damage zone before the damage zone occupies the entire bondline thickness. This is due to the stress concentration at the interface resulting from the sudden change in the elastic modulus. It was found that once the
situation of Fig. 9(d) was reached, a relatively small further increase in $G$ produced a single continuous damage zone extending from one interface to the other.

(a)

(b)

Thinner arm of ADCB

Thickness of damage zone upper half

Thickness of damage zone lower half

Thicker arm of ADCB

(c)

Crack tip
Fatigue crack growth occurs due to damage accumulation; therefore, any parameter that increases damage in the adhesive layer will expedite fatigue crack formation and development, thereby increasing crack growth rates. Figure 10 shows how the thickness and average length of the damage zone change with $G$ for the three bondline thicknesses (3D FE model). In this case the damage zone was taken to be the continuous area around the crack tip where the stress exceeded the proportional limit; i.e. neglecting the disconnected zones that could exist at the interfaces (Fig. 9(d)). The average damage zone length was defined as the damage zone area divided by the damage zone thickness. As predicted by Eqs. (1) and (2), the damage zone thickness increased linearly with $G$ (Fig. 10(a)); however, at a certain $G$ there was a pronounced increase in this rate of growth (i.e. slope in Fig. 10(a)) which corresponded to the stress at the aluminum-adhesive interface reaching the proportional limit of the adhesive (situation of Fig. 9(d)). This transition is evident between 200 and 300 $J/m^2$ for $t=380 \, \mu m$ and around 500 $J/m^2$ for $t=790 \, \mu m$ (Fig. 10(a)). The value of $G$ corresponding to this point where the adherend constraint first becomes evident will be termed $G_{\text{const}}$. No transition was observed for $t=130 \, \mu m$, because even at $G=100 \, J/m^2$ the finite element analysis showed that the stress at the aluminum-adhesive interface reached the proportional limit of the adhesive.
At each bondline thickness, for $G < G_{\text{const}}$ the mode-I fatigue behavior should remain unaffected by the bondline thickness, and the effect of adhesive thickness should thus become noticeable only when $G > G_{\text{const}}$. This is consistent with the trends seen in Fig. 4(a), where the effect of bondline thickness on $da/dN$ increases with increasing $G$ and decreasing thickness, and the behavior of the curves for $t=380 \mu m$ and $790 \mu m$ diverges at approximately 250 J/m$^2$, the value of $G_{\text{const}}$ for $t=380 \mu m$. It is also consistent with the fact that $G_{th}$ was the same at $t=380 \mu m$ and $790 \mu m$, that $G_{th}$ was smaller at $t=130 \mu m$, and that the damage zones at $G=100$ and 200 J/m$^2$ for $t=130 \mu m$ were longer than those for the two thicker bondlines (Fig. 10(b)).

In summary, fatigue crack growth is due to damage accumulation which depends on the stress level and the bondline thickness (volume) in which the damage accumulates. A decrease in the bondline thickness elevated the stress for a given $G$ and enlarged the relative size of the damage zone compared to the bondline. The increased stress and larger relative damage zone size caused a greater adhesive layer fatigue damage accumulation, adversely affecting the mode-I fatigue performance. The following sections will examine the effect of damage zone size and bondline thickness on mixed-mode fatigue and quasi-static fracture.

### 3.2. Effect of Bondline Thickness on Mixed-Mode Fatigue Behavior

A very slight increase in $G_{th}$ with bondline thickness was observed for the mixed-mode ADCB joints (Table 1). The difference between the $G_{th}$ values became statistically significant ($t$-test, 95% confidence) only when the bondline thickness increased from 130 $\mu m$ to 790 $\mu m$. As in the mode-I DCB experiments, the effect of bondline thickness became more pronounced at higher crack growth rates (Fig. 4(b)) (Chapter 6).

The FE results presented in Section 3.1 assumed mode-I loading where the crack propagated in the mid-plane of the adhesive layer. As expected, the increased phase angle in the ADCB joints caused the crack path to move closer to the more highly-strained adherend [22]. This shift in the crack path was quantified by measuring the residual adhesive thickness on the thinner (more highly strained) adherend in the present ADCB joints by using an optical profilometer (Nanovea ST 400, Microphotonics Inc., Irvine, CA, USA) to make line scans across the width of the joint. A datum was provided at the ends of each scan by removing a 2 mm wide band of adhesive using a solvent (mixture of methylene chloride and methyl alcohol; Glue Buster, Kosmic Surf-Pro Inc., St. Amable, Quebec). The average residual adhesive thickness at threshold measured in this manner was 20 $\mu m$ for ADCB joints having $t=380 \mu m$. 

Fig. 10  Effect of $G$ on: (a) the mode-I damage zone thickness normalized by the adhesive thickness, $t$, and (b) the average damage zone length in a DCB joint at the three tested bondline thicknesses.  Average damage zone length was calculated by dividing the damage zone area by the damage zone thickness.

Under mode-I loading, since the loading was symmetric and the crack grew in the mid-plane of the bondline, the proportional limit stress was reached at both interfaces simultaneously,
so that the damage zone became affected by both adherends at the same load condition. However, under mixed-mode loading the crack path was closer to the more highly strained adherend, and the damage zone could be affected by only one adherend. In this case, the effect of adherend constraint on the damage zone size would become more significant when the stress at the second interface also reached the proportional limit.

A 3D FE model was used to investigate the effect of $G$ on the thickness of the upper and lower halves of the mixed-mode ADCB damage zone depicted in Fig. 9(b). As with the mode-I FE model, the damage zone was defined as the continuous region about the crack tip in which the von Mises stress reached or exceeded the proportional limit of the adhesive, $\sigma_p=30$ MPa. In order to separate the effects of constraint from those of the mixed-mode loading, the crack was initially located in the mid-plane, and the bondline thickness was chosen to be sufficiently large ($t=2$ mm) that adherend constraint did not affect the damage zone. Figure 11 shows that the predicted upper damage zone thickness was larger than the measured 20 $\mu$m residual adhesive thickness for all $G>100$ J/m$^2$; however, the lower part of the damage zone was only about 60 $\mu$m thick in the measured range of $G_{th}$ from 114 – 132 J/m$^2$ (Table 1). Consequently, the lower damage zone can be assumed to remain unaffected by the constraint of the opposing thicker adherend in all cases. It is recognized that Fig. 11 underpredicts the damage zone thickness when it approaches the bondline thickness, because of the neglect of constraint effects. However, the FE results confirmed that at threshold, even at $t=130$ $\mu$m, the von Mises stress at the lower interface was indeed smaller than the proportional limit; i.e. the fatigue threshold damage zone did not reach the lower adherend as in Fig. 9(d) and was affected only by the upper adherend (Fig. 12). The fact that the damage zone intersected only the upper (thinner) adherend and did not extend to the lower (thicker) adherend was consistent with the experimental results, which showed an insignificant effect of bondline thickness on the mixed-mode $G_{th}$ results (Table 1). In other words, the degree of constraint was the same at $G_{th}$ for the three tested bondline thicknesses and therefore no differences would be expected. As $G$ and the crack propagation rate increased, the lower portion of the damage zone expanded, eventually reaching the lower adherend, as per Fig. 11. At this point the lower adherend constraint elongates the damage zone and elevates the stresses in the adhesive layer as was described for mode-I DCB joints in Section 3.1. As a result, as the applied $G$ increases beyond $G_{th}$, the effect of adherend constraint and bondline thickness on the fatigue behavior becomes larger. This is consistent with the experimental $da/dN$ results at higher $G$ levels seen in Fig. 4(b).
Fig. 11  Effect of strain energy release rate, $G$, on the thickness of the upper and lower halves of the damage zone in a hypothetical ADCB joint with $t=2$ mm. The region between the crack tip and the thinner arm of the ADCB was called “upper half”, and the region between the crack tip and the thicker arm of the ADCB was called “lower half” of the damage zone (Fig. 9(b)).

Table 1 shows that increasing the phase angle from 0° to 18° decreased the $G_{th}$ significantly. This is opposite to the experimental results for a different aluminum adhesive system in Chapter 3 in which the phase angle had a negligible effect on $G_{th}$. This apparent
contradiction is explained by the observation that the mixed-mode crack path in Chapter 3 was much farther from the thinner adherend than that in the present ADCB joints. Consequently, the effect of adherend constraint was not evident in Chapter 3 so that DCB and ADCB specimens produced the same $G_{th}$. However, the crack path in the present ADCB joints was much closer to the thinner adherend, and as a result the increased constraint produced a significantly smaller $G_{th}$.

4. Effect of Bondline Thickness on Fracture Behavior

The finite element model was used to study the fracture behavior from crack initiation, at the strain energy release rate, $G_c^i$, through the rising part of the R-curve to the steady-state critical strain energy release rate, $G_c^s$ as measured in Chapter 6. The critical strain energy release rate depends on the damage zone size [4,8]. After the first instance of crack initiation and propagation at $G_c^i$, the damage zone and micro-cracks in the crack tip region start to form and develop. This development of the damage zone with crack propagation causes the required applied strain energy release rate, $G_c$, to increase until the damage zone is fully developed to a steady-state condition at $G_c^s$. Such an R-curve behavior was shown in Chapter 6 to occur for the present adhesive system in an aluminum ADCB joint with a bondline thickness of 380 µm. The 3D FE model was used to calculate the damage zone area in the middle of the joint width for a selection of the measured forces and crack lengths from Chapter 6. A damage zone defined on the basis of the adhesive proportional limit stress is probably smaller than the actual damage zone that includes other damage such as micro-cracking. Nevertheless, Fig. 13 shows that the predicted damage zone development with crack length follows the observed typical R-curve trend, reaching a steady-state size after approximately 10 mm of crack growth.

4.1. Effect of Bondline Thickness on Crack Initiation

Crack initiation at $G_c^i$ represents the first instant of crack growth under quasi-static loading, and corresponds to the smallest damage zone (Fig. 13). Therefore, as with fatigue, crack initiation can be adversely affected by any parameter that enlarges the damage zone, indicating the elevation of stress at a constant applied $G$.

Mode I

As shown in Section 3.1, the adherends provided constraint to the mode-I damage zone at $G$ values of 200-300 J/m$^2$ when $t$=380 and 400-500 J/m$^2$ for $t$=790 µm. Since the measured mode-I $G_c^i$ was 438±83 J/m$^2$ at $t$=790 µm (Table 1), the effect of adherend constraint on the
damage zone was likely very small. However, at \( t=380 \, \mu \text{m} \) the damage zone was influenced by adherend constraint resulting in the lower \( G_c^i = 344\pm50 \, \text{J/m}^2 \). At \( t=130 \, \mu \text{m} \), adherend constraint increased the damage zone stresses causing initiation markedly, and as a result a significantly lower \( G_c^i \) was measured (184\pm62 \, \text{J/m}^2).

Fig. 13  Change in the damage zone area, \( A_d \), and the critical strain energy release rate, \( G_c \), with crack length under quasi-static loading for an ADCB joint with \( t=380 \, \mu \text{m} \). Damage zone area was calculated from the 3D FE model and in the middle of the joint width. The crack tip in the FE model was placed at the experimentally measured crack location in the bondline. \( A_d \), and \( G_c \) normalized by their respective maximum values.

**Mixed-mode**

As mentioned in Chapter 6, crack initiation measurements were made after the fatigue threshold measurements on the same specimens. Therefore, the crack tip location for initiation measurements was determined by the location of the fatigue threshold crack. Under mixed-mode loading, it was seen that the crack moved toward the more highly-strained adherend; i.e. the upper arm of the ADCB in Fig. 1. As a result, and similar to the argument regarding the effect of \( t \) on the mixed-mode \( G_{th} \) in Section 3.2, the upper half of the stress in the damage zone was elevated by the partial constraint of the upper adherend for the three tested bondline thicknesses (Fig. 11). This explains the lower \( G_c^i \) for ADCB compared to DCB specimens (Table 1). At \( G=300 \, \text{J/m}^2 \) (close to \( G_c^i \) of ADCB at \( t=380 \, \mu \text{m} \) and 790 \, \mu \text{m}), the lower half of the damage zone remained unaffected by the lower adherend, and the level of constraint was very similar for both
\( t = 380 \, \mu m \) and \( 790 \, \mu m \) resulting in values of \( G_c^i \) that were not significantly different. However, decreasing adhesive thickness to \( t = 130 \, \mu m \) caused the constraint induced by the lower adherend to influence the damage zone thereby decreasing \( G_c^i \) to 163 J/m\(^2\) (Fig. 11).

### 4.2. Effect of Bondline Thickness on the Steady-State \( G_c \)

For the current range of bondline thicknesses, \( G_c^s \) increased linearly with \( t \) (Table 1 and Chapter 6). Figure 14 shows how the damage zone area in the middle of the specimen width changed with bondline thickness using the 3D FE model at \( G = 600 \, J/m^2 \). As will be discussed later, the trend shown in Fig. 14 was independent of the selected \( G \). For relatively thin bondlines, the damage zone was fully restricted by the adherends; i.e. as seen in Fig. 15, the relative damage zone thickness, \( t_d/t \), was equal to 1. As the adhesive thickness increased to 600 \( \mu m \), the damage zone, which was still constrained by the adherends, could grow, resulting in an increase in the damage zone thickness, \( t_d \). The damage zone length also decreased due to the decrease in the stress (Figs. 5, 6 and 7); however this decrease was much smaller than the linear increase in \( t_d \) with bondline thickness. This condition continued until \( t \) exceeded 600 \( \mu m \) when the damage zone was not fully restricted by the adherends; i.e. it no longer occupied the entire bondline thickness. From this point on, the damage zone area decreased with \( t \) because the damage zone length and thickness decreased. For \( t > 1 \, mm \) the adhesive constraint played no role on the size of the damage zone at \( G = 600 \, J/m^2 \), and thus the damage zone area remained relatively constant. Since the damage zone size depends on the applied \( G \), the shape, but not the trend, of Fig. 14 would change if a different \( G \) was assumed.

The fracture energy in toughened epoxies depends on the damage development in the adhesive [4,8]. The change in the damage zone area with bondline thickness, \( t \), in Fig. 14 is similar to how \( G_c^s \) changes with \( t \) in structural toughened adhesives [8], which results in an optimum bondline thickness which maximizes \( G_c^s \). It is also consistent with Hunston et al.’s [12] experimental results for the change in the size of the stress whitened zone with bondline thickness. As the toughness of an adhesive increases, the damage zone at \( G_c^s \) enlarges, and thus a larger optimum bondline thickness should be expected.

Consistent with measuring a higher \( G_c^s \) with increasing adhesive thickness for the range of bondline thicknesses tested in the current work (Table 1 and Chapter 6), the damage zone area, \( A_d \), at \( G_c^s \) also increased with the bondline thickness, as shown in Fig. 16. This is consistent with the above discussions on the relation between fracture toughness and damage zone size.
Fig. 14 Change of damage zone area with bondline thickness for a mode-I DCB at $G=600 \text{ J/m}^2$. Damage zone area was calculated from the 3D FE model in the middle of the joint width.

Fig. 15 Change in the damage zone thickness, $t_d$, relative thickness, $t_d/t$, and damage zone length, $l_d$, with bondline thickness, $t$, for a mode-I DCB joint at $G=600 \text{ J/m}^2$. 
Fig. 16  Change in the damage zone area, $A_d$, with bondline thickness at the experimentally measured mixed-mode $G_c^s$. $A_d$ and $G_c^s$ are non-dimensionalized by their respective maximum values at $t=790 \mu$m.

5. Conclusions

Finite element modeling was used to study the fatigue and fracture behavior of a highly toughened epoxy bonding as a function of the bondline thickness. The fatigue studies were performed at a broad range of crack growth rates, including the fatigue threshold, under both mode I and mixed-mode conditions.

In general, at a given strain energy release rate $G$, a decreasing bondline thickness tended to increase the stress triaxiality and elevate the stresses near the crack tip. This caused the damage zone thickness and length to increase, resulting in greater fatigue damage accumulation at a given $G$ and poorer fatigue performance for thinner bondlines.

As the applied $G$ increased beyond the threshold, the effect of the bondline thickness on the fatigue behavior became more pronounced as the damage zone became thicker and the stress levels were affected by adherend constraint.

The measured mode-I fatigue threshold damage zone was too small to be affected by the adherends when the bondline thickness, $t$, was greater than 380 $\mu$m, and therefore $G_{th}$ remained constant for $t \geq 380 \mu$m.

Under mixed-mode loading, since the fatigue crack path was very close to the more
highly-strained adherend, the damage zone at threshold was constrained by this adherend equally for the three measured bondline thicknesses. As a result, an insignificant effect of bondline thickness on $G_{th}$ was observed. The effect of adhesive thickness became more pronounced as the mixed-mode crack growth rate increased and the damage zone enlarged to the point where it was constrained by the opposite adherend. At this point, cracks in the 130 $\mu$m bondline mixed-mode joints began to grow much faster than in the thicker adhesive layers.

The change in the damage zone size with bondline thickness also explained the effect of adhesive thickness on the initiation and steady-state critical strain energy release rates. An optimum adhesive thickness for fracture loading of toughened adhesive joints was determined based on the maximization of the damage zone size.
6. References


Chapter 8

Effect of Surface Roughness

1. Introduction

Bonding surfaces are often abraded to remove weak or contaminated areas and to increase mechanical interlocking and the effective bonding area. For example, grit blasting can produce a significant improvement in joint strength on aluminum surfaces, although bond strength did not continue to increase with further roughening [1,2]. In contrast, Shahid and Hashim [3] found a linear relation between surface roughness and the cleavage strength of steel adhesive joints. Zhang et al. [4] measured a continuous increase in the interfacial fracture resistance of aluminum layered double cantilever beam joints bonded using an epoxy adhesive. Carbon/epoxy composite single lap shear joints also showed a continuous increase in the bond strength with adherend roughness [5]. The tensile strength of aluminum lap shear joints bonded using an epoxy resin showed an increase with roughness, but reached a constant value at an average roughness, $S_a$, of 3 µm [6]. Steel tubular single lap joints showed the highest static shear strength at $R_a$ between 1.5 µm and 2.0 µm, becoming weaker as the bonding surfaces were made rougher still [7]. Uehara and Sakuri [8] found different levels of optimum roughness for the tensile strength of epoxy and cyanoacrylate adhesive joints bonding steel. They also conducted shear and peel tests, finding that the effect of roughness was smaller in shear and disappeared altogether in the peel tests [8].

Surface roughness was also found to influence the fatigue life of steel tubular single lap joints subjected to a cyclic torque [9]. The fatigue strength increased up to an average roughness, $R_a$, of 1.5-2 µm, and then decreased suddenly as the roughness was increased to 3 µm. The failure surface at $R_o=0.6$ µm was fully interfacial, but was partially cohesive at $R_d=2.0$ µm. Similar torsional fatigue tests have yielded a range of optimum roughness values depending on the adhesive system [7,10]. The stress analysis of Kwon et al. [10] suggested that the optimum roughness depends on the adhesive layer thickness. The fatigue threshold, $G_{th}$, of aluminum cracked lap shear joints was increased by more than 80% as the adherend roughness was increased from $R_o=0.77$ µm to 1.33 µm(Chapter 4 and ref. [11]). This was attributed to the
increase in the surface area and the change in the failure mode from interfacial at $R_a=0.77$ µm to cohesive at $R_a=1.33$ µm.

A wide variety of explanations have been proposed for the positive and negative effects of increasing surface roughness: an increase in the bonding area [3-6,8,12], increased mechanical interlocking [5], a reduction in adhesive wettability [6], the deviation of crack path away from a weaker interfacial region [3,5,12,13], and an increase in stress concentration at asperity tips [14]. While there has been considerable work on the effect of surface roughness on the fracture behavior of adhesive joints, relatively few papers have studied the effect of roughness on fatigue behavior, and no published work exists on the relationship between the fatigue threshold and surface roughness. This case is of particular interest, because recent work has shown that fatigue crack paths move even closer to the interface as the crack speed decreases [11,15]. This paper presents experimental data concerning the effect of surface roughness on the fatigue threshold and fatigue crack growth rates of aluminum-epoxy joints under both mode I and mixed-mode loading. The results are compared with quasi-static mixed-mode fracture measurements of both the crack initiation and steady-state strain energy release rates.

2. Experimental Approach

2.1 Specimen Preparation

A highly-toughened single-part epoxy adhesive was used to bond aluminum double cantilever beam (DCB) and asymmetric double cantilever beam (ADCB) specimens, which were used for mode I and mixed-mode loading, respectively. The geometries of the joints are shown in Fig. 1. Specimens were fabricated from 12.7 mm×19.05 mm (1/2”×3/4”) and 25.4 mm×19.05 mm (1/2”×3/4”) AA6061-T651 flat bars. An adhesive bondline thickness of 380 µm was established using spacing wires in the bondline. Prior to bonding, the adherends were roughened as described below, and then washed with running tap water and a cotton cheesecloth, and dried for 30 min at 55 °C. The aluminum bars were then pretreated using the P2 etching process [16]; i.e. degreased with acetone and etched using an aqueous solution of ferric sulfate and sulphuric acid. Prior to applying the adhesive, the aluminum bars were rinsed using distilled water and dried at 55°C for 30 min. In order to promote wetting, the adhesive viscosity was reduced prior to application by preheating it to 55°C for 1 h. A thermocouple embedded in the bondline was used to ensure that the adhesive was cured for at least 30 min at 180°C. Excess adhesive was removed from the sides of the joint after curing using a disc sander with water as a coolant,
followed by gentle sanding using a belt sander and 120 grit paper. To improve the visibility of the crack and remove any surface damage, the sides of the joint were finally sanded with 600 grit sandpaper, and a thin coating of diluted white correction fluid was applied. Five different roughnesses were produced as follows:

- Abrading with an orbital sander using a silicon carbide nylon mesh abrasive pad produced an average roughness $R_a = 1.3 \, \mu m$.
- An orbital sander with a P60 grit sandpaper produced an average roughness $R_a = 1.9 \, \mu m$.
- Using grinder discs with grits P80, P36 and P16 resulted in average roughnesses of 3.9 \, \mu m, 6.4 \, \mu m and 9.0 \, \mu m, respectively, measured along the length of the specimen. The grinding discs were held so that the scratches produced in the surface were perpendicular to the length of the specimen.

![Diagram](a)

![Diagram](b)

**Fig. 1** Geometry of (a) DCB (mode I) and (b) ADCB (mixed mode) joints.

A summary of the roughnesses and the approaches used is given in Table 1. The area scans and the surface roughness measurements were performed using an optical profilometer.
Roughness was measured in a longitudinal direction along the bars; i.e. in the crack growth direction. The repeatability of the roughening process was found to be satisfactory among the four samples produced at each roughness. The average roughness calculated from four measurements on any one specimen showed no statistically significant difference (t-test, 95% confidence) with the grand average $R_a$ of the four specimens. The data of Table 1 are for measurements performed on a single specimen and measured prior to the etching. It was found that P2 etch did not significantly change the $R_a$ of the aluminum surfaces (t-test, 95% confidence). Figure 2 shows the two dimensional and three dimensional topographies of the roughened surfaces over a scan area of $10 \times 10$ mm. Remnants of the original microtopography of the extruded aluminum surface can be seen at $R_a = 1.3 \, \mu m$. The orbital sanding using the P60 grit sandpaper ($R_a = 1.9 \, \mu m$) produced a uniform microtopography. The grooves formed by the grinding disc are evident in Figs. 2(c)-2(e).

Table 1  Surface roughness of aluminum bars produced using different roughening approaches. Four measurements were performed for each roughness. “SD” is standard deviation. Roughness measurements were in the longitudinal direction along the bars with a scan length of 15 mm.

<table>
<thead>
<tr>
<th>$R_a \pm SD$ ($\mu m$)</th>
<th>Apparatus</th>
<th>Paper</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.3 ± 0.2</td>
<td>Orbital finisher</td>
<td>silicon carbide nylon mesh abrasive pad</td>
</tr>
<tr>
<td>1.9 ± 0.1</td>
<td>Orbital sander</td>
<td>P60 sandpaper</td>
</tr>
<tr>
<td>3.9 ± 0.2</td>
<td>Grinder</td>
<td>P80 grinding disc</td>
</tr>
<tr>
<td>6.4 ± 0.6</td>
<td>Grinder</td>
<td>P36 grinding disc</td>
</tr>
<tr>
<td>9.0 ± 0.3</td>
<td>Grinder</td>
<td>P16 grinding disc</td>
</tr>
</tbody>
</table>

2.2 Fatigue Testing

All fatigue experiments were carried out at a cyclic frequency of 20 Hz, under displacement control, with a constant displacement ratio, $R = \delta_{\text{min}}/\delta_{\text{max}} = 0.1$. Previous work in Chapter 4 showed that fatigue testing under force and displacement control yields very similar results, but displacement control threshold testing is easier to control and usually requires less time. A dry condition (11% - 15% relative humidity) was achieved by performing the experiments in a desiccant chamber. The fatigue tests began with an applied load producing the highest $G$ of interest, and the crack slowed to the threshold value ($10^{-6}$ mm/cycle) as the displacement was held constant.
Fig. 2 The micrographs and 3D images of the aluminum bars with surface roughnesses of: (a) 1.3 µm, (b) 1.9 µm, (c) 3.9 µm, (d) 6.4 µm and (e) 9.0 µm. The vertical axis in the 3D topographies is in µm. Scan area was 10mm×10mm. The direction of crack growth and roughness measurement in the micrographs was from right to left.

The unloading joint compliance approach [17] was used to measure the fatigue crack length. The joint compliance was measured during the unloading portion of the cycle using the load cell output and a clip gauge attached to the end of the specimen. A CCD camera (2 mm field of view) on a motorized linear stage was used to measure the crack length and relate it to the measured joint compliance for a given specimen type using the approach of ref. [18]. A least squares regression was used to fit a third-order polynomial to the normalized crack length, \(a/w\), versus the normalized specimen compliance, \(CEB\), for fatigue joints:

\[
a/w = c_1 \times (CEB)^3 + c_2 \times (CEB)^2 + c_3 \times (CEB) + c_4 \tag{1}
\]

where \(a\) is the crack length, \(w\) is the specimen length from the loading pins, \(C\) is the compliance, \(E\) is the tensile modulus of the adherends, and \(B\) is the specimen width. \(c_1\) to \(c_4\) are the regression fit constants.

2.3 Fracture Testing

The fracture tests were performed on the aluminum ADCB joints. The same load was applied on both arms of the ADCB to produce a mixed-mode loading condition (\(\psi = 18^\circ\)) at the crack tip [19]. The phase angle is a measure of the mode ratio of loading defined as
\[ \psi = \arctan \left( \sqrt{\frac{G_{II}}{G_I}} \right), \]

where \( G_I \) and \( G_{II} \) are the mode I and II components of the strain energy release rate. The ADCB specimens were loaded with a constant cross-head speed of 1 mm/min. The crack length was measured from the center of the loading pins using a microscope mounted on a micrometer stage with a resolution of 0.01 mm. Crack growth was stable in this system, so that many crack extension events could be recorded with a single ADCB specimen. To measure the critical load at each crack length, the cross-head displacement was started and stopped repeatedly in the vicinity of the expected fracture load (each time at a constant crosshead speed of 1 mm/min) until a drop in the applied load was observed (Fig. 3). Each start-stop cycle lasted for about 3 seconds, with the load increment between each cycle being in the range of 20 – 30 N. This maximum load prior to the drop was taken as the critical fracture load for the measured crack length if visual inspection through the microscope confirmed that the macro-crack had propagated. After measuring the new macro-crack length, the ADCB was unloaded and the same procedure was followed again beginning at the new crack length.

Fig. 3 Schematic illustration of the force vs. displacement during loading in order to determine the quasi-static critical force for fracture tests. Black dots indicate the instances when the actuator was stopped momentarily to determine whether the load dropped at a constant displacement.
2.4 Strain Energy Release Rate Calculation

The strain energy release rate, \( G \), for DCB and ADCB joints was calculated from the measured force and crack length using the developed analytical beam-on-elastic-foundation model in Chapter 3 as follows:

\[
G = 12(Pa)^2(A + B)
\]  

where

\[
A = \frac{1}{2 E_u h_u^3 (1-t_i/h_u)^3} \left[ 1 + 0.667 \left( 1 - t_i/h_u \right)^3 \left( 1 + t_i/h_u (2 E_u/E_a - 1) \right)^{0.25} \frac{h_u}{a} \right]^2
\]

\[
B = \frac{1}{2 E_l h_l^3 (1-t_u/h_l)^3} \left[ 1 + 0.667 \left( 1 - t_u/h_l \right)^3 \left( 1 + t_u/h_l (2 E_l/E_a - 1) \right)^{0.25} \frac{h_l}{a} \right]^2
\]

and \( P \) is the force per unit width, \( E \) is the elastic modulus, \( t \) is the adhesive thickness and \( h \) is the adherend thickness. The subscripts \( a, u \) and \( l \) refer to the adhesive, and the upper and lower substrates, respectively.

As was shown in Chapter 3, the model predicted \( G \) values for aluminum and steel DCB and ADCB joints were within 2\% of those predicted using a two-dimensional elasto-plastic finite element model (Section 3.1.2) for crack lengths of 40-120 mm.

3. Effect of Surface Roughness on Fatigue Behavior

3.1. Mixed-Mode ADCB Specimens

Aluminum ADCB joints were chosen to study the effect of surface roughness under a mixed-mode loading condition (\( \psi = 18^\circ \)). Since the mode II component in an ADCB joint will tend to move the crack path toward the more strained adherend [15] (i.e. the thinner one), it was expected that the fatigue behavior under mixed-mode conditions would be influenced by the surface roughness on the thinner adherend.

The surface roughness had a pronounced effect on the fatigue threshold under the mixed-mode condition, as seen in Fig. 4, where the solid symbols show \( G_{th} \) as calculated in the conventional way using the apparent crack surface area (the product of the specimen width and the crack growth length). The meaning of the open symbols is discussed in the next section. The rising trend, the plateau and the decreasing trend of Fig. 4 were statistically significant (\( t \)-test, 95\% confidence); i.e. proceeding left to right on Fig. 3, the \( G_{th} \) values at \( R_a=1.3 \) \( \mu m \) and \( R_a=1.9 \)
µm were statistically indistinguishable (121±12 J/m² and 144±30 (± standard deviation)); both of these $G_{th}$ values were smaller than the values at $R_a=3.9$ µm and $R_a=6.4$ µm (178±10 J/m² and 178±19 J/m²); and these maximum values of $G_{th}$ were in turn larger than the value for the roughest surface $R_a=9.0$ µm (142±8 J/m²).

In all these experiments the crack path was within the adhesive, but close to the interface of the thinner adherend. This had been verified in earlier fatigue threshold testing with this adhesive same system at $R_a=1.3$ µm using XPS (x-ray photoelectron spectroscopy) surface analysis in Chapter 3.

![Fig. 4 Effect of surface roughness on $G_{th}$ of aluminum ADCB joints. Error bars show ±1 standard deviation. The number of experiments is given above each data point. The solid symbols show $G_{th}$ calculated conventionally using the nominal, apparent crack surface area, and the open symbols show $G_{th}$ calculated using the actual fracture surface area measured using the optical profilometer.](image)

**3.1.1 Bonding Area and Fracture Surface Area**

Increasing surface roughness increases the mechanical interlocking between the adhesive and the adherend and the area available for bonding. These effects were investigated using the optical profilometer to make line scans on the roughened surfaces in the longitudinal and transverse directions with measurement points every 0.5 µm. As with the roughness
measurements, these scans were made on the aluminum bars prior to etching. $L_{rl}$ and $L_{rw}$ were defined as the ratio of the actual profile length to the projected profile length in the longitudinal and transverse directions, respectively. This is similar to the definition of $R_{lo}$ based on ISO 4287-1:1984 [20]. The ratio of the actual surface area to the projected (apparent) profile area, $A_r$, was estimated by multiplying $L_{rl}$ and $L_{rw}$. $R_{lo}^2$, measured in the longitudinal direction, has been used as a measured of the change in the bonding area with surface roughness [3].

Figure 5 shows that $L_{rl}$, $L_{rw}$ and $A_r$ increased with increasing $R_a$, and did not display the maximum seen in the $G_{th}$ vs $R_a$ trend of Fig. 4. Therefore, although the increase in bonding area may contribute to the initial increase of $G_{th}$ with $R_a$, it cannot explain the existence of a maximum.

![Figure 5](image-url)

Fig. 5 $L_{rl}$, $L_{rw}$ and $A_r$ as a function of $R_a$. Points are the average of four measurements for a scan length of 15 mm. The standard deviation was approximately 4% of the average for measurements at each $R_a$.

When the crack is very close to the interface, such as in the present mixed-mode case, an increase in the surface roughness can force the crack path to become more three-dimensional as it encounters asperities, thereby increasing the actual fracture surface area and possibly pinning the crack, retarding its advance. Both effects would increases the apparent strain energy release rate required to grow the crack. However, these effects should also be proportional to $L_{rl}$, $L_{rw}$ and $A_r$, and therefore increase monotonically with increasing $R_a$. Hence, although it may
contribute to the observed increase in $G_{th}$ with the smaller $R_a$ (Fig. 4), it also does not explain the observed maximum.

It was of interest to determine how much of increase in $G_{th}$ was due to an increase in the actual fracture surface area compared with the apparent area (i.e. width of the specimen times the crack extension). The actual fracture surface area was measured using the optical profilometer to obtain $L_{rl}$ and $L_{rw}$ on the residual adhesive on the fracture surface in the threshold region of a specimen. For each tested specimen three line scans were performed at threshold along the width and length of the specimen. $A_r$ was calculated from the average $L_{rl}$ and $L_{rw}$. For each $R_a$, $G_{th}$ was then recalculated using $A_r$ instead of the apparent crack area as shown in Fig. 4. As expected, $G_{th}$ decreased because the actual fracture area increased, and the effect of $R_a$ became smaller, but did not disappear. For instance, the previously observed 55% increase in $G_{th}$ by increasing $R_a$ from 1.3 µm to 3.9 µm, was reduced to 32% when the actual fracture area was used. Therefore, the effect of increasing roughness may be partially due to the change in the fracture surface area, but other factors such as crack pinning must also play a role.

### 3.1.2 Crack Path

Figure 6 compares the fracture surfaces on the thinner arm of the aluminum ADCB joints for $R_a=1.3$ µm and $R_a=6.4$ µm. It is seen that increasing the roughness increased the amount of residual adhesive; i.e. resulted in a more cohesive failure. This observation is consistent with other literature results where an improvement in the fracture behavior due to an increase in surface roughness was attributed to the deflection of the crack path away from the interface and a consequent increase in the residual adhesive thickness [13,14]. It has been proposed that this is caused by the stress concentrations at the tips of asperities generating a crack path within the adhesive that is farther from the average adhesive-adherend interface [12-14]. Furthermore, increasing roughness makes the abrupt transition in elastic modulus more gradual as the asperities transfer load to the adhesive, thereby decreasing the average stress concentration for a crack propagating in a path at the tips of the asperities [12]. Both of these effects would tend to increase $G_{th}$ with increasing $R_a$, but they still do not lead to a maximum followed by a decrease in $G_{th}$ with increasing $R_a$. The effect of the abrupt change in modulus across a smooth adherend-adhesive interface is illustrated in Fig. 7, which shows the reduction in von Mises stress at the crack tip singular node in an aluminum ADCB joint at increasing distances from the interface. The data were from a 2D elasto-plastic finite element model (ANSYS® 12, Ansys Inc., Canonsburg, PA) using plane182 elements and singular elements to model the crack tip.
adhesive was modeled with a multi-linear stress-strain curve derived from tensile tests, which was previously shown in Chapter 6. The adherends were assumed to behave elastic-perfectly-plastic, with a tensile elastic modulus of $E=68.9$ GPa and a tensile yield stress of $\sigma_y=270$ MPa. For the adhesive $E_a=1.5$ MPa and $\sigma_y=30$ MPa.

Fig. 6 Fatigue failure surface of the thinner arm of an ADCB joint at (a) $R_a=1.3$ µm and (b) $R_a=3.9$ µm. Crack propagation in the direction of the arrow.
Figure 7 shows that any mechanism that causes the crack to deviate from the interface can lower the stresses at the crack tip and improve the fatigue behavior.

Finally, crack growth is associated with the formation and development of a damage zone ahead of the crack [21]. The deviation of the crack path from the interface due to the increase in surface roughness may allow a larger volume of the adhesive to deform plastically and so increase the fracture energy.

In addition to the hypothesis that asperity tips can generate a crack path that is removed from the nominal interface, it has been proposed that at some value of roughness, the increased stress concentration at the tips of the asperities will reduce the fracture strength of adhesive joints [6]. Therefore, a combination of the mechanisms discussed in the section could possibly explain the observed maximum in $G_{th}$ (Fig. 4).

![Fig. 7 Von Mises stress at the crack tip as a function of the distance from the upper adherend smooth aluminum interface, $d$. Finite element predictions for an aluminum ADCB joint and at $G=200$ J/m$^2$. The geometry of the ADCB joint was according to Fig. 1(b). $t$ is the adhesive thickness equal to 380 µm.](image)

3.1.3 Wettability and Void Formation

It has been shown that an increase in surface roughness can reduce the wettability of a substrate and hence the bond strength [22]. This effect would be accentuated if the adhesive is
highly viscous, or if there was insufficient time for adhesive flow before curing, and it provides another mechanism by which a maximum $G_{th}$ could arise (Fig. 4).

In present experiments, wetting was maximized by preheating both the adhesive and the aluminum to 55°C prior to adhesive application, and the bonded specimens were held at room temperature for at least 20 min before putting them in the oven for curing. These procedures were carried out to minimize the chance of the adhesive not wetting the substrate thoroughly.

To investigate whether the decrease in the $G_{th}$ at $R_a=9.0$ µm was due to an increase in void formation, a cross-section of the thinner arm of representative ADCB joints with $R_a=1.9$ µm, 6.4 µm and 9.0 µm were polished using a succession of abrasive papers (180, 240, 360, 600, 800 and 1200 grit). The samples were then polished with 6 µm diamond paste followed by 1 µm colloidal silica, and were carbon coated. Scanning electron microscopy did not reveal any voids that might be associated with the valleys of the rougher specimens.

Increasing the surface roughness also raises the possibility of air entrapment when the adhesive is applied [12]. In addition, as the surface roughness increases, it becomes more likely that small pieces of aluminum may remain unattached or poorly attached to the substrate after cleaning. Indeed, the fatigue failure surfaces of specimens with $R_a\geq 6.4$ µm had some sites of interfacial failure (less than 5% of the fracture area), with embedded aluminum pieces in the adhesive fracture surface (Fig. 8). No interfacial failure was observed for $R_a<6.4$ µm. The number of interfacial fracture sites increased to 10-20% of the fracture area when the roughness increased to $R_a=9.0$ µm, suggesting that trapped air bubbles or weakly attached aluminum asperities may have caused some decrease in the fatigue performance of the specimens at $R_a=9.0$ µm.

It is concluded that the increase in $G_{th}$ with surface roughness can come from several sources: an increase in the available bonding area, increased mechanical interlocking and crack growth retardation due to the substrate topography, and the deviation of crack path from the interface. However, at some roughness value, the crack path is far enough from the interface, and thus, no further increase in roughness affects the $G_{th}$. For very rough surfaces, the $G_{th}$ drops again, which could be due to the possibility of local interfacial failures coming from void formation.

The crack growth rate graphs ($da/dN$ vs. $G$) of aluminum ADCB’s at $R_a=1.3$ µm, 3.9 µm and 9.0 µm are compared in Fig. 9. For clarity, only one representative test is shown at each roughness. It is seen that at relatively high crack growth rates, compared to threshold, the graphs tend to be indistinguishable. This is consistent with our earlier observation that the crack path
moves farther from the interface as the crack speed increases [11,15], thereby becoming less sensitive to the surface roughness. This suggests that the effect of surface roughness should increase as the phase angle increases, and also as the crack speed slows near the threshold region. This hypothesis is examined in the next section using fatigue experiments on DCB joints under mode I loading where the crack propagated near the mid-plane of the bondline.

Fig. 8 Some interfacial failures observed for a sample at $R_a=6.4 \, \mu m$. Crack propagation from in the direction of the arrow.

3.2. Mode-I DCB Specimens

Two roughness values were chosen for mode I loading, $R_a=1.3 \, \mu m$ and $R_a=6.4 \, \mu m$, corresponding to the two extreme values of the fatigue threshold under mixed-mode loading ($G_{th} = 121\pm12 \, J/m^2$ and $178\pm19 \, J/m^2$, respectively). Three experiments were conducted in each case, showing no statistically significant difference between the $G_{th}$ values at the two roughnesses ($G_{th} = 195\pm3 \, J/m^2$ and $191\pm7 \, J/m^2$ at $R_a=1.3 \, \mu m$ and $R_a=6.4 \, \mu m$, respectively). The crack growth rate graphs of two representative specimens for each roughness are compared in Fig. 10, showing no effect of roughness on the fatigue crack growth rates under mode I loading. Therefore, consistent with the ADCB fatigue results at high crack speed, and as expected for cases where
the crack path is far from the interface, no effect of surface roughness on the mode I fatigue results was observed.

Fig. 9  Fatigue crack growth rate of aluminum ADCB at $R_a=1.3 \, \mu m$, 3.9 $\mu m$, and 9.0 $\mu m$.

Fig. 10  Effect of surface roughness on mode I fatigue crack growth rates of aluminum DCB. Two representative experiments are shown for each roughness.
4. Effect of Surface Roughness on Fracture Behavior of ADCB Specimens

Compared to cyclic loading, quasi-static loading introduces a relatively high strain energy release rate and crack speed. It was shown previously in Chapter 4 that quasi-static fracture tests at the same loading phase angle produce a more cohesive failure compared to fatigue threshold testing. To further verify that surface roughness affects the behavior of an adhesive joint only when the crack path is very close to the interface, quasi-static fracture tests were conducted on aluminum ADCB joints.

The principal objective was to measure the critical strain energy release rate for initiation, $G_c^i$, and the steady-state critical strain energy release rate, $G_c^s$, as a function of surface roughness. The $G$ calculation was the same as that used in the fatigue tests, Chapter 3.

Fracture tests using ADCB specimens produced a typical R-curve [23,24]. After crack initiation at $G_c^i$, the first several crack growth sequences occurred at an increasing critical energy release rate, $G_c$, as the damage zone at the crack tip developed to its steady-state form [25] (Fig 11). The steady state critical strain energy release rate, $G_c^s$, was considered to be the average value over the “plateau” (steady-state) region. The failure was fully cohesive in the adhesive (Fig. 12). Figure 13 shows the average $G_c^i$ and $G_c^s$ for the three tested roughnesses of $R_a=1.3$ µm, 1.9 µm and 6.4 µm. The error bars represent ± standard deviation calculated using the three measurements conducted for $G_c^i$ at each $R_a$, while the number of data points used to calculate $G_c^s$ was 96, 29 and 34 at $R_a$ of 1.3 µm, 1.9 µm and 6.4 µm, respectively. It is clear that no effect of surface roughness was evident in $G_c^i$ and $G_c^s$. Since the crack path in these fracture tests was relatively far from the interface (although still closer to the thinner adherend than the thicker one), this independency of fracture results on substrate roughness is consistent with the mode I fatigue results and mixed-mode ADCB fatigue results at higher crack growth rates.

5. Conclusions

Surface roughness had a significant effect on $G_{th}$ under a mixed-mode fatigue loading where the crack path was very close to the surface. The smallest $G_{th}$ was measured for the lowest tested roughness, $R_a=1.3$ µm, and increased about 50% as the surface roughness was increased to $R_a=3.9$ µm. This maximum value was also measured at $R_a=6.4$ µm, but then decreased 20% for a very rough surface of $R_a=9.0$ µm.

The increase in $G_{th}$ with surface roughness up to $R_a=3.9$ µm could be attributed to several factors: increase in bonding and fracture surface area, crack growth retardation due to crack path
deflection around asperities, and a shift in the failure locus away from the interface caused by stress concentrations at the tips of roughness asperities. This last effect is hypothesized to keep the crack tip away from regions of stress concentration closer to the aluminum-adhesive interface, where crack path propagation would otherwise occur more easily. The insensitivity of $G_{th}$ to roughness for $3.9 \, \mu m \le R_a \le 6.4 \, \mu m$ could be due to the crack path being far enough from the interface to be insensitive to these effects of substrate roughness.

The decrease in $G_{th}$ at $R_a = 9.0 \, \mu m$ was attributed to the increase in stress concentration at the tip of the roughness asperities, and also to void formation resulting from entrapped air and possibly weakly attached asperities.

Under mixed-mode loading, the effect of surface roughness on the fatigue crack growth rate decreased as the crack growth rate increased. This was due to the crack path shifting farther from the interface as the strain energy release increased.

![Fig. 11](image)

**Fig. 11** R-curve behavior of an ADCB joint at roughness of $R_a = 1.3 \, \mu m$.

![Fig. 12](image)

**Fig. 12** Fatigue and fracture failure surface of the thinner arm of an ADCB joint at $R_a = 1.3 \, \mu m$. 

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Fig. 13 Effect of surface roughness, $R_a$ on the critical crack initiation strain energy release rate, $G_{c,i}$, and critical steady-state strain energy release rate, $G_{c,s}$ of ADCB joints. Given values are the average values (±1 standard deviation). For $G_{c,i}$, three measurements were performed for each roughness. Number of data points on the plateau for $G_{c,s}$ measurements at $R_a=1.3$, 1.9 and 6.4 µm were 96, 29 and 34, respectively.

Experimental results for mode I fatigue, both the fatigue threshold and the fatigue crack growth rates, and quasi-static fracture, both $G_{c,i}$ and $G_{c,s}$, showed no dependency on the surface roughness. This was due to the crack path being relatively far from the interface and well within the adhesive.

The use of a different substrate material and roughening procedure might affect the microtopography and wettability of the bonding surface, and hence produce different results. However, an indication of the generality of the present observations is provided by the fact that similar trends have been reported in the literature with steel joints and other adhesives. For example, as mentioned previously references [7] and [8] found optimum $R_a$ values in cases where failure after quasi-static loading was close to the interface and was thus affected by the roughness. The torsional fatigue life of steel joints was also found to exhibit a maximum at a particular $R_a$ [9].

It is concluded that surface roughness has an appreciable effect only when the crack growth rate is low under mixed-mode loading; i.e. near the fatigue threshold. Under these
conditions the optimum roughness increased $G_{th}$ by up to 50%. At relatively high phase angles, the effect of roughness may become pronounced at crack growth rates larger than the fatigue threshold.
6. References

Chapter 9
Conclusions and Future Work

1. Summary and Conclusions

A fracture-mechanics energy-based approach was followed to characterize the near-threshold and fatigue crack growth rate behavior of structural adhesive joints. To be able to design adhesive systems for cyclic loading, the effect of different parameters on the fatigue behavior was studied. The important conclusions of the research have been categorized as follows:

1.1. Fracture Studies

The behavior of aluminum and steel adhesive joints bonded with a high-strength, toughened epoxy adhesive was studied under quasi-static loading. A method to predict the ultimate strength of adhesive joints was evaluated for the quasi-static loading of a variety of cracked lap shear (CLS) and single lap shear (SLS) joints. The conclusions are summarized as:

1. A previously established fracture-based, analytical approach for predicting the ultimate loads in common adhesive joints (CLS and SLS) can be applied to an epoxy adhesive that is an order of magnitude tougher than the earlier generation of (un-toughened) epoxy adhesives, provided the joint analysis takes into account the degree of subcritical crack growth. The approach is based on the observation that the strength of an adhesive bondline can be characterized by the fracture envelope for that particular adhesive system (i.e. the critical strain energy release rate, $G_c$, as a function of the mode ratio of loading, $\psi$). The subcritical crack growth length for both CLS and SLS specimens was approximately equal to the length of the rising part of the R-curve from the DCB specimens used to measure the fracture envelope. Using this approach, the average absolute error in the ultimate strength predictions was less than 10% for both aluminum and steel CLS and SLS joints.

2. During the fracture envelope measurement using aluminum and steel DCB specimens, the toughening behavior of the adhesive produced a relatively long rising part of the R-curve ($G_c$ vs. crack length) prior to reaching a steady-state critical strain energy release rate,
This extensive, stable subcritical crack growth also occurred during the loading of the CLS and SLS joints, thereby changing the joint geometry considerably and altering the strain energy release rate, $G$, and to a smaller extent, the crack tip mode ratio, $\psi$. It was found that accurate predictions of the ultimate load could be made if $\psi$ and $G$ are calculated using the final geometry of the CLS and SLS joints after subcritical crack growth equal to that observed in the DCB fracture tests. It was shown that the prediction of the ultimate load is relatively insensitive to the assumed amount of subcritical crack growth. As well, because the subcritical crack growth was so large, the ultimate joint strength was independent of the geometry of the end of the joint overlap (e.g. fillet shape). The detailed geometry of the end of the adhesive layer is expected to affect only the crack initiation load. In this thesis, the importance of understanding and including the subcritical crack growth region, rising part of an R-curve, in the fracture final failure design of toughened epoxies was shown for the first time in the literature. Considering the subcritical crack length in fracture joints based on the quasi-static measurements of DCB joints is also another significant contribution to this field of study.

3. Due to the R-curve behavior of the adhesive, the method can only be used to predict the ultimate load of joints having an overlap at least as large as the average subcritical crack growth length; otherwise the adhesive crack tip damage zone will not correspond to $G_c^s$. For joints with shorter overlaps, the rising part of the R-curve behavior would need to be modeled to permit a failure criterion based on $G_c$ as a function of the subcritical crack length, rather than the steady-state value $G_c^s$. Another limitation of the present approach is that it is applicable only to elastically deforming adherends since the equations used to calculate the strain energy release rate and the phase angle are based on this assumption, and the fracture envelope ($G_c^s$ as a function of the phase angle) was measured using elastic DCB specimens.

4. The joint strength increased as the elastic modulus of the adherend increased as seen when comparing the steel and aluminum joints. This was simply due to the relation between load, stiffness and the strain energy release rate. However, no difference was observed in the critical strain energy release rates that could be attributed to the adherend modulus. The effect of substrate material on fracture behavior of adhesive joints is currently controversial. There are very few papers on this topic, which show opposite trends. Therefore, this conclusion adds to the available results in the literature.

5. Although the approach has been demonstrated for SLS and CLS joints of aluminum and
steel, it is also applicable to other elastically deforming two-dimensional joint such as double lap joints. A “sandwich element” is isolated from the overlap end of the joint, and the force and moment reactions acting on the sandwich are used to calculate the strain energy release rate and the phase angle of loading.

6. The high toughness and visco-elastic nature of the adhesive made it preferable to identify the critical fracture load during the DCB fracture envelop testing using load drop rather than the earlier optical method of detecting crack advance. These DCB fracture tests were relatively insensitive to the cross-head speed over the range 0.5 – 5 mm/min.

1.2. Fatigue Threshold Behavior

The fatigue threshold and slow crack growth rate behavior of the highly-toughened epoxy adhesive was studied as a function of the starting condition (fatigue precrack and fillet), testing approach and interfacial bond strength. The conclusions can be summarized as follows:

1. The fatigue threshold measured at the tip of a fully developed fatigue crack was the same as that for an uncracked fillet provided that the crack path after initiation was cohesive. When the crack path became interfacial after beginning cohesively, a lower $G_{th}$ was measured from a fatigue pre-crack. As a result, it is concluded that measuring $G_{th}$ from an established fatigue crack provides a better assessment of an adhesive system for design purposes. This can be considered as a contribution of this research, since such a detailed study to compare these two common threshold measurement techniques does not exist. Surface roughness had no effect on $G_{th}$ and the average number of cycles to crack initiation when loading at $G_{th}$ from an intact fillet.

2. Cyclic loading at low loads in the threshold region caused crack initiation in a relatively small number of cycles compared to the expected total fatigue life of the joint. This suggests that for the current adhesive system the crack initiation phase can be safely ignored in life prediction modeling under low load conditions.

3. Fatigue tests conducted under force control yielded the same threshold and crack growth rate as those made with displacement control using load and displacement ratios of 0.1 and 0.5. This result supports the use of displacement control, which is usually a shorter and more convenient testing method than load control. The fatigue test results were independent of $dG/da$, and it was discovered that artificial
differences between force and displacement controlled tests can result if the adhesive layer compliance is neglected in the calculation of the strain energy release rate when plotting the data. This conclusion is another important contribution of the thesis. The available literature seems to show controversy results. It was shown that such controversy was most likely an artifact of ignoring the adhesive compliance in the $G$ calculation.

4. The crack path in fatigue was observed to be a function of both the phase angle and the crack speed. Mixed-mode fatigue testing at low crack speeds near the threshold tended to drive cracks close to the interface of the more highly strained adherend and was highly sensitive to the bond strength of this interface. In contrast, typical quasi-static mixed-mode fracture tests, such as DCB, cracked lap shear or single lap shear joints, could not distinguish between adhesive systems having subtle differences in bond strength because the crack paths tended to be further from the interface. This was also true of mode I fatigue testing since the crack path tended to be in the middle of the adhesive layer rather than being driven toward the more highly strained adherend, as happens under mixed-mode loading. Higher crack growth rates caused the crack path to move away from the interface and the fatigue behavior also became less sensitive to the interfacial bond strength. This effect was due to the mechanics of fatigue crack growth, and was not a function of environmental attack since the present experiments were conducted in dry air. It is therefore concluded that mixed-mode fatigue testing in the vicinity of the threshold is a highly effective way of assessing the interfacial bond strength of an adhesive system. Subtle changes in the adhesive, substrates, pretreatment or surface roughness were found to affect the mixed-mode, low speed fatigue results, but had very little influence on the either the mixed-mode fracture behavior or higher speed crack growth. This conclusion, a very significant contribution of this thesis, is of particular importance in applications experiencing high cycle mixed-mode fatigue loading near the threshold region. In such cases, the cohesive failure surfaces associated with good bonding can become unexpectedly interfacial if prior testing has not included fatigue at low crack growth rates.

5. It was shown that the effect of load and displacement ratio on the measured $G_{th}$ can be eliminated by calculating $G$ based on $\Delta P = P_{max} - P_{min}$ in a cycle, or by presenting the threshold according to the stress-based fracture mechanics approach in terms of $\Delta K$. This conclusion is of particular significance in the design of adhesive joints.
based on the fatigue threshold. A fatigue threshold test at a single load or displacement ratio should be enough for adhesive joint design purposes.

1.3. Effect of Mode Ratio on Fatigue

The fatigue behavior of different adhesive systems under mixed-mode loading was investigated in order to examine the possibility of characterizing the fatigue behavior of an adhesive system as a fatigue envelope, similar to fracture envelope which was previously examined for quasi-static loading in Chapter 2. A broad range of phase angles, 0° to 50°, was achieved by utilizing DCB, two types of ADCBs, and CLS joints. Various adhesive systems and fatigue failure modes were tested by changing the substrates, the surface roughness and the adherend surface pretreatment. The conclusions can be summarized as follows:

1. Under cyclic loading, the effect of $\psi$ on the $G_{th}$ and the fatigue crack growth rate was similar to that seen in quasi-static loading, i.e. $\psi$ affected the fatigue behavior only at $\psi>25^\circ$. In the literature, the effect of $\psi$ on $G_{th}$ is unclear and usually only mode I and one mixed-mode phase angle are compared.

2. The fatigue performance was found very sensitive to the failure pattern. The more cohesive the failure at higher phase angles, the higher the $G_{th}$ and the better the fatigue performance.

3. The threshold strain energy release rate, $G_{th}$, was 4-5% of the steady-state critical strain energy release rate of quasi-static fracture, $G_c^s$, for phase angles between 0° and 50°. Comparing this with the literature suggests that the increased fracture toughness of the present adhesive has not resulted in a commensurate improvement in the fatigue behavior.

4. Surface roughness was found to improve the adhesive bonding under cyclic loading. For the aluminum adhesive system at $\psi<25^\circ$, an increase in the surface roughness increased the residual adhesive thickness on the more highly strained arm and resulted in a fully cohesive failure. This resulted in an improvement in the fatigue behavior at higher phase angles.

5. Ignoring the presence of the adhesive will significantly underestimate the strain energy release rate, $G$, calculations at low loading phase angles. The beam on elastic foundation model resulted in a very good agreement with the finite element calculations for $G$. The analysis showed that ignoring the deflection of the crack path from the mid-plane of the adhesive layer towards one interface, which is caused by
the increase in the loading phase angle, will only slightly underestimate the calculated $G$ and overestimates the phase angle at the crack tip.

6. Comparing the rougher aluminum adhesive system ($R_a=1.33 \, \mu m$) and the Zn-phosphated steel systems, which produce similar fatigue failure modes, showed a very small effect of the substrate material on the fatigue threshold and fatigue crack growth rates. The mode-I fatigue crack growth rates for steel joints were slightly higher compared to the aluminum joints, when compared at the same $G$. Currently, there is no published work on how substrate material affects the cohesive behavior of adhesive joints under cyclic loading. Therefore, this conclusion is a significant contribution to fatigue studies of adhesives. The change in the substrate material can affect the fatigue performance if it affects the bonding because of the changes in the surface preparation of the new system.

1.4. Effect of Bond Strength

The effect of bond strength was studied by changing the surface treatment (Zn-phosphated vs. degreased) in steel joints, and surface roughness in aluminum joints.

1.4.1. Effect of Surface Treatment on Fatigue

It was found that surface treatment can affect the fatigue behavior by changing the failure mode. The change in the failure mode, from interfacial to cohesive, improved the fatigue behavior of the steel adhesive system at all crack speeds. For example, the $G_{th}$ values of Zn-phosphate treated DCB and ADCB joints were almost 4-5 times greater than those of the degreased joints.

1.4.2. Effect of Surface Roughness on Fatigue and Fracture

1. Surface roughness had no effect on $G_{th}$ and the average number of cycles to crack initiation when loading at $G_{th}$ from an intact fillet in CLS joints.

2. Surface roughness had a significant effect on $G_{th}$ under a mixed-mode fatigue loading (ADCB joints) where the crack path was very close to the surface. The smallest $G_{th}$ was measured for the lowest tested roughness, $R_a=1.3 \, \mu m$, and increased about 50% as the surface roughness was increased to $R_a=3.9 \, \mu m$. This maximum value was also measured at $R_a=6.4 \, \mu m$, but then decreased 20% for a very rough surface of $R_a=9.0$
µm. The effect of surface roughness on the fatigue threshold of adhesive joints was studied for the first time in this thesis.

3. The increase in $G_{th}$ with surface roughness up to $R_a=3.9$ µm could be attributed to several factors: increase in bonding and fracture surface area, crack growth retardation due to crack path deflection around asperities, and a shift in the failure locus away from the interface caused by stress concentrations at the tips of roughness asperities. This last effect is hypothesized to keep the crack tip away from regions of stress concentration closer to the aluminum-adhesive interface, where crack path propagation would otherwise occur more easily. The insensitivity of $G_{th}$ to roughness for $3.9$ µm $\leq R_a \leq 6.4$ µm could be due to the crack path being far enough from the interface to be insensitive to these effects of substrate roughness.

4. The decrease in $G_{th}$ at $R_a=9.0$ µm was attributed to the increase in stress concentration at the tip of the roughness asperities, and also to void formation resulting from entrapped air and possibly weakly attached asperities.

5. Under mixed-mode loading, the effect of surface roughness on the fatigue crack growth rate decreased as the crack growth rate increased. This was due to the crack path becoming farther from the interface as the strain energy release increased.

6. Experimental results for mode I fatigue, both the fatigue threshold and the fatigue crack growth rates, and quasi-static fracture, both $G_{c_i}$ and $G_{c_s}$, showed no dependency on the surface roughness. This was due to the crack path being relatively far from the interface and well within the adhesive.

7. The use of a different substrate material and roughening procedure might affect the microtopography and wettability of the bonding surface, and hence produce different results. However, an indication of the generality of the present observations is provided by the fact that similar trends have been reported in the literature with steel joints and other adhesives for both quasi-static and cyclic loading.

8. It is concluded that surface roughness has an appreciable effect only when the crack growth rate is low under mixed-mode loading; i.e. near the fatigue threshold. Under these conditions, the optimum roughness increased $G_{th}$ by up to 50%. At relatively high phase angles, the effect may become pronounced at crack growth rates larger than the fatigue threshold.

9. Consistent with the expectations from the ADCB results, increasing surface roughness from $R_a=0.77$ µm to $R_a=1.33$ µm increased $G_{th}$ for aluminum CLS joints.
(ψ=50°) by over 80%. An increase in the residual adhesive thickness at threshold was also observed.

1.5. Effect of Bondline Thickness on Fatigue and Fracture

The effect of bondline thickness on the fatigue and quasi-static fracture behavior of aluminum joints bonded using a toughened epoxy adhesive was studied. Finite element modeling was used to explain the experimental results. The fatigue studies were performed at a broad range of crack growth rates, including the fatigue threshold, under both mode I and mixed-mode conditions. The adhesive used to bond the adherends was a toughened epoxy. The important conclusions can be summarized as follows:

1. In general, at a given $G$ level, a decreasing bondline thickness tended to increase the stress triaxiality and elevate the stresses near the crack tip. This resulted in a damage zone that elongated and increased in thickness with decreasing bondline thickness, as long as it was not constrained by the adherends. As a result of these effects, the fatigue damage accumulation at a given $G$ was larger as the bondline thickness decreased, and consequently, the fatigue performance decreased.

2. As the applied $G$, and thus the crack growth rate, increased, the effect of the bondline thickness on the fatigue behavior became more pronounced. The crack growth rate was more sensitive than the fatigue threshold to changes in the adhesive thickness. This behavior was attributed to the dependency of $G_{const}$ to the bondline thickness, i.e. the thinner the bondline, the lower the $G$ at which the damage zone became affected by the adherend constraint.

3. Because the mode-I threshold damage zone was too small to be affected by the substrate for $t \geq 380 \mu m$, $G_{th}$ remained relatively constant for $t \geq 380 \mu m$.

4. Under mixed-mode loading, since the crack path was very close to the interface, the damage zone at threshold was only constrained by the more strained adherend. As a result, a very insignificant effect of bondline thickness on $G_{th}$ was observed. This observation may simplify the design of adhesive joints for fatigue loading. This is a significant contribution of the thesis, since there are very few papers dealing with the effect of adhesive thickness under mixed-mode loading and there is no paper on how $t$ affects the mixed-mode $G_{th}$. The thinner the bondline, the lower the $G$ at which the damage zone was influenced by both adherends. Therefore, the effect of adhesive thickness became more pronounced as the crack growth rate increased.
5. The measured critical strain energy release rate for quasi-static fracture increased linearly with bondline thickness from 130-780 µm. The change in the damage zone size with bondline thickness explains the effect of adhesive thickness on $G_c^s$. An optimum adhesive thickness for fracture loading of toughened adhesive joints was determined, based on the adhesive properties.

2. Future Work

1. A very important observation in this thesis was the dependency of the mixed-mode fatigue crack path to the fatigue crack growth rate (or equivalently the applied $G$). Such behavior resulted in higher sensitivity of mixed-mode near-threshold fatigue behavior to subtle changes in the interfacial bond strength. Several hypotheses are proposed in Chapter 4. Future work can assess the validity of the proposed mechanisms or prove other reasons for this behavior.

2. The adhesive is a visco-elastic material. Therefore, the fatigue behavior can also be a function of the strain rate at the crack tip region. As a result, the fatigue threshold envelope ($G_{th}$ vs. $\psi$) can vary with the testing frequency. It is suggested to study the effect of cyclic frequency on the near-threshold behavior of a toughened epoxy adhesive system. Change in the loading frequency can also change the crack tip temperature during cycling. Therefore, the relation between the frequency and the resultant crack tip temperature at different crack growth rates, and how such heating can affect the fatigue performance of the joint, needs to be studied.

3. Structural toughened epoxy adhesives are mostly heat-cured. Due to the thermal expansion mismatch between the adhesive and adherends, a curing residual stress will be formed in the adhesive layer. It is suggested to develop a finite element model to predict the thermal stresses in the adhesive layer. The model can then be employed to investigate the contribution of the residual stress to the total stresses (mechanical and thermal) developed in the adhesive layer. Based on such analysis, one can comment on the assumption of ignoring the curing residual stress in the fatigue analyses. The discussion should be based on the fatigue crack growth rates, since it is expected that the error should increase as the crack growth rate decreases, i.e. closer to the threshold region.
4. An energy-based approach was followed to study and characterize the near-threshold and higher crack growth rates fatigue behavior of structural adhesive joints. This approach proposes a design method based on an insignificant or a specified low crack growth rate, which is different from the stress-based (S-N) approach. In the S-N approach, the analysis is based on the applied stress and the number of cycles to the failure of the joint. The latter approach is widely used in industry. It is suggested to compare the calculated S-N graph from the energy-based approach for some standard adhesive joints, and compare it with the experimental measurements. The crack initiation phase is ignored in the fatigue studies using the energy-based approach, while it contributes to the total life of a joint. The contribution of crack initiation to the total life is expected to vary with the applied stress and the resultant crack growth rate. Therefore, to satisfy that the S-N data can be easily derived from the energy-based data, the proposed study should be conducted at a broad range of stress levels.

5. In the present study, the characterization of the near-threshold fatigue behavior of different adhesive joints was based on experimental studies. A fatigue damage accumulation model can be developed to predict the fatigue crack growth rate as a function of $G$.

6. The present research was for fresh undegraded adhesive joints. To be able to design joints for real-life conditions, it is necessary to understand how the environmental conditions (humidity and temperature) affect the fatigue behavior of a joint exposed to the aggressive environmental conditions. This needs to be conducted both in short-term and long-term conditions. Studying the fatigue behavior at high temperatures may also cause some creep crack growth in the adhesive layer.