Investigation of Microscale Damage Processes Near Adhesive-Composite Interfaces

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The increasing use of composite materials in the aerospace industry has necessitated significant advancements in the prediction of damage in composite structures. Adhesive joints have recently become more widespread, as such joints offer key advantages over bolted joints, such as causing no damage during hole drilling and providing weight savings. Adhesive joints however, fail catastrophically. To advance the understanding of failure of composite adhesive joints, the problem is approached at the microscale to investigate the fundamental damage processes.

Testing of miniature adhesive joints (Lapped area: 5 mm × 7 mm) has been carried out using a micro tensile testing apparatus in an SEM chamber. Video recordings of the tests allow examination of the evolution of damage processes during joint failure. Samples are tested under Mode I dominant and Mode II conditions. SEM images of the post failure appearance of the failure surface are presented and failure mechanisms under Mode I and Mode II conditions are compared in the context of the ASTM standard for adhesive joint assessment.

In conjunction with the experimental tests, a micromechanical finite element model of the interface region between a composite adherend and adhesive layer has been developed. An existing two-dimensional microscale RVE damage model is extended into three-dimensions, where accurate stress-strain response in comparison to experimental data is shown. Parameter studies on Mode I and Mode II strength at the fibre-matrix interface found that the Mode II interfacial strength had a negligible effect on the response of the RVE under loading in the transverse plane.

The ply model is extended to represent the first ply and half of the adhesive region of an adhesive composite joint. The ply model is separated from an elastic-plastic adhesive layer using damageable cohesive elements. Parameter studies under Mode I and Mode II conditions were undertaken to demonstrate the ability of the model to reproduce the failure appearance of the joints in the experimental tests. Under Mode I conditions, bonds failed in both the adherend and adhesive, while under Mode II conditions, failure occurred exclusively at the adhesive-adherend interface.

The damage parameters of the adhesive layer RVE are incorporated into a two-dimensional global scale model through cohesive zone modelling. The results are compared with the experimental data. It was found that the microscale RVE underestimated the fracture energy of the experimental damage processes.
Declaration

The substance of this thesis is the original work of the author and due reference and acknowledgment has been made, where necessary, to the work of others. No part of this thesis has already been submitted for any degree and is not being concurrently submitted in candidature for any degree.

Donal O’Dwyer

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# Contents

**Nomenclature**  
xiv

<table>
<thead>
<tr>
<th>Section</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Introduction</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>1.1.</td>
<td>Background</td>
<td>1</td>
</tr>
<tr>
<td>1.2.</td>
<td>Motivation</td>
<td>3</td>
</tr>
<tr>
<td>1.3.</td>
<td>Problem description and objectives</td>
<td>4</td>
</tr>
<tr>
<td>1.4.</td>
<td>Overview</td>
<td>6</td>
</tr>
<tr>
<td>2. Literature Review</td>
<td></td>
<td>8</td>
</tr>
<tr>
<td>2.1.</td>
<td>Introduction</td>
<td>8</td>
</tr>
<tr>
<td>2.2.</td>
<td>Damage in Composite Materials</td>
<td>8</td>
</tr>
<tr>
<td>2.3.</td>
<td>Micromechanical modelling of Composite Failure</td>
<td>11</td>
</tr>
<tr>
<td>2.3.1.</td>
<td>Development of micromechanics</td>
<td>11</td>
</tr>
<tr>
<td>2.3.2.</td>
<td>Material Properties</td>
<td>16</td>
</tr>
<tr>
<td>2.3.2.1.</td>
<td>Matrix elastic-plastic properties</td>
<td>17</td>
</tr>
<tr>
<td>2.3.2.2.</td>
<td>Fibre-Matrix interface properties</td>
<td>19</td>
</tr>
<tr>
<td>2.3.3.</td>
<td>Micromechanical studies</td>
<td>20</td>
</tr>
<tr>
<td>2.4.</td>
<td>Adhesive composite joints</td>
<td>25</td>
</tr>
<tr>
<td>2.4.1.</td>
<td>Surface treatment</td>
<td>26</td>
</tr>
<tr>
<td>2.4.2.</td>
<td>Composite-adhesive joints: Experimental tests</td>
<td>27</td>
</tr>
<tr>
<td>2.4.3.</td>
<td>Composite-adhesive joints: numerical studies</td>
<td>31</td>
</tr>
<tr>
<td>2.5.</td>
<td>Summary of literature review</td>
<td>34</td>
</tr>
</tbody>
</table>
3. In-situ SEM Mechanical Testing of Miniature Bonded Joints 38
   3.1. Introduction .......................................................... 38
   3.2. Material and test procedures ...................................... 40
       3.2.1. Mode I dominant test ........................................ 43
       3.2.2. Mode II test ................................................... 44
   3.3. Results ................................................................. 46
       3.3.1. Mode I dominant tests ....................................... 47
              In-situ analysis .................................................. 47
              Failure surface fractography .................................. 49
       3.3.2. Weak interface lap-shear bend test ......................... 55
              In-situ results ................................................... 55
              Failure surface fractography .................................. 56
       3.3.3. Mode II tests .................................................... 59
              In-situ analysis .................................................. 60
              Failure surface fractography .................................. 60
   3.4. Conclusions ........................................................... 62

4. Numerical Determination of Fibre-Matrix Interfacial Strength Parameters 66
   4.1. Introduction .......................................................... 66
   4.2. Modelling Strategy .................................................. 68
       4.2.1. Model Development ........................................... 68
       4.2.2. Boundary Conditions ......................................... 69
       4.2.3. Material Properties ........................................... 70
              HTA Fibres ......................................................... 70
              6376 Matrix ........................................................ 71
              Interface Region .................................................. 72
4.3. Results ................................................................. 74
  4.3.1. Interfacial Stress State ........................................ 75
    Single fibre RVE .................................................. 75
    Multi-fibre RVE ................................................... 76
  4.3.2. Overall response in transverse tension ...................... 77
    Influence of interface strength, transverse tension .......... 78
  4.3.3. In-plane Shear ................................................ 80
    Strain Hardening Effects Under In-Plane Shear ............... 80
    General response under in-plane shear ....................... 84
  4.3.4. Transverse Shear ............................................. 86
    Influence of interface strength, transverse shear .......... 86
4.4. Conclusions ...................................................... 89

5. Micromechanical modelling of damage processes at composite-adhesive
   interfaces ......................................................... 91
  5.1. Introduction .................................................... 91
  5.2. Model description ............................................... 92
    5.2.1. Adhesive layer properties ............................... 93
    5.2.2. Adhesive-adherend interface properties ............... 94
    5.2.3. Composite ply properties ............................... 97
    5.2.4. Mesh and boundary conditions ......................... 98
  5.3. Results ........................................................ 99
    5.3.1. Model stiffness ........................................... 99
    5.3.2. Adhesive-adherend interface Strength Variation ....... 100
      Low strength adhesive-adherend interface ................. 103
      Intermediate strength adhesive-adherend interface ...... 103
      High strength adhesive-adherend interface ............... 107
    5.3.3. Failure modes ............................................ 111
  5.4. Conclusions .................................................. 115
6. Utilisation of Micromechanical Models to Predict Joint Failure

6.1. Introduction

6.2. Interpretation of micromechanical results

6.3. Joint model

6.3.1. Cohesive element parameters

6.3.2. Results of joint model

Adhesive layer fracture energy

6.4. Conclusions

7. Conclusions, design recommendations and future work

7.1. Conclusions

7.1.1. SEM testing

7.1.2. Micromechanical modelling of composite and adhesive bond damage

Composite ply microscale model

Composite-adhesive interface microscale model

7.1.3. Linking of micro- and macro-scales

7.2. Design recommendations

7.3. Recommended Future work

7.3.1. Epoxy damage model

7.3.2. Geometric alterations

7.3.3. Alternative adhesive types

7.3.4. Use of microscale models to predict interlaminar Damage

7.3.5. In-situ SEM testing

7.3.6. Investigation of joint test geometries

Bibliography
A. Code Files
   A.1. Periodic Constraint Equations ........................................... 157
       A.1.1. RVE model ............................................................... 158
   A.2. Python Scripts .............................................................. 162
       A.2.1. Composite ply model: transverse tension ......................... 162
       A.2.2. Composite ply model: transverse shear .......................... 166
       A.2.3. Composite ply model: in-plane shear ............................ 166
       A.2.4. Adhesive layer model: transverse tension ....................... 167
       A.2.5. Adhesive layer model: in-plane shear ............................ 168

B. Model Parameter Studies
   B.1. Model Size Variation ..................................................... 170
   B.2. Mesh Sensitivity ............................................................ 171
List of Figures

1.1. Microscale analysis of a bonded joint between composite components. . . 3

2.1. Failure modes of unidirectional composite plies. . . . . . . . . . . . . . . . 9
2.2. Illustration of homogenisation and repeating unit cell. . . . . . . . . . . . . 12
2.3. Idealised fibre distributions in (a) and (b), compared to a distribution cre- ated using the NNA in (c) and an actual fibre distribution in (d). . . . . . . 14
2.4. Separation of the fibre-matrix interface in a carbon-fibre composite. From Hobbiebrunken et al. (2006). . . . . . . . . . . . . . . . . . . . . . . . . . . . 17
2.5. Failure surfaces. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 18
2.6. Stress distribution in the initial and failed configurations (a) shows a crack path where one defect remained independent of the final failure path (b) shows a crack path which included both initial defects. Adapted from Alfaro et al. (2010). . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 22
2.7. Comparison of shear bands in an actual composite and predicted forma- tion in an RVE, under the same loading conditions, adapted from González and Llorca (2007). . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 24
2.8. Peel and shear stress in a single lap adhesively bonded joint. . . . . . . . 27
2.9. Failure surfaces (adapted from Kim et al. (2006)), showing three damage mechanisms on a single failure surface. . . . . . . . . . . . . . . . . . . . . . . 28
2.10. Failure surface from a DCB test, showing a mix of failure types (From Ash- croft et al. (2001)). . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 29
2.11. Plots of the stress state through the mid-plane of a single lap joint, from Vaidya et al. (2006). ................................................................. 30
2.12. Failure surface showing fibre tear failure (FTF) and cohesive failure (CF) (from Khalili and Shokuhfar et al. (2008)). ................................. 31
2.13. Correlation of failure mode and strength of composite adhesive joints, from Kim et al. (2008). ............................................................ 33
2.14. Results from an adhesive microscale RVE under peel loading, compared to an in-situ SEM micrograph of a DCB specimen, loading is in the vertical direction in both cases. (Salomonsson and Andersson, 2008) .......... 35

3.1. CAD representation of the microtester constraints. The red box approximates the scanning range of the SEM. ................................. 41
3.2. Miniature specimen test geometry. (a) Test configuration (b) Specimen dimensions. All dimensions are in mm. Bondline: 50 μm. Specimen thickness: 7mm. ................................................................. 42
3.3. Layup configuration, temperature, T, and pressure, P, supplied by the autoclave. ................................................................. 42
3.4. Stress distribution through the midsection of the adhesive layer, Mode I dominant conditions. ................................................................. 44
3.5. Stress distribution through the midsection of the adhesive layer, Mode II dominant conditions. ................................................................. 45
3.6. In-situ analysis, Mode I dominant test, sample: B2. (a) Initial state. (b) Crack initiation, at 112 N. (c) Final failure at 209 N (d) Positions of frames on the load-displacement curve. ................................................................. 48
3.7. Failure surface, Mode I dominant test. A bundle of fibres, pulled from the adherend are visible, remaining on the surface of the adhesive. (Sample: B3) ................................................................. 50
3.8. Type 1, Adhesive failure. Fibre tracks on the adherend and the adhesive surfaces indicate failure at the interface between the adhesive and the adherend. (Sample: B4) ............................................. 51

3.9. Type 3, Thin layer cohesive failure (Sample: B3) ............................................. 51

3.10. Type 5, Light fibre tear failure. Fibre tracks are visible, accompanied by single fibres, adjacent to the adhesive. (Sample: B3) ............................................. 52

3.11. Type 7, Mixed failure. Regions of fibre tear failure, thin layer cohesive failure and light fibre tear failure are visible, immediately adjacent to each other. (Sample: B3) ............................................. 53

3.12. Mode I dominant composite failure ................................................................. 54

3.13. Crack growth through the epoxy adhesive and matrix materials under:
   (a) Mode I conditions (b) Mixed mode and (c) Mode II conditions. In all cases, the dashed lines represent predicted crack path, adapted from Greenhalgh (2009) ......................................................... 54

3.14. Experimental load-displacement results .......................................................... 56

3.15. In-situ analysis of joint failure with a weak composite-adhesive interface.
   (a) The initial state of the joint. (b) Just prior to catastrophic final failure, no damage is visible. (c) Positions of the Figures (a) and (b) on a load-displacement curve. (Sample W2) ......................................................... 57

3.16. (a) Failure surface of a joint with a weak interface, showing mirror, mist and hackle crack growth (b) Defect on the surface of the adherend. (c) Transition from tide mark pattern to a riverline pattern. .............................. 58

3.17. In-situ analysis, Mode II test, sample C2. (a) Initial state. (b) Adhesive layer at F=411N. (c) Final failure at F=711N. (d) Positions of frames on a load-displacement curve. .............................. 61

3.18. Failure surface, Mode II test. The adherend surface is visible, separated from the region where the adhesive remained on the adherend by the transition region. (Sample: C6) ......................................................... 61
3.19. (a) Cusps found on the adherend surface after Mode II failure, sample C4.
(b) Cusps seen on the adhesive surface after Mode II failure, sample C7.
(c) Feature from the transition region of a Mode II test. Surfaces of the adhesive and the composite adherend are visible, the regions are connected by the indicated Mode I cracks, sample C3.

4.1. Dimensions of microstructure (Note: Not to scale)

4.2. Pure shear applied to control nodes of an RVE in the transverse plane.

4.3. Cohesive Element details (a) Interface Local Orientations (b) Traction separation law

4.4. Loading conditions (a) Transverse tension (b) In-plane shear (c) Transverse shear

4.5. (a) Single Fibre RVE (b) the normal and shear interfacial tractions under transverse tension (c) normal and shear interfacial tractions under transverse shear

4.6. (a) Fibre in a multi-fibre array, marked “A” about which normal and shear interfacial tractions are shown in (b) under transverse tension and (c) under transverse shear

4.7. Crack path under transverse tension indicated by high levels of equivalent plastic strain (PEEQ)

4.8. Stress-strain results under transverse tension (a) Variation of interfacial Mode I strength (b) Variation of interfacial Mode II strength

4.9. Stress-strain results under transverse tension

4.10. Pure shear state with relation to the fibre direction (not to scale)

4.11. Stress strain results under in-plane shear, the hardening effect is visible at shear strains higher than approximately 4%

4.12. Longitudinal stress in the fibre

4.13. Shear stress in the fibre

4.14. Fibre rotation (θ) relative to the initial and final orientation,
Nomenclature

4.15. Fibre Rotation, Elastic and Plastic models ........................................ 85
4.16. In-plane Shear (Note: the outline of the deformed RVE has been extended
   in the fibre direction for clarity) ......................................................... 85
4.17. Stress strain results under in-plane shear ........................................ 86
4.18. Plastic shear strain (PEEQ) band under transverse shear (Note: a deforma-
   tion scale factor of 2 has been applied for this figure) ........................ 87
4.19. Stress-strain results under transverse shear (a) Variation of Mode I inter-
   facial strength (b) Variation of Mode II interfacial strength .................. 88

5.1. Adhesive layer model as applied to a composite joint, including local re-
   ference orientations. The dimensions L and x are the width of the RVE and
   the weakened central region of the adhesive-adherend interface respect-
   ively. ..................................................................................................... 93
5.2. Comparison of the stress-strain behaviour of the adhesive and matrix
   materials. .............................................................................................. 95
5.3. Adhesive-adherend interface traction-separation curve. Note: diagram
   not to scale. ......................................................................................... 96
5.4. Schematic of adhesive layer model .................................................... 98
5.5. Correlation of model elastic moduli with analytical solution. ............... 101
5.6. Stress strain results ........................................................................... 102
5.7. (a) Mode I failure at a weak adhesive-adherend interface ($t_{hc}^c = 50$ MPa)
   showing small amounts of plastic yielding in the adhesive only. (b) Mode
   II failure of a weak adhesive-adherend interface ($t_{hs}^c = 15$ MPa), separation
   of the adhesive and composite occurred without significant plastic yield-
   ing. ........................................................................................................ 104
5.8. (a) Equivalent plastic strain after Mode I failure of an intermediate strength 
\( t_c^n = 70 \text{MPa} \) adhesive-adherend interface. Uneven failure of the adhesive-
adherend interface, due to proximity of the surface fibres is visible. (b) 
Mode II failure of an intermediate strength \( t_s^c = 25 \text{MPa} \) adhesive-adherend interface. Regions marked (A) are regions of high plasticity in the adhes-
ive, due to uneven failure of the adhesive-adherend interface. .......................... 106

5.9. (a) Mode I failure at a strong adhesive-adherend interface \( t_n^c = 100 \text{MPa} \). 
The entire failure process is within the composite region. (b) Mode II 
deformation of a strong \( t_s^c = 40 \text{MPa} \) adhesive-adherend interface. De-
formation was confined to the adhesive layer in the form of plastic yield-
ing, and no debonding or damage at the adhesive-adherend interface 
was found. ................................................................. 108

5.10. Bond strength variation ................................................................. 110

5.11. Mixed failure under Mode I loading, with regions of composite and inter-
facial failure marked. ................................................................. 112

5.12. Qualitative comparison of experimental data and model results. ................. 114

6.1. Procedure outline, linking the micro and macro scale modelling ................. 120

6.2. Traction-separation curves, extracted from the models in chapter 5, for 
strong and weak bondlines under Mode I, and under Mode II deformation. 122

6.3. Model details. the adhesive layer thickness is exaggerated for clarity (ac-
tual thickness: \( 50 \times 10^{-3} \text{mm} \)). ................................................................. 123

6.4. Traction separation curves, as applied to the joint model adhesive layer. . 126

6.5. Plot of \( \sigma_{22} \) before damage initiation (Mode I stress - see orientations in 
Figure 6.3). ................................................................. 126

6.6. Plot of \( \sigma_{22} \) after damage initiation (see orientations in Figure 6.3). Stress 
concentrations and damaged cohesive elements are highlighted. ................. 127

6.7. Comparison of 2D joint model and experimental results. ......................... 127

6.8. Effect of increasing fracture energy of the cohesive zone in the joint model. 129
6.9. Evolution of the damage parameter, D, measured from the start of the damage zone, along the bondline. ........................................ 130

7.1. RVEs showing (a) current planar interface and (b) proposed wavy interface. 139

7.2. Model of interply region within a laminate. (a) Shows stress in the through-thickness direction (b) examines the same stress contour over surface fibres, showing internal variations in the stress field. ...................... 142

7.3. Edge-on view of fibres under the SEM .................................... 144

A.1. Nodesets, as applied to the composite ply RVE and the adhesive layer RVE 157

B.1. Model sizes (a) 30 µm (b) 60 µm (c) 100 µm ............................. 170

B.2. Stress-strain plots of three different sized adhesive layer models ....... 171

B.3. RVE used in mesh sensitivity study ........................................ 171

B.4. Relationship between the mesh size and resultant strength of the RVE. .. 172
List of Tables

3.1. Failure types under Mode I dominant conditions following ASTM D5573-99 (Note: all Mode II test samples failed in Type 1 failure) .............. 47

4.1. Elastic properties of constituent phases ........................................ 71
4.2. Interfacial stiffnesses ................................................................. 72
4.3. Interface Energies (Varna et al., 1997) ....................................... 73
4.4. Interfacial strength values ............................................................ 76

5.1. Material properties ................................................................. 94
5.2. Adhesive-adherend interface cohesive parameters ....................... 97
5.3. Fibre-matrix interface parameters, measured radially from the centre of each fibre. ................................................................. 98
5.4. Mode I stress at failure and failure type for each model. ............... 111

6.1. Cohesive parameters ............................................................... 123
6.2. Transversely isotropic elastic properties of the composite material. .... 124
6.3. Adhesive material elastic moduli and equivalent cohesive element prop-
     erties. ...................................................................................... 125
Nomenclature

Lowercase Roman Symbols

\( d_f \)  Average fibre diameter

\( h_c \)  Height of composite region

\( k \)  Initial stiffness of cohesive element

\( k^d \)  Damaged stiffness

\( k_a \)  Adhesive stiffness

\( k_c \)  Composite stiffness

\( k_T \)  Total model stiffness

\( k_{n} \)  Corrected stiffness of cohesive element, normal direction

\( k_{s} \)  Corrected stiffness of cohesive element, shear direction

\( t \)  Interfacial traction

\( t_n^c \)  Critical interfacial traction-normal direction

\( t_{s1}^c \)  Critical interfacial traction-first shear direction

\( t_n \)  Interfacial traction-normal direction

\( t_{s1} \)  Interfacial traction-first shear direction

\( w \)  Weakened length of bondline
Nomenclature

$w_{RVE}$ Width of RVE

$x$ Undeformed co-ordinate of control node

$x'$ Deformed co-ordinate of control node

$x_d$ Distance from damage initiation

$y$ Undeformed co-ordinate of control node

$y'$ Deformed co-ordinate of control node

**Uppercase Roman Symbols**

A Area

D Damage Variable

$D_{PS}$ Pure Shear Deformation Matrix

E Modulus of Elasticity

$E_{22}$ Elastic modulus, transverse direction

$E_{comp}$ Composite elastic modulus

$F_{12}$ Combined shear load

$G^c$ Critical Fracture Energy

$G_n$ Mode I Fracture Energy

$G_s$ Mode II Fracture Energy

$G_{12}$ In-plane shear modulus

$G_{23}$ Transverse shear modulus

$G_{comp}$ Composite shear modulus

$G_c$ Interfacial mixed mode interfacial energy
Nomenclature

\( G_{S1} \) Interfacial interface energy-shear direction

\( G_{S2} \) Interfacial interface energy-second shear direction

L Bondline length

N Control node

S Strength of weakened region

T Characteristic element length

V Volume

Abbreviations

BK Benzeggagh-Kenane

CF Cohesive Failure

DCB Double Cantilever Beam

ENF End Notch Flexure

FE Finite Element

LDT Large Deformation Theory

NNA Nearest Neighbour Algorithm

PEEQ Equivalent Plastic Strain

RSA Random Sequential Adsorption

RVE Representative Volume Element

SDT Small Deformation Theory

SEM Scanning Electron Microscope
Nomenclature

Greek Symbols

\( \alpha \) Initial fibre-loading angle

\( \alpha' \) Reduced fibre-loading angle

\( \bar{\epsilon}_{ij} \) Average strain

\( \bar{\sigma}_{ij} \) Average Stress

\( \Delta_{\text{applied}} \) Applied displacement

\( \delta^c_n \) Critical separation, normal direction

\( \delta^c_s \) Critical separation, shear direction

\( \epsilon \) Strain

\( \epsilon_{ij} \) Strain in i-j direction

\( \eta \) BK criterion mode mixing parameter

\( \gamma \) Shear Strain

\( \gamma_{12} \) In-plane shear strain

\( \lambda \) Stretch

\( \phi \) Mohr-Coulomb friction angle

\( \sigma \) Stress

\( \sigma_c \) Compressive strength of the matrix

\( \sigma_{ij} \) Stress in i-j direction

\( \sigma_{\text{max}} \) Strength of composite bond, Mode I

\( \sigma_n \) Yield stress under shear of the matrix

\( \sigma_t \) Tensile strength of the matrix
Nomenclature

$\tau$  Shear Stress

$\tau_{max}$  Strength of composite bond, Mode II

$\tau^c_m$  Yield stress under shear of the matrix

$\theta$  Fibre rotation angle
1. Introduction

1.1. Background

Certain combinations of materials, can incorporate the best properties of each material in a new composite material. The concept is not new; primitive ancient structures used wattle and daub walls, to combine the properties of straw with clay. The construction industry adopted steel reinforced-concrete in the nineteenth century to add tensile strength to concrete through embedded steel bars. More recently, the aerospace industry, in the drive to improve fuel efficiency of aircraft and reduce costs, have turned to composite materials to provide lightweight solutions as an alternative to conventional materials, such as aluminium. Standard aerospace composite materials combine the workability and toughness of a polymeric resin with the high strength and stiffness of glass or carbon fibres. The long, slender fibres, when bound together by the polymeric matrix, give the material much of the high strength and stiffness of the fibres while the matrix greatly improves toughness relative to that of the brittle fibres, to provide a material with properties desirable to the aerospace industry.

The percentage weight of composite materials used in aircraft construction is rising steadily, from 2% composite by weight in the Boeing 707 (first flight 1957 (Tenek and Argyris, 1997)) towards the 15% mark, common in aircraft today (Airbus A320). In excess of 50% of the structural weight of “next generation” aircraft (Boeing 787 and Airbus A350XWB) comprises of composite materials.

With the advent of large and structurally important composite components (e.g. aircraft fuselages), high confidence in manufacturing processes is needed. Compos-
1.1 Background

Composite manufacturing for industry standards generally involves the creation of a laminate through a process called "laying up" which involves building up layers of fibres which are pre-impregnated with the resin material (pre-preg). The laminate is then placed in an autoclave at high temperature and pressure to cure the matrix. Size constraints imparted on components arising from restrictions in size of the autoclave and controlled environment, mean that large components are generally not manufactured in one piece. This implies that joints between components are critical to the performance of an overall structure.

Methods of joining composite components are under constant development. Mechanical joining, in the form of rivets or bolts currently dominates composite assemblies, as a result of the suitability of mechanical joints in metal structures. The transition to composite materials retained the trusted bolting technique. Confidence in adhesive bonding is not as high, as designers are wary of the catastrophic failure behaviour that is characteristic of such bonds. However, there are definite advantages to the adhesive jointing technique, which make it particularly attractive to the aerospace industry, primarily, the weight savings associated with replacing a large metal bolt with a thin adhesive layer. A less obvious issue, which is more critical to composite-to-composite joints than in metal-to-metal joints, is that damage is induced to laminates during the manufacturing process when preparing bolted joints. A hole which is drilled through a composite laminate introduces at the bolthole. The accumulation of such damage over thousands of holes in a structure may have an effect on structural integrity. This type of damage is not associated with adhesive joints; therefore the approach is extremely attractive to designers.

This thesis analyses the microscale damage processes of adhesive joints during a failure event, following the methodology outlined in Figure 1.1, where a joint between composite components in an aircraft is analysed at different length scales using different techniques.
1.2. Motivation

Failure processes in composite-to-composite joints are more complicated than those in metal-to-metal joints because of the involvement of the composite adherends in the failure process, due to the relatively low through thickness strength of the composite. Analysing the damage processes which occur during failure of an adhesive bond between two composite adherends will help to increase the understanding of such failures and provide tools for failure prediction.

Currently, validation of design is predominantly undertaken using experimental testing regimes, involving test samples ranging in scale from single bolt coupons to full scale components. Testing in this manner introduces large costs to the design process, especially in the case of full scale components. For this reason, virtual testing, using numerical methods, is an attractive alternative, as it generally amounts to significantly lower cost. In the case of composite materials, the accepted approach for such studies is the use of finite element analyses to assess the stress state in a laminate in terms of the orientations of the constituent plies. A failure criterion is subsequently
applied to determine if failure will take place in a ply. Such criteria generally take the effects of micromechanical failure mechanisms into account in an average sense, through assignment of different strength values, depending on the orientation of each ply (Trias, Costa, Fiedler, Hobbiebrunken and Hurtado, 2006). This approach is capable of producing reasonably accurate predictions for failure of composite components.

However, an approach to prediction of composite failure which is gaining popularity is consideration of the microscale damage processes within a composite material. A comprehensive prediction of failure of composite materials needs to take into account the different failure mechanisms at the scale of the individual fibres and such an approach is expected to lead to more accurate design and assessment approaches.

Micromechanics based methodologies have been explored for composite materials at the ply level, where the field is currently well advanced (Vaughan and McCarthy, 2011a,b, González and Llorca, 2006, 2007, Totry et al., 2010). However, the area of bonded composite joints has not been extensively explored using micromechanics based approaches, therefore this thesis focuses on this topic.

1.3. Problem description and objectives

This thesis focuses primarily on an adhesive layer and composite adherend in the failure processes in composite bonded joints. Fractography on failed surfaces of adhesive joints has been carried out by e.g. Ashcroft et al. (2001), Kim et al. (2006), but the microscale damage processes have not been extensively explored for unidirectional composites. It is proposed to capture damage processes in bonded composite joints through tests on miniature bonded joints. The first objective of this work is to investigate the damage processes using experimental testing, in the chamber of a Scanning Electron Microscope (SEM). Correlation of video evidence of damage progression with load-displacement results will allow crack propagation, or lack thereof, to be investigated, and active damage processes to be tracked. For completeness, this will be accompanied by a post failure fractographic analysis of the failure surfaces of the joint,
1.3 Problem description and objectives

to identify the damage processes hidden within the joint, away from the SEM beam during testing.

Microscale models of composite damage have been developed in the literature. Aspects of investigations have previously included; extraction of microscale results to the macroscale (Matouš et al., 2008), fibre placement (Trias, Costa, Mayugo and Hurtado, 2006), material properties of the matrix and fibre phases (Hobbiebrunken et al., 2007), and good damage response has been reported from two-dimensional models (González and Llorca, 2007). The second objective of this work is to extend a previously developed methodology for two-dimensional analysis of a composite material (Vaughan and McCarthy, 2010, 2011a,b) into a three-dimensional model. This extension is necessary in terms of adhesive bonds, as surface plies of composite materials tend to be aligned with the joint loading direction, inducing a shear condition which is not possible to investigate using two-dimensional models. Appropriate experimental results exist for laminates under the proposed shear condition (O’Higgins et al., 2011), which will be used to validate the new models.

A literature review by Banea and da Silva (2009) highlighted the complicated nature of failure in composite adhesive joints. In this review, it is concluded that an accurate model of composite adhesive joint failure should have the capability to model failure in the composite, in addition to at the interface between the adhesive and composite regions and deformation of the adhesive layer itself. The third objective of this work is to represent the phenomenological failure behaviour at the composite-adhesive interface. Such a model has not been published previously. This model is an extended version of the three-dimensional composite ply model. The model contains a microscale representation of the three critical regions; the composite, the composite-adhesive interface and the adhesive layer. Loadings representative of the stress state experienced at the boundary of the adhesive and composite adherends of a composite joint will be applied to the microscale model. The objective of the new model will be to recreate a range of failure behaviours resulting from the experimental tests in the first objective.

The final objective of this work is to create a link between the microscale modelling
and the macroscale testing. Such an approach has been outlined previously (Kulkarni et al., 2009) but to the author’s knowledge, has not yet been implemented. The purpose of the micro-macro link is to allow the effect of damage mechanisms acting at the microscale to be incorporated into macroscale failure prediction and design procedures.

1.4. Overview

In Chapter 2, a review of published literature, related to microscale damage processes in composite materials is presented. An introduction to the field of composite micromechanics is provided and important examples of the method are presented. The results of the most effective microscale approaches are presented, which provide a starting point for the studies in Chapter 4 and Chapter 5. A number of examples of experimental analyses of tests on composite adhesive joints are also presented, and show the current state of experimental testing on adhesive joints, for comparison with Chapter 3.

Chapter 3 presents a novel experimental testing regime, investigating the failure characteristics of bonded composite joints under Mode I dominant and Mode II conditions. The tests are carried out on miniature adhesive joints, and the testing apparatus is located in the chamber of an SEM. The test set up allows for the damage progression to be recorded in-situ, which gives resolution of the microscale damage processes. These processes are further analysed using post-failure fractography.

In Chapter 4, a three-dimensional micromechanical model is developed, which captures damage processes within a composite ply. Fibre-matrix debonding and matrix plasticity are incorporated to capture the main deformations within a composite ply during failure. The damage response of the model is compared to published data for the material. A parameter study investigates the relative influence of the peel and shear strengths at the fibre-matrix interfaces in the micromodel. This study is used to determine a single set of interfacial parameters, capable of reproducing failure strengths and damage processes under a variety of different loading conditions. This is accom-
panied by a microscale quantification of the fibre-rotation leading to the known shear stiffening effect of laminates under in-plane shear.

In Chapter 5, the composite micromodel is extended to include a region of adhesive. The model represents the first ply of a composite adherend, the adhesive layer and the interface between the two regions in a bonded joint. Damage is included in the composite using the damage model developed in Chapter 4, and the separation of the adhesive from the composite is captured through a layer of cohesive elements. The adhesive material is characterised using elastic-plastic properties, similarly to the matrix material in the composite model. Mode I and Mode II conditions are applied to the model, and comparisons are made with the appearance of failure surfaces resulting from the tests in Chapter 3.

Chapter 6 provides a link between the microscale modelling of Chapter 5 and the experimental tests of Chapter 3. A two-dimensional joint model is presented, which uses cohesive zone modelling to consider damage of the adhesive layer and first ply of the adherends. The parameters of the joint model cohesive zone are extracted from the microscale model of the interface region, presented in Chapter 5, and the results are compared to the three point bending tests carried out in Chapter 3.

Chapter 7 provides a summary of the main results of each section of the thesis. These points are accompanied by recommendations for future work, relating to the work carried out here.
2. Literature Review

2.1. Introduction

A review of published work is presented to provide information concerning the damage and failure processes of composite laminates and adhesive composite joints. Special consideration is given to the micromechanical aspect of damage in composite materials and adhesive bonds with composite adherends. An introduction to the field of numerical micromechanics is provided, and current state of the art models and studies are described. This is followed by a review of numerical and experimental analyses of adhesively bonded composite joints and focus is maintained, where possible, on the micromechanical aspect of adhesive joint failure.

2.2. Damage in Composite Materials

The failure of composite materials is a complex process. In Herakovich (1998), it is shown that many local failures can occur prior to the final catastrophic failure of a laminate, and that the first signs of damage need not correspond directly to the final failure of the laminate. Regions of microscopic damage coalesce and develop, influenced by the local pattern of fibre positions and ply orientations, to produce an extremely complex final failure morphology at the laminate level. It is accepted that damage in composite materials is highly sensitive to the orientation of the fibres relative to applied loadings. Different damage processes can be induced based on the configuration of the applied load relative to the fibre orientation. This is significant when damage is
2.2 Damage in Composite Materials

Considered at the ply level within a laminate, as multiple ply orientations are regularly used, resulting in multiple modes of failure in a single laminate. Interlaminar damage and interactions of failure mechanisms in adjacent plies may also contribute to the damage response of a laminate. A description of the damage response of a unidirectional carbon-fibre ply under the most common loadings follows.

The strength of a unidirectional ply along the fibre direction is controlled by the fibre’s axial strength. Carbon fibres are extremely strong and stiff in the axial direction. Therefore, under tension aligned with the fibre direction, once the stress in a fibre exceeds its failure strength and the fibre fails, the load carried by this fibre is rapidly redistributed among its neighbours. This localised redistribution of stress can cause fibre failure in neighbouring fibres, leading to a rapid propagation of fibre damage through the laminate. The matrix is unable to support the redistribution of the stress from the failed fibres and the failure pattern in Figure 2.1a is produced, at 90° to the applied load. This thesis does not consider tensile loading in the plane of the fibres, as failure in this loading is dominated by the failure of the fibres, with little damage progression, and is not an interesting case at the microscale.

![Figure 2.1: Failure modes of unidirectional composite plies.](image)
Compresssion in the axial direction produces a micro-buckling failure of the fibres. Their long, slender shape means that the fibres, despite the support of the surrounding fibres, remain susceptible to buckling. Experimental tests in this orientation generally produce a “dirty” failure surface, due to the lack of a clean separation between compressive failure surfaces, destroying the failure morphology (Herakovich, 1998). However, micrographs in Greenhalgh (2009) show clear kink bands of fibres under axial compression, forming the pattern illustrated in Figure 2.1b.

Tensile failure in the transverse plane is attributed to fibre-matrix debonding and is shown in Figure 2.1c. Tension transverse to the fibres produces failure initiation at the fibre-matrix interfaces. This increases the amount of load carried through the matrix, which deforms plasticity until final failure occurs in the highly deformed matrix material along a plane orientated at 90° to the loading direction.

Compression in the transverse plane produces plastic yielding in the matrix material. Shear bands are formed at approximately 45° to the applied load as shown in Figure 2.1d. Debonding has been reported along the shear bands in modelling and experimental work (González and Llorca, 2007) at high applied load. In the absence of this debonding, failure occurs through compressive failure of the material in the matrix shear bands.

Failure under transverse shear occurs as concentrated matrix yielding along planes of maximum shear, accompanied by debonding of fibres within the shear band, shown in Figure 2.1e.

Composite failure response under in-plane shear is characterised by intense shear bands of plastic yielding in the matrix. High shear strains are possible, as shown in Totry et al. (2010) where experiments have demonstrated that debonding may not be active in high strength composite materials up to ~15% in-plane shear strain. Failure in this configuration occurs along shear bands lying along the fibre direction, as shown in Figure 2.1f.

It is clear that the different failure types which occur in composite materials complicate the numerical prediction of material properties. For this reason, the field of mi-
Micromechanical modelling has become increasingly prevalent, as more accurate strength predictions are sought.

### 2.3. Micromechanical modelling of Composite Failure

Historically, numerical predictions of composite failure have not been made at the microscale, but at a level where the failure of individual fibres and the surrounding matrix are considered in an average sense. Stresses in the individual plies of a layup are assessed in terms of strength values associated with each different fibre orientation. This provides an efficient numerical method when failure is considered in a structural component. Defining the root causes of the differing directional strengths of a composite ply, using computational micromechanics is a rapidly expanding field of research.

#### 2.3.1. Development of micromechanics

The fundamental assumption for the implementation of the micromechanical approach (Hill, 1963) is that an apparent homogeneous stress state of a mixture can be resolved into inhomogeneous stress fields at the microscale. Thus, a “representative volume” was proposed by Hill (1963) to analyse the microscale stress field. The volume is required to be typical of the whole mixture on average and one which contains enough inclusions for resultant moduli to be independent of applied surface traction or displacement. The regular cylindrical shape of carbon fibres and the good adhesion of epoxy resin to carbon fibres means that a composite material is well suited to the representative volume approach. Reiterating the requirements of Hill in terms of fibrous composites, two considerations need to be addressed before analysis can be carried out; firstly, the macroscale fibre volume fraction of the material requires accurate reproduction at the microscale and, secondly, the volume under consideration...
2.3 Micromechanical modelling of Composite Failure

![Figure 2.2: Illustration of homogenisation and repeating unit cell.](image)

is required to be of sufficient dimensions to ensure effects of edge applied boundary conditions are negligible. Upon verification of these conditions, it is possible to obtain macroscale material properties of a composite ply, using the properties of the constituent microscale components.

The concept of a Repeating Unit Cell (RUC) is introduced, as it assumes that a material can be represented through an infinite array of identical cells. This is illustrated in Figure 2.2, where a material showing a random distribution of inclusions is represented using the repeated geometry of a small region, to approximate the material characteristics of the original material. Material data are extracted from the cell through a homogenisation procedure, carried out over the volume of the representative volume.

Homogenised results are calculated from the representative volume by averaging the individual stress and strain values ($\sigma_{ij}$ and $\epsilon_{ij}$ respectively) over the representative volume. This produces an average macroscopic stress ($\bar{\sigma}_{ij}$) and strain ($\bar{\epsilon}_{ij}$) as follows (Sun and Vaidya, 1996):

$$\bar{\sigma}_{ij} = \frac{1}{V} \int_V \sigma_{ij} dV \quad (2.1)$$

$$\bar{\epsilon}_{ij} = \frac{1}{V} \int_V \epsilon_{ij} dV \quad (2.2)$$

where $V$ is the volume of the representative cell.

Representative Volume Elements (RVEs) for composite materials were proposed, ini-
2.3 Micromechanical modelling of Composite Failure

tial versions of which consisted of a central fibre, surrounded by a square or hexagonal array of fibres, which use the concept of an RUC to assume that the fibre distribution conforms to the square or hexagonal arrangement throughout the material (Drago and Pindera, 2007) (see Figure 2.3 (a) and (b), from Keane (2009)). It is possible to use such regular arrays to extract accurate elastic responses at a relatively small computational cost.

The boundary conditions applied to the representative volume are an extremely important factor to be considered. Drago and Pindera (2007) provide a summary of different applicable boundary conditions. Three boundary deformations were investigated; homogeneous displacement, homogeneous traction and periodic conditions using a single fibre and a thirty six fibre RUC, arranged as a square array. Homogeneous displacement and traction boundaries induced edge effects to the RUC. However, through the use of a large number of fibres, the edge effects became negligible, and the central region of the RUC responded as a representative volume.

Periodic boundary conditions specify that the stress state of a periodic RVE is such that, when placed immediately adjacent to itself, at any value of applied strain, no discontinuity is detected between the edges of the RVE. It is also shown by Drago and Pindera (2007) that increasing the size of an RUC subjected to homogeneous traction or displacements produced results which tended towards those of smaller RUCs conforming to periodic conditions.

Using periodic square and hexagonal arrays, accurate predictions were produced for global elastic properties. However, once damage was considered, the distribution of the fibres was found to be an important factor. It was shown in Trias et al. (2006) that periodic square or hexagonal unit cells (as shown in Figure 2.3 (a) and (b)) underestimate the load to cause initiation of damage at the microscale. Arrays using regularly arranged fibres neglect the random spacing between fibres, and the associated interfacial stress concentrations that are found in a real composite microstructure, as seen in Figure 2.3c. Hojo et al. (2009) also investigated fibre arrangement irregularities and found that the inter-fibre distances and relative angle between neighbouring fibres and
Figure 2.3: Idealised fibre distributions in (a) and (b), compared to a distribution created using the NNA in (c) and an actual fibre distribution in (d).
the loading axis have a significant effect on the interfacial stress at the fibre-matrix interface. These studies demonstrate the requirement for representative volumes to reflect phenomenological fibre distributions, in addition to Hill’s original requirements, once damage is considered.

Large microstructures were initially developed using randomly placed fibres. The Random Sequential Adsorption (RSA) technique is the most popular of such random fields. This approach produces a fibre distribution which places fibres in an area as non-overlapping disks, whose centres are randomly assigned in the area. Fibres are placed in the region until a required fibre volume fraction is achieved. This model was shown to experience a jamming limit, where the technique could not achieve a fibre volume fraction exceeding approximately 54% (Vaughan and McCarthy, 2010). High strength composite materials regularly exceed this volume fraction. Therefore further development was required. The RSA was extended through the development of a stirring technique in Melro et al. (2008) and a shaking technique in Wongsto and Li (2005), which were implemented so that the fibres placed using the RSA technique are all displaced slightly, moving them closer to each other, allowing more fibres to be placed in the same area. As summarised in Vaughan and McCarthy (2010), these approaches are effective in generating the required volume fractions, however, they are computationally expensive.

In Vaughan and McCarthy (2010), the Nearest Neighbour Algorithm (NNA) was developed, based on statistical analysis of measurements taken from micrographs of a cross-section of a composite material (see Figure 2.3c). Statistical distributions of fibre diameters, along with the distances between each fibre and its first and second nearest neighbouring fibres were measured from a sample of an actual composite specimen. An algorithm was developed, which places an initial fibre randomly in an area, the diameter of which is assigned from the statistical distribution of fibre diameters. Starting from the initial fibre, second and third fibres are displaced from the initial fibre using distances assigned from the first and second nearest neighbour distance functions. The algorithm then moves onto the second fibre and places two more fibres similarly.
2.3 Micromechanical modelling of Composite Failure

This process is repeated until there is insufficient space for another fibre to be placed within the area. Fibres which crossed the boundary of the area were completed on the appropriate opposing face, honouring the periodic boundary condition. Using this technique, sufficiently high fibre volumes were produced to be considered representative of high-strength composite materials. When analysed, the resulting fibre distribution was found to recreate the phenomenological geometric features of a real composite and also correlate with the first and second nearest neighbour functions of the composite material. A sample fibre distribution, from Vaughan and McCarthy (2010) is shown in Figure 2.3d, where it can be seen that clusters of fibres and resin rich regions (regions without fibres) are created using the algorithm. It is clear that this figure provides a good correlation to the actual distribution in Figure 2.3c.

2.3.2. Material Properties

The properties of the constituent phases of the composite material determine the response of the microstructure to an applied deformation. It is important to identify and apply appropriate material properties to capture the correct damage behaviour during loading in different orientations, as shown in section 2.2.

Cracks through the epoxy material play a large part in the final failure appearance of a composite material. However, Hojo et al. (2009) point out that the transverse fracture strain of a composite material is often much less than that of a neat resin sample. It is also commonly noted that clean fibre surfaces are typically visible on failure surfaces of composite samples. Images from Hobbiebrunken et al. (2006), presented in Figure 2.4 are the best example which show that fibre-matrix debonding is clearly visible early in the damage progression, and the matrix is shown to withstand large deformations prior to failure of the matrix regions. A variety of approaches have been taken to model the mechanical response of the constituent materials within composite RVEs, a selection of which are presented in the following section.
2.3 Micromechanical modelling of Composite Failure

Figure 2.4.: Separation of the fibre-matrix interface in a carbon-fibre composite. From Hobbiebrunken et al. (2006).

Matrix elastic-plastic properties

The elastic-plastic properties of the matrix region are important considerations in micromechanical modelling of composite materials. It is known that polymer yielding is sensitive to hydrostatic pressure (Fiedler et al., 2001, 2005, Quinson et al., 1997), therefore, the selection of an appropriate yield criterion is essential for producing accurate microscale results.

The standard von Mises criterion is an example of a yield criterion which does not completely account for hydrostatic stress. The criterion can be expressed in terms of the three principal stresses, $\sigma_1, \sigma_2$ and $\sigma_3$, as (Lubliner, 1990):

$$
(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_1 - \sigma_3)^2 = 6\tau_c^2
$$

(2.3)

where $\tau_c$ is the yield stress in shear. Considering an equal increase in all three principal stresses will not have any effect on the yield point according to the von Mises criterion, despite an increase being seen in hydrostatic stress. Plotted in the $\sigma_1 - \sigma_2 - \sigma_3$ stress space, the yield surface is a right circular cylinder, equiangular to each of the axes, as shown in Figure 2.5a.
Applying plasticity theory to failure of materials such as soil, rock and concrete involves consideration of hydrostatic stresses. It is therefore convenient to adapt criteria from the field of geological research to the case of polymer epoxies. Jiang and Xie (2011) refer to the Mohr-Coulomb criterion as one of the most popular strength theories in geotechnical engineering. The criterion predicts failure at a point in a body if the shear stress ($\tau$) and normal stress ($\sigma_n$) at that point achieve a critical combination. The Mohr-Coulomb criterion can be written in the form of Equation 2.4 (Jiang and Xie, 2011).

$$\tau_c = \tau + \sigma_n \tan \phi$$  \hspace{1cm} (2.4)

where $\tau_c$ is the cohesion (yield stress under pure shear), and the angle $\phi$ is the internal angle of friction of the material. Expressed in terms of principal stresses, Lubliner (1990) gives the criterion as Equation 2.5.

$$f(\sigma_1, \sigma_3) = (\sigma_1 - \sigma_3) + (\sigma_1 + \sigma_3) \sin \phi - 2\tau_m^c \cos \phi = 0$$ \hspace{1cm} (2.5)

Plotted in the $\sigma_1 - \sigma_2 - \sigma_3$ space, a hexagonal based pyramid is formed, as shown in Figure 2.5b. Here, in comparison to Figure 2.5a, the dependence of the criterion on hydrostatic stress is illustrated, and yielding under tension occurs at a lower stress.

---

**Figure 2.5.:** Failure surfaces.
2.3 Micromechanical modelling of Composite Failure

than in compression.

Jiang and Xie (2011) highlight the sharp corners of the failure surface as sources of convergence difficulties in finite element analyses, for which the Drucker-Prager criterion, a modified version of the von Mises criterion (Jiang and Xie, 2011), has been proposed. This criterion provides a smooth yield surface, which incorporates a dependence on hydrostatic stress. In the case of composite micromechanics, the Mohr-Coulomb criterion is the more common choice. This is as a result of the Drucker-Prager criterion being employed as an approximation to the Mohr-Coulomb criterion to introduce hydrostatic stress dependence to the von Mises criterion. This causes the Drucker-Prager failure surface to circumscribe or inscribe a smooth conical shape to the Mohr-Coulomb failure surface pyramid. Chen and Han (1988) note that this introduces an over- or under-estimation, respectively, of the yield point of the material and therefore is less desirable than the original Mohr-Coulomb criterion.

**Fibre-Matrix interface properties**

It is well known that the separation of the fibre and matrix phases is a critical factor contributing to the failure behaviour of composite materials. This was demonstrated in Hojo et al. (2009), where it is highlighted that the strain to failure of the composite material is much lower than that of the constituent matrix material, which is attributed to the role of fibre-matrix debonding. Fracture at the fibre-matrix interface is therefore identified as a critical initiation point for damage in composite materials, and occurs prior to failure of the matrix. Figure 2.4 shows the progression of damage at the fibre-matrix interface during failure. The clean separation of the two phases is clear. Therefore, this mechanism may be represented by cohesive zone modelling. Numerous examples of traction-separation controlled cohesive zones at the fibre-matrix are available in the literature (Canal et al., 2009, González and Llorca, 2007, Segurado and Llorca, 2006, Totry et al., 2010, Vaughan and McCarthy, 2011a, b). When applied to an interface between two regions, traction-separation laws relate the traction across the faces of the region to the separation of the faces. The zone uses a linear elastic
relationship to describe the relative displacement of the faces, until a critical traction is reached. After this point is reached, the stiffness of the zone is degraded, through a relationship with the fracture energy of the interface, until the stiffness reaches zero, when no further interaction between the faces occurs. Further details of the method are presented in Chapters 4, 5 and 6, and are therefore not discussed further here.

2.3.3. Micromechanical studies

Using the NNA, RSA and random fibre distributions, extensive FEA of the microscale composite failure process under different loadings have been carried out. Examples of published investigations regarding failure behaviour under various loading conditions and numerical approaches are summarised in this section. This section focuses mainly on models containing elastic-plastic matrix properties, and fibres surrounded by cohesive zones.

Alfaro et al. (2010) used a model which incorporates cohesive elements in a (FE) mesh of randomly placed glass fibres in an epoxy matrix. Cohesive elements were placed around every fibre in the microstructure, and in addition, each element in the matrix region was surrounded by cohesive elements. A traction-separation law was used to reduce the stiffness of the cohesive elements once a pre-set traction across the elements was reached. Transverse tension was applied to an appropriately meshed RVE, and good phenomenological failure behaviour was produced. It was found that imperfections introduced to the microstructure deliberately, in the form of debonded fibres, behaved as nucleation sites for crack initiation which propagated through the matrix region. Images from this study are shown in Figure 2.6, where initiation of two cracks at fibre boundaries are purposely inserted prior to loading. In Figure 2.6a, one crack is seen to dominate, and provides the crack path through the RVE. Different defects in Figure 2.6b resulted in the final failure pattern incorporating both defects. Homogenisation of the results showed that higher fracture energy resulted from the second scenario. It was noted that the energy in the second scenario also exceeded
2.3 Micromechanical modelling of Composite Failure

that of the case where no defects were considered. The longer crack path created when both defects became involved in the final failure pattern increased the fracture energy. It is concluded that the presence of imperfections, and a diffuse damage process could have a positive effect on the fracture toughness of the RVE. It should be noted that this method proved to be sensitive to the mesh size, as a large mesh did not reflect the required phenomenological crack patterns, while an over-refined mesh caused the initial stiffness of the elements to contribute significantly to the results.

An alternative damage model used by Grufman and Ellyin (2008), involved an “element death” procedure, using elastic material properties. This involved the deletion of elements once a predetermined failure strain was exceeded in the element (deleting elements involved reducing the stiffness of the elements by a factor of $10^6$). After deletion of the appropriate elements, the solution was resubmitted to the solver, at the same increment. The mesh was analysed multiple times at each increment, with elements deleted at each iteration. The solution only advanced to the next increment once no elements were required to be deleted in the current increment. The process continued until final failure occurred. Under transverse tension, it was found that multiple damage sites were created, dependent on local microstructural features. This result was used to highlight the importance of fibre distributions, as a random distribution was found to produce a low strain to failure compared to a regular square array. The damage model was used to investigate the influence of RVE size on the resultant data. The investigation is an example of an RVE which does not employ periodic boundaries, and requires boundary conditions to be applied to a homogeneous region of material surrounding the RVE. To reduce the influence of this region on the homogenised data, a large RVE is required, and is therefore a less efficient RVE configuration than one employing periodic boundaries.

A different configuration for analysing microscale damage processes of unidirectional composite materials involves the creation of a random fibre distribution, using elastic fibre properties. The matrix region is assigned elastic-plastic material properties, while the fibre is connected to the surrounding matrix through a cohesive zone,
Figure 2.6.: Stress distribution in the initial and failed configurations (a) shows a crack path where one defect remained independent of the final failure path (b) shows a crack path which included both initial defects. Adapted from Alfaro et al. (2010).
defined by a traction-separation law. Such models have been used extensively to model different loading conditions, and a brief summary of a selection of such investigations follows.

Loading in the fibre direction is generally not approached using random fibre microstructures. This is due to the fact that much of the load is carried through the stiff fibres and failure occurs due to fibre rupture. Consideration of this mode of failure requires a model with a large dimension in the fibre direction, in order to capture the full extent of the damage process zone. Extension of a typical microstructure in this direction greatly increases the computational cost of the simulation. This case is of limited interest, as it is dominated by the fibre response. This is not the case in the transverse plane, which can be represented efficiently in two-dimensional models. Tension, compression and shear loadings produce different damage mechanisms when considered at the microscale and a summary of investigations in the transverse plane are presented in this section.

Transverse compression of composite microstructures was addressed by González and Llorca (2007). Plasticity was modelled using the Mohr-Coulomb criterion and fibre-matrix debonding behaviour was controlled using a traction-separation law, following a maximum stress criteria. The models showed excellent correlation to experimental micrographs of failed composite samples. Figure 2.7a shows a micrograph of damage in the form of fibre-matrix decohesion, concentrated along intense bands of plasticity in the matrix (shear bands), aligned at an angle to the applied compressive load. Analysis of the load configuration using micromechanics showed that the shear bands occurred at angles related to the Mohr-Coulomb friction angle of the matrix, \( \phi \), at approximately \( \pm (45^\circ + \frac{\phi}{2}) \) to the loading axis, as shown in Figure 2.7b, where \( \phi = 15^\circ \). The fracture energy of the interface was found to have a negligible effect on the compressive strength. Using similar models, Totry et al. (2008a) combined transverse compression with transverse shear. In the case of combined loading, similar damage processes were identified to those found under compressive failure. It is concluded that very similar damage processes dominate shear and compression loading, and it was
2.3 Micromechanical modelling of Composite Failure

![Image](image_url)

(a) Micrograph of a shear band, formed under transverse compression.

(b) Plastic strain in a microstructure under transverse compression, showing shear bands.

**Figure 2.7.** Comparison of shear bands in an actual composite and predicted formation in an RVE, under the same loading conditions, adapted from González and Llorca (2007).

verified that the loading path to failure (i.e. compression followed by shear or shear followed by compression) did not significantly affect the failure behaviour or strength results.

Damage under transverse tension and the effects of thermal residual stress were investigated by Vaughan and McCarthy (2011a). The Mohr-Coulomb criterion was used to define matrix plastic yielding and fibre-matrix debonding was controlled by a traction separation cohesive law. Parameter studies were carried out on the interfacial strength and fracture energy parameters of the interfaces. Thermally induced compression of the interface, while beneficial at high levels of interface strength caused debonding to occur at low levels of interfacial strength. Matrix plasticity and fibre-matrix debonding, found previously by Totry et al. (2008a) under transverse compression and shear, were also shown to cause failure in the transverse tensile case. Debonding was found to occur along a plane perpendicular to the loading axis, as found in the studies using matrix damage laws (Alfaro et al., 2010, Grufman and Ellyin, 2008), under the same loading. Matrix plasticity was found to be concentrated between the debonded fibres. In Vaughan and McCarthy (2011a), it is shown that the strength of the composite under transverse tension is highly dependent on the fibre-matrix interface strength.
2.4 Adhesive composite joints

The literature shows that current micromechanical models are capable of recreating the failure behaviour of composite materials with respect to stress-strain results and phenomenological failure appearance. The most promising approach is the use of a hydrostatically sensitive elastic-plastic matrix accompanied by a traction-separation damage law at the fibre-matrix interfaces. This approach has produced consistently accurate simulations, and reduces the mesh sensitivity, in comparison to studies involving damage modelling of the matrix.

2.4. Adhesive composite joints

Size constraints on the manufacturing process of composite materials make joining of components a necessary part of any major composite structure. The two methods available for joining composite components are mechanical fastening and adhesive bonding. The traditional choice is mechanical fastening, using rivets or bolts. However, adhesive joints, while not advantageous in all applications, can provide advantages over the mechanically fastened option. Advantages and disadvantages of the joining techniques include:

- Adhesive joints transfer the load between the two adherends over a larger area, and therefore provide a reduced stress concentration.
- Bolts or rivets are heavier than the corresponding adhesive.
- Adhesive bonding does not introduce damage to the composite adherends prior to bonding, as there is no prerequisite for a hole to be drilled through the adherends prior to bonding.
- Adhesive bonding requires careful preparation of the surfaces to be bonded and negligence in this area can be detrimental to joint performance.
- Tightening of bolts can cause damage in transverse compression during assembly, which is not an issue in adhesive bonding.
2.4 Adhesive composite joints

- Adhesive joints fail catastrophically, while bolted joints can be designed for progressive failure, allowing greater energy absorption, and opens the possibility to identify damage prior to final failure of a joint.

- Bolted joints have been the focus of extensive research and resulting simulations are extremely advanced. This contrasts with a lack of confidence in adhesive bond modelling, leading to a tendency to overdesign adhesive joints.

- Better sealing between adherends can be achieved using adhesive joints.

The advantages of adhesive bonding are sufficient to drive an increasing amount of research in the area. Of particular interest are the areas of surface preparation, test geometries and the development of numerical approaches to predict failure. Here, relevant examples from the literature are presented.

2.4.1. Surface treatment

Surface treatment of the adherends is an important requirement for all adhesive joints, and achieving a high quality adhesive bond is dependent on producing well prepared surfaces for bonding, regardless of adhesive material choice. Good surface preparation improves the interfacial contact of the surface and the adhesive, allowing the establishment of interatomic and intermolecular forces between the adhesive and the adherend in addition to mechanical interlocking. Kinloch (1987) highlights the importance of surface preparation, to enhance the interaction between the adhesive and the adherend by smoothing surface imperfections and defects and removing grease and oil from the surface, which may cause a weakened boundary layer, creating a “weak link” at the interface of the adhesive and adherend. Banea and da Silva (2009) attribute the failure of most adhesive bonds to poor fabrication processes, with surface preparations forming the most critical factor. Industry standards for preparation of composite surfaces consist of mechanical abrasion followed by removal of grease and dust from the abraded surface using wipes, impregnated with acetone or similar
2.4 Adhesive composite joints

2.4.2. Composite-adhesive joints: Experimental tests

Two modes of deformation dominate the analysis of the stress state in adhesive bonds, Mode I (peel) and Mode II (shear). These are illustrated in Figure 2.8 in terms of an adhesive single lap joint, under tensile loading. As noted by Kinloch (1987), this represents the most common practical configuration for an adhesive bond, but the stress distribution is not uniform along the bonded length. Tests are commonly carried out to produce separate modes of deformation to quantify the contribution of each mode to the failure behaviour to a more complicated stress state.

Kim et al. (2006) tested single lap joints, loaded in tension, to investigate the failure mode and strength of the joints while varying adhesive and bonding approaches. The difference in failure process between an adhesive bond between metals and composites is highlighted. Failure of a joint with metal adhesives is generally proportional to the adhesion strength of the adhesive, where failure is confined to the adhesive layer or the adhesive-adherend interfaces. Composite joints behave differently, as through thickness damage of composite laminates is a significant source of failure. In Kim et al. (2006), two different epoxy paste adhesives were tested, and compared to an epoxy film adhesive. Crack propagation was found to occur predominantly at the composite-adhesive interface, with some carbon fibres found to be pulled out of the composite adherend. Images of the appearance of multiple damage processes are provided, and reproduced here in Figure 2.9. Analysis of the fracture surfaces showed that damage
2.4 Adhesive composite joints

Figure 2.9.: Failure surfaces (adapted from Kim et al. (2006)), showing three damage mechanisms on a single failure surface.

occurred in the adhesive, at the adhesive-composite interface and within the composite adherend. Increasing surface roughness was found to increase the amount of fibres pulled from the composite, while also decreasing joint strength, as the delamination mode of failure provides a weak point in the assembly. The strongest bonds were found to be obtained by preparing the surfaces with fine sandpaper, thus providing a regular, but low degree of surface roughness. Decreasing the bondline thickness was also found to increase joint strength.

Due to bending of the adherends during testing of single lap bonded joints, a high opening Mode I stress is generated at the ends of the lap, meaning that, a true shear condition is not generated in the single lap joint. This has led to the development of tests which separate the peel and shear modes of failure. The most popular method for testing of Mode I failure is the Double Cantilever Beam (DCB) test geometry. In Ashcroft et al. (2001), DCB testing was used to separate a unidirectional layup of sixteen plies from an adhesive (a 0.2 mm thick epoxy film supported by a nylon carrier). A starter crack was created using a PTFE insert, and tests were carried out at a range of temperatures. At room temperature, a stick-slip pattern was clearly formed on the surface. Microscopy showed that cracks propagated through the top ply of the composite,
immediately adjacent to the adhesive layer. Fibres were revealed at the ply surface, while fibre imprints in the matrix material remained on the adhesive surface. It was concluded that the continuous fibres prevented the cracks propagating further into the composite. Figure 2.10 shows an SEM micrograph of a small region of the failed surface of a specimen which was tested at high temperature (90°C). This image is an example of different failure types occurring on the same bondline at the microscale. Clean fibres are noted as areas where the matrix has been removed from the top of the fibres. One fibre is seen to have been damaged, while regions of composite-adhesive interface failure and regions of adhesive failure are also visible.

Vaidya et al. (2006) highlight the fact that, under tensile loading of a single lap joint, peel stress is more detrimental to joint performance than the shear stress component. A comparison of the stress state at the midplane of an adhesive joint undergoing transverse bending and tensile loading is made. These plots are provided in Figure 2.11. Under in-plane loading, the peel and shear stress distributions are almost symmetrical (Figures 2.11b and 2.11d respectively), with positive peaks of shear and peel stresses occurring at the ends of the lapped area. Under transverse loading, it is clear that, while concentrations of peel and shear stress occur at the ends of the lap, there is now one positive, and one negative maximum in both cases (see Figures 2.11c and 2.11e). The high compressive peel stress (positive in Figure 2.11c) suppresses local...
2.4 Adhesive composite joints

Figure 2.11.: Plots of the stress state through the mid-plane of a single lap joint, from Vaidya et al. (2006).
2.4 Adhesive composite joints

Mode I crack growth at one end, and at the other end of the lap, Mode I cracks are promoted by the high opening, peel stress (negative in Figure 2.11c). Similarly, Khalili and Pirouzhashemi et al. (2008) show that the peel stress at the joint edges initiates failure in the tensile lap shear case through numerical analyses. A companion experimental paper is presented in Khalili and A. Shokuhfar et al. (2008). The focus of the investigation was on the influence of additions of fibres and micro-glass powder to the adhesive. Optical microscopy showed a range of failure types (see Figure 2.12) occurring on the same failure surface. Here it can be seen that the damage process included failure within the composite (fibre tear failure), as deduced from the presence of visible fibres, as well as failure within the adhesive (cohesive failure).

Importantly, experimental analyses in the literature indicate that a variety of failure modes are triggered during failure of an adhesive bond and the presence of multiple failure modes on a single fracture surface is not uncommon. This feature is also identified in the review paper of Banea and da Silva (2009), as an implication for the modelling of adhesive joint failure, which is detailed in the following section.

2.4.3. Composite-adhesive joints: numerical studies

Numerical models of bonds between composite materials are complicated by the laminated nature of the composite, and the heterogeneous structure of the composite
2.4 Adhesive composite joints

at the microscale. Separation of plies or surface fibres can cause failure away from the adhesive material during the damage process. For this reason, Banea and da Silva (2009) conclude that an accurate model of a bonded composite joint must be able to predict failure of the adhesive, adhesive-adherend interface and damage of the surface plies of the composite.

The problem is generally approached at the macroscale, an example of which is found in Kim et al. (2008) for a single lap joint, loaded under in-plane tensile conditions. Experimental results from a previous publication (Kim et al., 2006) showed the need for two failure criteria to be active in this configuration; failure of the adhesive, which included interfacial failure at the composite-adhesive interface, and delamination failure of the composite. Failure in the adhesive was proposed to be represented by an elastic-plastic material model, and final failure was deemed to have occurred once the entire adhesive region yielded plastically, and deformation occurred without any further increase in applied load. The yield point of the adhesive was not taken as the yield point of the adhesive material, but as that of an effective ultimate stress for the thin adhesive layer, calculated through correlation with the previously published experimental data. The new yield point was found to be 30% higher than that of the bulk adhesive, attributed to the interaction of the adherends with the adhesive in the thin layer of adhesive. Delamination of the composite adherend was represented by a quadratic delamination criterion, based on through thickness and shear stress components in the composite. The model was used to compare the onset of delamination and adhesive failure, and good correlation was produced with the experimental data. Figure 2.13 was produced using the modelling results showing that increasing the adhesive strength causes the strength of the bond to rise to a maximum, after which the joint strength reduces slightly to a plateau, where delamination failure of the composite dominates. It was concluded that the maximum joint strength occurs when delamination and adhesive failure occur simultaneously.

Cohesive zone models are frequently used to predict adhesive joint failure. Crack path selection becomes an issue with this method, as the locations of the cohesive ele-
Figure 2.13.: Correlation of failure mode and strength of composite adhesive joints, from Kim et al. (2008).

ments dictate the crack direction. It was shown by Evans et al. (1989) that the choice of test method and test specimen can influence the crack path. Experimental results show that cracks often develop along the adhesive-composite interface, before diverting into the composite, (Feih and Shercliff, 2005), meaning that great care is needed when using a layer of cohesive elements to represent the adhesive region. This issue is addressed by Li et al. (2006) through re-meshing of the model using extra cohesive elements in the composite region, once damage initiation in the region was predicted. This method showed a good match to experimental tests, and it is concluded that the characteristic strength of the cohesive zone model is a useful parameter for predicting crack initiation locations. A slightly different solution was used by Sörensen et al. (2009), here, cohesive elements formed the adhesive-composite interface, and the parameters of the cohesive zone accounted for fibre bridging and failure within the composite, in addition to interfacial failure. This method produced good correlation to experimental data of the complete failure of the specimen. Goyal et al. (2008) used a similar model, implemented using a traction-separation controlled cohesive behaviour at the adhesive-composite interfaces. Again, it was found that the method provided excellent correlation to experimental data under lap shear, DCB and cracked lap shear conditions.
2.5 Summary of literature review

Failure of adhesive joints is not regularly approached at the microscale. This is highlighted by Reina-Romo and Sanz-Herrera (2011), who provide one of the few exceptions, where a particle reinforced adhesive was modelled at the microscale. This allowed the investigation of different particle volume fractions, as this method allowed the discrete modelling of the particles. Deformations of the composite adherends were neglected, as was the pressure sensitivity of the adhesive matrix material. This work was intended as an initial study into the method, and recommendations are made for: extension of the model into three dimensions, analysis of imperfect bonding of the particles to the matrix, and the constraint effects and deformations resulting from consideration of the heterogeneous adherends.

Microscale mineral clusters in a polymer adhesive are also investigated by Salomonsson and Andersson (2008). In this study, the failure of the adhesive was attributed to the microcracking process at the microscale, which was simulated, similarly to Alfaro et al. (2010), through the insertion of cohesive interfaces between every continuum element in the mesh. Pressure sensitivity is assumed to be considered through the cohesive law, which does not consider compressive traction in the damage initiation criteria. The RVE was generated through analysis of images of the material, as regions of mineral clusters were identified using high-magnification SEM images. Periodic conditions were applied to the sides of the RVE and constant displacements were applied in peel and shear to the top edges. The model was tuned to the response of experimental tests. A good match to the experimental data under peel conditions was found, while the correlation to the shear data was not as strong, however, realistic deformation patterns were found, an example of which is shown in Figure 2.14, under peel conditions.

2.5. Summary of literature review

The damage response of composite materials is acknowledged to be an extremely complex issue and one which needs careful consideration in order to produce accur-
2.5 Summary of literature review

Figure 2.14: Results from an adhesive microscale RVE under peel loading, compared to an in-situ SEM micrograph of a DCB specimen, loading is in the vertical direction in both cases. (Salomonsson and Andersson, 2008)

ate numerical predictions of failure behaviour. While currently at an advanced stage, continuum models are capable of considering strength values from different damage mechanisms, but are incapable of resolving the specific microstructural failures which coalesce to create a global failure. Approaching the issue using micromechanics allows for resolution of these micro-damage processes. The first section of the literature review presented significant results from the composite micromechanics method.

Initial approaches, using idealised unit cells containing limited numbers of fibres, produced excellent elastic responses at the macroscale, through homogenisation of microscale results. It was subsequently shown that the unit cell approach was limited to the elastic case, as shown in studies which focused on the distribution of the fibres within composite plies. Variation of inter-fibre distances, which causes stress concentrations to occur, is a non-negligible effect once damage is considered. The stress concentrations are the result of reduced fibre spacings, and local increases in volume fraction due to clustering of fibres. The NNA (Vaughan and McCarthy, 2010) provides an excellent method for calculation of fibre positions that produces high fibre volume fractions, and reflects the random patterns exhibited by the actual microscale fibre placement in a real composite, for use in microscale models.

Using randomly distributed fibres in a periodic microstructure, it was possible to accurately study the damage behaviour of composites at the microscale. Approaches using damage models included: element deletion procedures and the use of cohesive elements placed between continuum elements. These approaches produced good responses in comparison to the experimental behaviour; however, great care needs to be taken to ensure mesh independence. A more accepted approach is the use of cohesive
2.5 Summary of literature review

elements to capture separation of the fibre and matrix. The debonding of the fibre-
matrix interface provides the loss of load-bearing capacity of the composite and it is
recommended to model the matrix using a pressure sensitive elastic-plastic material
model. This approach has repeatedly provided excellent correlation to experimental
data, in terms of stress strain results and the phenomenological failure appearance
under a variety of loads.

Adhesive joints have been addressed in the second section of the literature review.

Bonded joints are currently over-designed due to a lack of confidence in techniques
to accurately predict their failure behaviour (Banea and da Silva, 2009), but the ad-
vantages of the bonded joint over bolted and riveted alternatives make it a worthwhile
area for further research. The single lap joint is the most common configuration for
adhesive joints and produces a variation of stress state along the adhesive layer. The
dominant stresses are peel and shear stress, with concentrations of each at the ends of
the lapped area. Experimental studies, therefore, have to consider the contributions
of each of the stress states to the overall results. The experimental tests reviewed here
show a wide range of failure behaviours. Damage is shown occurring within the adh-
erend, within the adhesive, at the composite-adhesive interface and failure processes
are shown progressing from one to another. Results show that failure can involve fibres
being torn out of the adherend and deposited on the adhesive surface, and over a small
area, this failure can be accompanied by an interfacial failure and failure through the
adhesive.

Numerical solutions to adhesive joint failure have also been reviewed. Typically,
damage behaviour is captured using a cohesive zone model at the macroscale. It is
possible to assign properties to the cohesive zones which represent different failure
processes, allowing a simple cohesive zone model to produce good correlation with
the experimental data, regardless of the failure type. A less common approach is to
consider the microscale heterogeneous properties of adhesives which contain relativ-
ely large toughening particles. Similarly to the composite ply case, it was possible
to homogenise the results of the microscale models, to extract macroscale results. No
2.5 Summary of literature review

study exists, however, which considers the heterogeneous nature of a composite adherend at the microscale.

This thesis builds on the literature presented here, to develop a microscale representation of the interface between an adhesive and composite adherends. The model will be subjected to the dominant deformation modes experienced at the interface of the composite and adhesive in a loaded bonded joint. It is intended that the model will capture microscale interactions between the adhesive and the heterogeneous composite adherend, which is conspicuous in its absence from the literature. A new testing technique, suitable for high resolution of the damage progression in adhesive bonds will also be developed, and the results are considered using the standard failure descriptions for such tests. This will enable the comparison of the results of the tests and the reviewed literature. The results of the microscale model will be homogenised for use in a macroscale model. Analysis of the macroscale results in comparison to the results of the experimental testing regime, will link the micro- and macro- scales.
3. In-situ SEM Mechanical Testing of Miniature Bonded Joints

3.1. Introduction

Identification and understanding of the failure processes which occur during failure of an adhesive bond between composite materials will help improve the design process for this joint type. Adhesive joints produce a less severe stress concentration than bolted joints, as load transfer occurs over a larger area (Ashcroft et al., 2001, Kim et al., 2006). Numerical studies of adhesive lapped joints have investigated the stress field in the adhesive, and shown a stress state consisting of a combination of peel (Mode I) and shear stresses (Mode II) acting through the adhesive layer.

A common adhesive joint test configuration is the tensile lap shear joint. Numerous failure types have been observed occurring during failure (Kim et al., 2006, 2008, Song et al., 2010). This test, while providing a practical load case, is known to produce high mode mixity due to large peel stresses in the adhesive layer, making analysis of the experimental results difficult (Vaidya et al., 2006, Cognard et al., 2012).

Double cantilever beam (DCB) and end notch flexure (ENF) tests are popular methods of examining Mode I and Mode II failure behaviour separately. DCB tests conducted by Ashcroft et al. (2001) produced failure in the composite ply, adhesive layer and at the adhesive-adherend interface. The failure types were observed, post failure,

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1The results of this chapter have been accepted for publication as a peer-reviewed article in International Journal of Adhesion and Adhesives and is currently in the review process.
using a Scanning Electron Microscope (SEM). DCB and ENF tests were also used to investigate delamination of composite plies, and evidence in post-failure SEM images show the contrast between Mode I and Mode II failure types. Post-failure SEM analysis is regularly carried out on the failure surfaces of adhesive joints. Capturing of damage progression in adhesive joints prior to failure is difficult as the size and accessibility constraints of SEM operating chambers do not allow large samples or standard test platforms to be operated on. It is shown in Ting et al. (2006) that good quality SEM images of damage progression are attainable through interruption of tests. Images of the failure of polyethylene was captured using a previously developed technique to maintain the loading state during transfer from the test rig to the SEM. The analysis was very effective in demonstrating damage progression at different constraint conditions and loading rates.

Transverse bending of adhesive single-lap joints, a somewhat less popular test, has been carried out by (Vaidya et al., 2006, Grant et al., 2009). Vaidya et al. (2006) show the shear and peel stresses through the adhesive layer during bending and tension for single-lap joints. The peel stress at the end of the adhesive lapped area was found to be amplified under bending in comparison to tensile tests. Adhesive joints with steel adherends were tested in three- and four-point bending by Grant et al. (2009). It was found that under three-point bending, failure occurred at a constant bending moment at the edge of the overlap, where failure initiated. Four point bending was found to cause yielding of the adherends away from the lapped area, and as such, was not a good test of joint failure.

Under tensile loading, the lap shear joint geometry provides mixed mode loading with a large positive Mode I component, which drives crack progression. However, if the joint is loaded in compression, the Mode I stress is negative at the lap end and, thus, does not provide a Mode I crack driving force. However, the lap-shear joint is not widely used under compressive loading. A possible reason, suggested by Cognard et al. (2012), is the possibility of buckling occurring before shear failure of the adhesive layer. Cognard et al. (2012) investigated composite tubes, and an FE study was used
to show that the strength of a lap shear joint under compression exceeded that of a similar joint, loaded in tension. This was attributed to the high compressive peel stress at the ends of the overlapped region.

The identification of failure types through microscopy of the failed surface of adhesive joints is an important part of analysing the failure of adhesive joints. ASTM D5573 has been developed to standardise the analysis of composite adhesive joint failure. The seven types outlined in the standard are illustrated in (Banea and da Silva, 2009). The standard has been used to describe the failure of composite joints in (Taib et al., 2006, Avila and Bueno, 2004), and provides a convenient method for comparison of failure types resulting from different test methods and material combinations.

The objective of this chapter is to experimentally identify the microscale damage processes associated with Mode I and Mode II failure of a joint consisting of composite adherends, joined using an epoxy adhesive, through in-situ and post failure analysis. Miniature adhesive joints are tested in three lap-shear bending and lap shear compression configuration to create Mode I dominant and Mode II shear conditions, respectively. The tests are carried out in an SEM chamber, and real-time videos are recorded and analysed. A post failure fractographic analysis is also carried out on the samples.

3.2. Material and test procedures

A Deben 2kN microtest stage was used to carry out the tests in this chapter, and was positioned in the chamber of a Jeol JSM 5600 SEM. In order for the microtest apparatus to fit inside the SEM vacuum chamber, the size of the test platform is extremely small. This results in a size constraint being imposed on the specimen. In addition to the physical constraint on overall size, the range of the SEM was limited, and did not allow images to be obtained over the entire area of the sample. The approximate range of the SEM beam is shown as a red frame in a CAD drawing of the microtester, with a lap-shear bend specimen in Figure 3.1. The test specimens were designed to ensure that the entire adhesive layer fell within the range of the SEM beam. The overall length
3.2 Material and test procedures

Figure 3.1.: CAD representation of the microtester constraints. The red box approximates the scanning range of the SEM.

of the bonded samples was required to be less than 50mm. A schematic of the joint geometry chosen for the experimental testing is shown in Figure 3.2a with dimensions shown in Figure 3.2b.

A unidirectional laminate of HTA/6376 composite material was prepared. The prepreg tape was orientated to create a layup consisting of 16 layers of $0^\circ$ unidirectional plies (a $[0^\circ]_{16}$ layup). The layup followed the configuration showed in Figure 3.3, and was autoclave cured for 175°C for 2 hours. Peel ply was applied to one side, for ease of handling, while the opposite surface ply was laid directly onto the steel table surface.

The surfaces to be bonded were prepared using silicone carbide paper. Care was taken to produce a constant surface roughness. Prior to bonding, the wetted surface was wiped down using degreasing wipes. This removed any dust and loose particles, in addition to any grease or release agent deposited by the peel ply during its removal from the laminate.

The adhesive used was 3M 9323, a two part epoxy designed for high strength and toughness, and cured for the appropriate time at 70°C (3M, 2010). No additive particles (i.e. glass spacing beads or rubber toughening particles) exist in the adhesive. The overlap length was maintained at 5 mm across all samples. Even pressure was
3.2 Material and test procedures

Figure 3.2.: Miniature specimen test geometry. (a) Test configuration (b) Specimen dimensions. All dimensions are in mm. Bondline: 50 μm. Specimen thickness: 7mm.

Figure 3.3.: Layup configuration, temperature, T, and pressure, P, supplied by the autoclave.
applied along the bondline using clamps during the curing process to ensure a constant glueline thickness of 50 microns. Prior to final curing of the adhesive, the spew fillet, produced during the application of pressure to the bondline, was removed using a razor blade.

To allow improved resolution of microstructural details under the SEM beam, all surfaces to be exposed to the SEM beam were prepared. The samples were sanded incrementally using silicone carbide paper with a range in grading from 400P to 2500P. The samples were subsequently polished using 1 micron and 0.05 micron diamond suspensions. To avoid charging of the epoxy adhesive under prolonged exposure to the SEM beam, all samples were gold coated. A 10 nm gold coat, applied using sputter deposition, was found to be sufficient to provide good quality images.

### 3.2.1. Mode I dominant test

The lap-shear bend loading configuration was chosen to generate Mode I dominated failure (see Figure 3.2a). The loading studs were positioned 17.5 mm on either side of the fixed centre stud, which was positioned at the centre of the overlap region.

A linear elastic two-dimensional plane strain finite element (FE) analysis was carried out using ABAQUS software (ABAQUS-Inc., 2010). The model was meshed using approx. 100,000 2D elements (CPE4). The load was applied through analytically rigid studs, with frictionless contact between the studs and the adherend surface. No adhesive spew fillet has been considered for this analysis. Peel and shear stresses were extracted along the centre-line of the adhesive, and the results are presented in Figure 3.4. The values have been normalised by the maximum peel stress and plotted along the length of the overlap. The plots are comparable to those shown previously in the literature review, from Vaidya et al. (2006), where more comprehensive FE studies were carried out on a full size joint subjected to transverse bending loads. The results of the FE study here demonstrate that no new stress concentrations are generated in the smaller geometry. Under transverse bending load, the Mode I and Mode II plots
3.2 Material and test procedures

Figure 3.4: Stress distribution through the midsection of the adhesive layer, Mode I dominant conditions.

in (Vaidya et al., 2006) show \(\frac{\tau_{12}}{\sigma_{22}}_{\text{max}} \approx 0.38\), while the FE analysis here produced \(\frac{\tau_{12}}{\sigma_{22}}_{\text{max}} = 0.31\).

It is clear from Figure 3.4 that there is a large opening (peel) stress at one end of the overlap and a large compressive peel stress at the other. This allows for accurate prediction of the location of crack initiation, thus narrowing the target area for the SEM beam during testing. As noted by Khalili and Shokuhfar et al. (2008), this geometry does not provide pure Mode I conditions. The shear stress component reaches 31% of the maximum peel stress. However, failure of the joint in bending is attributed to the tensile stress concentration at the lap end.

3.2.2. Mode II test

A popular test for determination of bond strength is the tensile lap shear test (Kinloch, 1987). In this configuration, however, secondary bending of the joint induces an opening peel stress at the overlap ends. Reduction of the peel stress component is achieved by using tabs to avoid eccentricity of the applied loads, however, as noted
by Taib et al. (2006), the normal (peel) component of the stress state initiates failure. Therefore, large opening (peel) stress is not acceptable in a shear test.

A compressive lap shear test is carried out here on an identical specimen geometry to the lap-shear bend test. The test setup is shown in Figure 3.2a. The boundary conditions for the test are shown in Figure 3.5, and the specimens were clamped, as shown, over 10 mm. The same FE model as used in the previous section was subjected to an axial compression. The normalised peel and shear stress are plotted in Figure 3.5. It can be seen here that the peel stress at both ends of the adhesive layer is compressive, meaning that Mode I cracks cannot initiate. The presence of compressive peel stress is also suggested by Guo et al. (2006) as a reason for possible higher single-lap joint strengths under compression compared to tension. The shear stress is seen to reach a maximum close to the ends of the lap. Under compression, the possibility of buckling was precluded by the use of thick adherends. The geometry does not form a slender column therefore buckling of the structure is not expected.
3.3. Results

Sixteen identical samples were manufactured, and divided equally between Mode I and Mode II tests. The in-situ SEM camera allows for an edge-on view of crack growth, as illustrated in Figure 3.2a. After final failure of the samples, the surfaces of the broken samples were gold coated and returned to the SEM chamber, with the beam focusing on the failure surface, allowing for a more complete view of the failure to be captured.

Due to the complex nature of composite adhesive joint failure, the standard, ASTM D5573 (2005), has been used here to classify the failure characteristics created during failure. Seven failure types are expected from an adhesive composite joint failure and are described as follows:

- Type 1, Adhesive Failure: separation of the adhesive-composite interface.
- Type 2, Cohesive Failure: failure within the adhesive layer.
- Type 3, Thin-layer cohesive failure: failure close to the interface, within the adhesive, adhesive remains predominantly on one adherend.
- Type 4, Fibre-tear failure: failure within the composite, with fibres deposited on the failed adhesive surface.
- Type 5, Light-fibre-tear failure: matrix material left on adhesive surface after failure, few fibres transferred to adhesive.
- Type 6, Stock-break failure: failure of the adherend away from the adhesive lap.
- Type 7, Mixed failure: any combination of the above failure types.

Table 3.1 provides a breakdown of the Mode I samples by failure type, referencing the above list. All Mode II samples failed in Type 1 failure (adhesive failure at the adherend-adhesive interface) and are not included in Table 3.1.
3.3 Results

<table>
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Table 3.1: Failure types under Mode I dominant conditions following ASTM D5573-99. (Note: all Mode II test samples failed in Type 1 failure)

3.3.1. Mode I dominant tests

The loading studs of the lap-shear bend test were positioned 17.5 mm either side of the fixed centre stud. The specimens were placed in the loading fixture outside the SEM chamber and aligned to ensure that the central loading stud aligned precisely with the centre of the overlap region. A small preload (5N) was applied to ensure that the sample remained in place during the transfer of the loading stage to the chamber of the SEM. The alignment of the specimens was verified immediately prior to evacuation of the SEM chamber. All tests were carried out under the same conditions, and identical test set up procedure was followed.

A wide range of the failure types listed in the ASTM D5573 standard were found to occur during failure of the lap-shear bending specimens. Stock break (Type 6) failure has been avoided through the design of the samples, but there is evidence of six other failure types occurring.

In-situ analysis

Figure 3.6 shows the progression of interfacial damage along the bondline. The view is as illustrated in Figure 3.2a, where the SEM beam looks at the edge of the joint. Frames extracted from the in-situ video are shown in Figure 3.6, accompanied by a load-displacement curve in Figure 3.6d, showing the load level at which the images were extracted. Visible crack growth at the adhesive-adherend interface was found to
Figure 3.6. In-situ analysis, Mode I dominant test, sample: B2. (a) Initial state. (b) Crack initiation, at 112 N. (c) Final failure at 209 N (d) Positions of frames on the load-displacement curve.
3.3 Results

initiate at low loads (Figure 3.6b). This occurred at both sides of the adhesive layer, and was a common characteristic of the tests where failure occurred at the bondline. The cracks appear a small distance away from the corner of the adherend, which is attributed to the SEM camera being unable to detect crack growth away from the upper surface of the joint. As loading progressed, the crack on the side of the adherend corner tended to dominate, as shown in Figure 3.6c. This is consistent with predicted stress levels which are higher in the region near this corner, as shown by Magalhaes et al. (2005). Small amounts of cohesive failure are also seen in Figure 3.6c, however, this happened just before failure of the joint, and does not appear to play a major part in the failure process of the joints tested here. Prior to final failure, large deformations were detected in the adhesive and final failure occurred suddenly. The bonds thus show an ability to maintain load bearing capacity despite cracks appearing along the edge of the adhesive layer.

Failure occurred completely within the composite adherend in one specimen, at a lower applied displacement than specimens which failed in the bondline. In this case, no sign of damage in the bondline was evident under the SEM beam until sudden final failure occurred, as the delamination failure process in the composite occurs rapidly. A possible reason for this is the propagation of cracks from a defect in the composite region, causing the delamination of the surface plies. This highlights the issue of the sensitivity of testing at this scale to small local defects.

**Failure surface fractography**

Post-failure analysis of the fracture surfaces reveals more details of the failure types which occurred within the joint.

A typical image of the failure surface of a Mode I test is shown in Figure 3.7. The adhesive layer remained on the surface of one adherend, while the highlighted fibres have remained adhered to the adhesive layer and separated from the opposing adherend. These are indicative of adhesive (Type 1) and fibre tear failure (Type 4) failure respectively.
3.3 Results

Figure 3.7: Failure surface, Mode I dominant test. A bundle of fibres, pulled from the adherend are visible, remaining on the surface of the adhesive. (Sample: B3)

After final failure, the broken halves of the joints were coated with 10 nm of gold and placed in the SEM chamber for further examination of the failure surfaces.

Imaging of the failure surface was undertaken using the SEM perpendicular to the failure surface (see inset of the following images). Figure 3.8 shows a region of adhesive failure (Type 1), where it is possible to identify the adherend surface, and adjacent raised regions of adhesive material remaining on the adherend surface. It can thus be inferred that failure migrated from one adherend to the other.

Thin layer cohesive failure (Type 3) is shown in Figure 3.9, where fibres are visible at the top of the adherend, partially covered with adhesive (Regions of adhesive are discernible through the tendency for new surfaces, revealed through large deformations in the adhesive, to charge under the SEM beam, therefore a lighter shade is produced on the adhesive in comparison to the composite resin). Only the upper surface of the fibres are visible, and there are no visible fibre tracks to indicate failure in the composite. Therefore it can be said that failure occurs within the adhesive, close to the adherend surface, as a thin layer cohesive failure.
Figure 3.8.: Type 1, Adhesive failure. Fibre tracks on the adherend and the adhesive surfaces indicate failure at the interface between the adhesive and the adherend. (Sample: B4)

Figure 3.9.: Type3, Thin layer cohesive failure (Sample: B3)
3.3 Results

Evidence of light fibre tear failure (Type 5) was found on closer examination of some adhesive failure surfaces. Such a region is shown in Figure 3.10. Fibre tracks, where fibres have been pulled away from the matrix are indicated. The smooth, dark appearance of the surface around the tracks signifies the presence of matrix material with low levels of plasticity, indicating that failure in the region close to the surface of the composite has occurred. Fibre fragments remaining on the surface of the adherend are also visible. The region of highly deformed adhesive is characteristic of Mode I failure in a tough, two part epoxy, similar to that in Figure 3.9, where it was found on the surface of the adherend.

The majority of the failed specimens were observed to have mixed failure (Type 7) surfaces. Numerous examples of this mode of failure exist in the literature (Bonhomme et al., 2009, Greenhalgh, 2009). Figure 3.11 shows different failure types occurring in adjacent regions. A region of thin layer cohesive failure can be seen to surround an island of light fibre tear failure. The dominant feature in the figure is the large group of fibres that adhered to the surface of the adhesive, in a fibre tear failure. Examination of the fibre tear regions showed failure characteristics which signified Mode I failure.
3.3 Results

**Figure 3.11.:** Type 7, Mixed failure. Regions of fibre tear failure, thin layer cohesive failure and light fibre tear failure are visible, immediately adjacent to each other. (Sample:B3)

of a composite material. A close up of the failure pattern produced when the group of fibres in Figure 3.11, were pulled from the adherend is shown in Figure 3.12. Fibre surfaces are revealed and the tracks of the pulled out fibres are also visible. The clean appearance of these features, particularly the fibre tracks in this image show that separation of the fibres and matrix takes place without large deformations around the circumference of the fibre. Large deformation does take place in the matrix, in the regions between the debonded fibres, marked in the image as scarps. In a pure Mode I failure, a flat surface would be expected, as illustrated in Figure 3.13a. It is clear that the fracture surfaces do not adhere to this shape. However, following the interpretation of such features in Greenhalgh (2009), it is possible that the features highlighted as scarps are Mode I dominated scarps and mixed mode scarps. Mode I dominated scarps can be seen to arrange themselves in an ordered pattern. Scarps are formed when cracks form on slightly different planes, and join up, creating shallow steps between crack planes, as illustrated in Figure 3.13b. These cracks initiate at the edge of the fibres and grow into the resin rich regions simultaneously, leading to the inverted
3.3 Results

Figure 3.12.: Mode I dominant composite failure

Figure 3.13.: Crack growth through the epoxy adhesive and matrix materials under: (a) Mode I conditions (b) Mixed mode and (c) Mode II conditions. In all cases, the dashed lines represent predicted crack path, adapted from Greenhalgh (2009)

“V” shape in this image. Cracks formed in epoxy materials under different mode mixity conditions, is addressed in Greenhalgh (2009). As the Mode II component of the crack driving force increases, the scarps become more upright (see Figure 3.13c), and the less organised features highlighted as the mixed mode scarps are formed. Once Mode II conditions dominate, the features are called cusps, which are shown in greater detail in the Mode II tests, presented in section 3.3.3.

The close proximity of these different failure types suggests that fluctuations in bond strength can change the type of failure occurring. It can be assumed that a stronger bond will result in the fibres being pulled from the adherend and that a weaker in-
3.3 Results

interface will produce interface failure. It is possible for local heterogeneities to dictate whether thin layer cohesive or light fibre tear failure occurs in the case of a strong interface. In Khalili and Shokuhfar (2008), failure of large scale composite joints are also categorised according to ASTM D5573, and it can be seen that the range of failures produced using the miniature joints in this work is similar to the range produced by the large scale joints.

3.3.2. Weak interface lap-shear bend test

The range of failure loads for the joints is expanded to provide a larger dataset against which to compare the results of the microscale modelling in the following chapters. A second set of joints, geometrically identical to those presented above were prepared. The surface preparation in the preceding section was deemed to be very good, as failure often occurred away from the interface, in the adhesive and composite regions. It was therefore decided to compare the failure behaviour of a weak adhesive-composite interface to the high strength bonds. The interface was weakened through poor surface preparation in comparison to the previous tests. Dust was removed from the surface of the adherends using a lint free cloth. However, no mechanical abrasion or degreasing agent was used to prepare the surface. This falls well below the standard surface preparation for this type of bond (Kinloch, 1987) and is therefore expected to result in much lower joint strength. Two samples, designated W1 and W2, were tested under lap-shear bend conditions.

In-situ results

Lap-shear bend tests were carried out on the weak joints, and the results are shown in Figure 3.14, as the “weak bondline” data. Accompanying these results are the data from the preceding section, which are referred to as “strong bondline” data. Good correlation of the initial stiffness was achieved between the two sets of results, however, the tests with the weakened bondline failed, as expected, at a much lower load than
Figure 3.14.: Experimental load-displacement results

those with good surface preparation. The average failure load for the strong joints was 225 N, while the average failure load for the weaker joints was 90 N. The improved surface preparation therefore produced a 250% increase in joint strength.

Analysis of the in-situ SEM video revealed a brittle failure, with no crack growth detected prior to catastrophic failure of the joint and very little deformation of the adhesive layer observed. This is in contrast to the progressive crack growth, visible in Figure 3.6 on the joints with the strong interface. Figure 3.15 shows frames from one of the tests immediately prior to final failure, compared to the initial state of the bond, and it is clear that no significant damage has initiated between the two frames, and that no yielding or damage within the adhesive layer took place.

**Failure surface fractography**

Figure 3.16a shows a failure surface of the weak joints. It can also be seen that the overall appearance of the surface is very different from the previous tests, as large riverlines radiating from a shiny region dominate the failure surface in this case. This replaces the range of failure patterns, resulting from the tests on the stronger joints.

Three distinct regions are visible, a shiny region of the fracture surface is marked
Figure 3.15.: In-situ analysis of joint failure with a weak composite-adhesive interface. (a) The initial state of the joint. (b) Just prior to catastrophic final failure, no damage is visible. (c) Positions of the Figures (a) and (b) on a load-displacement curve. (Sample W2)
3.3 Results

Figure 3.16: (a) Failure surface of a joint with a weak interface, showing mirror, mist and hackle crack growth (b) Defect on the surface of the adherend. (c) Transition from tide mark pattern to a riverline pattern.
as the mirror region in Figure 3.16a. Defects on the adherend surface in this region
caused extremely poor adhesion between the adhesive and the adherend. Figure 3.16b
demonstrates a defect protruding from the adherend surface, found within the shiny
region (the poor definition of the image at the defect is a result of sharp edges on the
defect charging under the SEM beam; images at lower accelerating voltages did not
provide any significant improvement). Tide marks can be seen on the right hand side
of the defect in Figure 3.16b. The paler rings, caused by stable crack growth, leading
to yielding and high deformation in the material, and a rough topology is seen in this
region. Dark rings are regions of rapid crack growth with very little plastic deformation
of the material. The tide marks lie perpendicular to the direction of crack growth. Such
marks indicate a change in speed of a crack front (Greenhalgh, 2009), in a stick-slip
crack growth pattern, which in this case, appear to have initiated at the defect in the
Figure 3.16b.

A close up of the mist region, in which the riverlines in 3.16a initiated, is given in Fig-
ure 3.16c. The crack front is seen in transition from stick-slip crack growth to propaga-
tion in the form of riverlines. Extremely small Mode I cracks can be seen to initiate in
the lighter rings of the tide marks, which grow larger and amalgamate to form large
riverlines. Tide marks and riverlines, both forms of Mode I crack propagation, domi-
nate the failure surface and indicate an accelerating brittle crack front.

The mirror, mist and hackle regions, are analysed in the case of composite materials
in Greenhalgh (2009). These features are seen as indicators of an accelerating crack,
increasing in speed from the mirror region, where crack initiation occurs, through the
mist region to extremely fast propagation in the hackle region.

3.3.3. Mode II tests

All compression tests were carried out under identical conditions. The samples were
placed in the compression grips outside the SEM chamber for ease of access and to
prevent damage to the SEM. The samples were loaded into the compression grips, and
3.3 Results

secured to the test platform before transfer to the SEM. Failure under Mode II conditions produced almost identical failure patterns across all samples tested. All samples failed at the adhesive-adherend interface (Type 1), after significant deformation of the adhesive layer. Significant scatter in the results of the Mode II tests preclude their inclusion here. Confidence with regard to the presence of Mode II conditions in the adhesive layer is gained from analysis of the in-situ video and post failure analysis of the fracture surfaces, where Mode II failure is clearly induced in the tests conducted here.

In-situ analysis

Analysis of the in-situ video showed that deformation under Mode II loading was largely confined to the adhesive layer. Frames from the in-situ video are presented in Figure 3.17, along with the load deflection curve for the sample. The initial test state is presented in Figure 3.17a. It can be seen in Figure 3.17b and 3.17c that only limited crack growth at the interface occurred prior to final failure. Large deformations in the adhesive layer were immediately followed by catastrophic failure.

Failure surface fractography

All samples in this load configuration failed in the same manner, and failure surfaces were almost identical under visual inspection. Failure occurred through adhesive failure (Type 1) only, the range of failure modes visible in the Mode I dominant tests was not reproduced here. Three distinct regions were visible on all failure surfaces, the clearest example of which is shown in Figure 3.18. At the end of the adherend, no adhesive remained, meaning the adherend surface was visible. The middle third of the lapped area was a transition region, where isolated sections of adhesive remained adhered to the surface of the adherend. The final third, furthest from the end of the adherend was covered in adhesive. This regular pattern indicates that cracks initiated at the end of the adherend, at the interface between the adherend and the adhesive. Symmetry of the geometry produced cracks initiating from both sides simultaneously. As the cracks progressed towards the centre of the lap, regions of differing local bond
3.3 Results

Figure 3.17: In-situ analysis, Mode II test, sample C2. (a) Initial state. (b) Adhesive layer at F=411N. (c) Final failure at F=711N. (d) Positions of frames on a load-displacement curve.

Figure 3.18: Failure surface, Mode II test. The adherend surface is visible, separated from the region where the adhesive remained on the adherend by the transition region. (Sample: C6)
strength and local inhomogeneities create the transition region.

The most prevalent failure characteristic in the Mode II tests was the cusp (see Figure 3.19), a well known shear failure formation. When the shear stress is resolved into principal stresses, a tensile traction, at 45° to the final failure surface, is formed. This tensile component opens microcracks inclined to the failure surface, which form cusps, as illustrated in Figure 3.13c (Bonhomme et al., 2009, Greenhalgh, 2009). Cusps were found to occur on the adhesive and adherend sides of the failure surfaces. The low resin toughness in the composite, combined with the presence of fibres, ensures the cusps on the adherend surface (Figure 3.19a), were small. Cusps on the adhesive surface were large, as shown in Figure 3.19b, signifying a tough and ductile adhesive (Greenhalgh, 2009).

A detailed view of a feature in the transition region is presented in Figure 3.19c. Failure at adhesive and adherend surfaces are visible. The walls of the feature show brittle Mode I failure characteristics, signified by textured microflow (Greenhalgh, 2009). This implies that damage progressed to the centre of the lapped area through Mode II failure at opposing adherend interfaces. At the transition region, the cracks propagated through the adhesive layer as Mode I cracks, linking the two damaged interfaces.

### 3.4. Conclusions

Failure mechanisms in adhesively bonded composite joints have been examined using novel in-situ SEM and post failure analysis on samples under Mode I dominant and Mode II conditions. A finite element analysis showed that stress states leading to Mode I dominant and Mode II failure were produced in the adhesive layer for each case.

In-situ SEM provided a valuable insight into the failure processes during loading and failure. Under Mode I conditions, it was possible to observe progressive crack growth at both adherend-adhesive interfaces. As the cracks extended along the interface, some damage within the adhesive layer was detected. Under Mode II conditions, the in-situ results showed little or no damage prior to final failure. Clearly visible, however, was
Figure 3.19.: (a) Cusps found on the adherend surface after Mode II failure, sample C4. (b) Cusps seen on the adhesive surface after Mode II failure, sample C7. (c) Feature from the transition region of a Mode II test. Surfaces of the adhesive and the composite adherend are visible, the regions are connected by the indicated Mode I cracks, sample C3
3.4 Conclusions

the large deformation confined to the adhesive layer.

The fracture surfaces of the failed joints were analysed. The Mode I tests produced a wide range of failure types. Of the seven failure types referenced in the ASTM standard for analysing adhesive failure, six have been shown in these tests. It was found that very different failure modes occurred in close proximity to each other. From this evidence it is suggested that local fluctuations in bond strength changed the failure type.

Additional tests were carried out under lap shear bending, to investigate the strength and failure behaviour of joints with poor quality adhesion between the adhesive and composite region. The strength of the joints was reduced through the use of an extremely poor surface preparation technique relative to the original joints. Comparison of the load-displacement results for the strong and weak bondlines showed that the strength of the weak joints was reduced by a factor of 2.5 in comparison to the original joints. Analysis of the in-situ video, captured during the testing of the weakened joints, showed very little deformation of the adhesive. Additionally, no crack growth at the adhesive-composite interface was detected prior to final failure. Fractographic investigation of the failure surface showed a mirror, mist and hackle pattern, as cracks accelerated across the lapped area of the joint.

It was found that all joints tested under Mode II conditions failed at the adhesive-adherend interface. Mode II failure characteristics, were found on all failure surfaces. The wide range of failure types found in the Mode I tests was not obtained in the Mode II tests.

This chapter provides an insight into the failure mechanisms, occurring at the microscale, between a composite adherend and an epoxy adhesive. The results show similar post-failure appearance to large scale adhesive joints, while allowing a novel view of the damage processes at high magnification. The following chapters build a micromechanical model to investigate these damage processes.

The results of this chapter show that failure in low strength joints produced damage which was limited to the interface between the adhesive and the composite. Failure of high strength joints resulted in a much more complex damage process. Through
3.4 Conclusions

proper preparation of the sample surfaces, the top ply of the composite and the adhesive layer became involved in the damage progression. This observation has implications for the following microscale modelling chapters, as it will be necessary to show failure mechanisms corresponding to the experimental results here. The models will be required to show different failure mechanisms in strong and weak bonds. A range of failure mechanisms will be required from the micromodels under Mode I conditions, while Mode II failure has been shown here to be limited to the composite-adhesive interface, and the micromodels need to reflect such behaviour.
4. Numerical Determination of Fibre-Matrix Interfacial Strength Parameters

4.1. Introduction

In this chapter, a previously developed method for creating microstructural models of the composite material HTA/6376 is extended, allowing the microscale damage response of the material in three dimensions to be simulated. This is an important step in creating the option for application of the approach to more complicated cases, i.e. inter-laminar failure and adhesive joints.

Fibre-matrix debonding has been identified as a critical controlling factor for composite failure (Ghosh et al., 2000). Therefore, the focus of most recent micromechanical investigations have centred around the characterisation and identification of fibre-matrix interface parameters. Interface fracture energies were calculated by Varna et al. (1997) for increasing levels of debonding around a single fibre in a region of matrix. The spatial distribution of the fibres within the matrix was also recognised as an important contributor to initiation of debonding in Hojo et al. (2009). Therefore, models have been developed to recreate the distribution of fibres within the matrix accurately.

Note: the results of this chapter have been published as two peer reviewed publications under the titles *Investigation of strain hardening effects under in-plane shear of unidirectional composite materials* (O’Dwyer et al., 2012), in a special issue of Computational Materials Science, and in the Journal of Composite Materials *Investigation of Interfacial Strength Parameters in a Carbon Fibre Composite Material* (O’Dwyer et al., 2013b).
4.1 Introduction

These models have been used to form representative volume elements (RVEs) which describe a microscopic volume of the composite, from which the material behaviour is extracted through a homogenisation procedure. Using such models, the fibre-matrix interface strength has been the focus of investigations by many authors (Totry et al., 2010, Hojo et al., 2010, Vaughan and McCarthy, 2011a,b). Interfacial debonding has been investigated under transverse tension, compression and shear using 2-D models (Vaughan and McCarthy, 2011a,b, González and Llorca, 2007) and also under in-plane shear using 3-D models (Totry et al., 2010, 2008b). Recently, (Okabe et al., 2011) provided a good insight into effects of fibre distribution and an accurate simulation of the damage detected experimentally in (Hobbiebrunken et al., 2006) was produced.

Among the published experimental data was an effect, highlighted by Totry et al. (2010), where the composite appeared to exhibit strain hardening at high in-plane shear strains. Fibre rotation was given as the reason for the observed strain hardening, and this effect is quantified here. Previous studies (Totry et al., 2010, Vaughan and McCarthy, 2011a,b, Romanowicz, 2009, Zhang et al., 2010) assumed interface properties with the shear strength of the interface equal to the normal strength. This assumption has good correlation to experimental data under transverse shear, transverse tension and compression. However, investigation under in-plane shear by Totry et al. (2010) indicates a high interfacial shear strength. In this work, it is shown that in the transverse plane, the interfacial shear strength has a much smaller effect than the interfacial normal strength. It is then shown that assuming the interfacial shear strengths to be equal and of higher magnitude than the normal strength at the interface provided parameters which could provide accurate response under transverse tension and shear and in-plane shear.
4.2 Modelling Strategy

4.2.1. Model Development

The material under investigation, HTA/6376, is a high strength, carbon fibre/epoxy material. The microstructure of HTA/6376 has been characterised previously by the nearest neighbour algorithm (NNA) of Vaughan and McCarthy (2010) where the fibre volume fraction was calculated as \( \sim 59.2\% \) and the average fibre diameter is 6.6 \( \mu \text{m} \). The statistically equivalent representation of the fibre distribution, as produced using the NNA, has been shown capable of producing accurate results under transverse tension and shear in Vaughan and McCarthy (2011a,b) respectively. The RVE size used in the present study was 40 \( \times 40 \times 0.4 \mu \text{m} \) (See Figure 4.1) and the fibre volume fraction was 62.4\%. The size of the RVE was comparable to those used in similar three-dimensional studies, where an increase in RVE size was found to have a negligible effect on the results (Totry et al., 2010, 2008b). The model is meshed using predominantly eight node brick (C3D8) elements with six node wedge (C3D6) filler elements. Interface regions, around each fibre, are meshed with COH3D8 cohesive elements. The models contained approximately 100,000 elements with 5-10\% cohesive elements.

Analysis of the in-plane strain hardening was undertaken using four conditions, as follows:

![Dimensions of microstructure (Note: Not to scale)](image_url)
4.2 Modelling Strategy

- Elastic Small Deformation Theory (SDT) model: Elastic material properties used, linear geometry.

- Elastic Large Deformation Theory (LDT) model: Elastic properties used, non-linear geometry.

- Plastic SDT model: Elastic-plastic matrix properties, elastic fibre properties, linear geometry.

- Plastic LDT model: Elastic-plastic matrix properties, elastic fibre properties, non-linear geometry.

Under in-plane shear, the SDT analysis did not take the rotations of the fibres into account, but the LDT analysis allows the effects of fibre rotation to be calculated. The four different models, allowed individual stress contributions to the strain hardening effect to be analysed separately.

In all cases, the effects of residual stresses have been omitted, in favour of identifying damage behaviour due to mechanical loading. Investigation of thermal residual stress has been carried out previously using similar models to those used here (Vaughan and McCarthy, 2011a, b).

4.2.2. Boundary Conditions

Periodic boundary conditions have been applied, in the three primary directions, using constraint equations. The equations were applied to all external face, edge and corner nodes to ensure no displacement or stress discontinuities occurred over the edges of the RVE. The corner nodes were designated as the “control” nodes, through which the displacements were applied. The displacements of the remaining external nodes were constrained relative to the control nodes. Tensile loads were applied at 90° to the top surface of the RVE, and a periodic Poisson's contraction occurred transversely to the applied displacement. Under shear conditions, pure shear was enforced throughout the loading, calculated using the tension-compression pure shear defor-
4.2 Modelling Strategy

Figure 4.2.: Pure shear applied to control nodes of an RVE in the transverse plane.

...mation matrix in Beatty (2000), applied to the control nodes. It has been ensured that the pure shear deformation is maintained after failure of the RVE.

Under shear conditions, deformations were controlled by the pure shear deformation matrix, $D_{PS}$ (Beatty, 2000), given in Equation 4.1.

$$D_{PS} = \begin{bmatrix} \lambda^{-1} & 0 \\ 0 & \lambda \end{bmatrix}$$  \hspace{1cm} (4.1)

where $\lambda = 1 - \gamma$, and $\gamma$ is the shear strain. This deformation matrix produces a tension compression case, however, the displacements of the control nodes, marked N1 - N4 in Figure 4.2, have been calculated to produce a more conventional configuration, as shown.

The deformed coordinates, $x'$ and $y'$, of each of the control nodes are calculated as:

$$\begin{bmatrix} x' \\ y' \end{bmatrix} = D_{PS} \begin{bmatrix} x \\ y \end{bmatrix}$$  \hspace{1cm} (4.2)

where $x$ and $y$ are the original, undeformed coordinates of the control nodes.

4.2.3. Material Properties

HTA Fibres

Hojo et al. (2009) published orthotropic linear elastic properties for the HTA fibres, which are presented in Table 4.1. It may be noted that the fibres are significantly stiffer in the longitudinal direction. The fibres were assumed to be linear elastic and damage in the fibre has not been considered for this study.
4.2 Modelling Strategy

Table 4.1: Elastic properties of constituent phases

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<th>6376 Matrix</th>
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<tr>
<td>$G_{31}$ (GPa)</td>
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</table>

6376 Matrix

It is known that the yield behaviour of polymer materials is sensitive to hydrostatic stress (Quinson et al., 1997, Haward and Young, 1997). Thus, the von Mises or Tresca yield criteria are unsuitable to represent the behaviour of the highly constrained matrix between the fibres in micromodels of composite materials. This has been addressed here through the use of the Mohr-Coulomb yield criterion, which accounts for the dependence of the yield point on hydrostatic stress. This model has been shown to be capable of matching experimental stress-strain data, and also shear banding effects (Vaughan and McCarthy, 2011a, b, González and Llorca, 2007, Totry et al., 2008a, b).

The Mohr-Coulomb criterion is defined by

$$\tau = \tau_m^c - \sigma_n \tan \phi$$  \hspace{1cm} (4.3)

The criterion predicts yield in the material when the shear stress, $\tau$, exceeds the yield stress under shear of the material, $\tau_m^c$. The effect of hydrostatic stress is taken into account through multiplication of the normal stress, $\sigma_n$, at a point, by $\tan \phi$, which controls the sensitivity of the material to hydrostatic stress, where $\phi$ is the friction angle of the material. A value of $\phi = 0^\circ$ reduces the criterion to the Tresca maximum shear stress criterion, while a value of $\phi = 90^\circ$ returns the Rankine maximum stress criterion.
### 4.2 Modelling Strategy

#### Table 4.2: Interfacial stiffnesses

<table>
<thead>
<tr>
<th>$k_n$ (GPa)</th>
<th>$k_{s_1}$ (GPa)</th>
<th>$k_{s_2}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3640</td>
<td>1400</td>
<td>1400</td>
</tr>
</tbody>
</table>

The failure surface can be represented in terms of the principal stresses as:

$$f(\sigma_1, \sigma_3) = (\sigma_1 - \sigma_3) + (\sigma_1 + \sigma_3)\sin\phi - 2\tau_m^c \cos\phi = 0 \quad (4.4)$$

The compressive and tensile strengths of the matrix material, $\sigma_c$ and $\sigma_t$, respectively may then be written in terms of $\tau_m^c$ and $\phi$ as:

$$\sigma_t = 2\tau_m^c \frac{\cos\phi}{1 + \sin\phi} \quad \text{and} \quad \sigma_c = 2\tau_m^c \frac{\cos\phi}{1 - \sin\phi} \quad (4.5)$$

This approach to determine the Mohr-Coulomb parameters was first used by González and Llorca (2007), and subsequently used to characterise the 6376 matrix in Vaughan and McCarthy (2011a, b). Experimental data, published by Fiedler et al. (2005), provided values for $\sigma_t$ and $\sigma_c$ as 103 MPa and 264 MPa respectively, which when used in Equation 4.5, returned values of $\tau_m^c = 82$ MPa and $\phi = 26^\circ$ respectively. The isotropic elastic properties of the matrix are presented in Table 4.1.

#### Interface Region

The interface has been modelled as a cohesive region, with the initial stiffness of the interface chosen to maintain stress continuity with the surrounding matrix and a negligible displacement discontinuity between fibre and matrix, prior to the onset of damage. The cohesive elements thickness was $1 \times 10^{-4}$ μm and the stiffness values are given in Table 4.2.

A traction-separation law is used to characterise the interface region through the use of cohesive elements. Local orientations are assigned to the cohesive elements, as shown in Figure 4.3a. The normal direction, $n$, extends radially from the centre of each fibre; the $s_1$ shear direction is assigned to shear in the hoop direction and the $s_2$ shear direction is assigned parallel to the fibre longitudinal direction. The initiation of
4.2 Modelling Strategy

Table 4.3.: Interface Energies (Varna et al., 1997)

<table>
<thead>
<tr>
<th>$G^C_{n1}$ (J/m$^2$)</th>
<th>$G^C_{s1}$ (J/m$^2$)</th>
<th>$G^C_{s2}$ (J/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>25</td>
<td>25</td>
</tr>
</tbody>
</table>

damage is controlled by the quadratic nominal stress criterion (ABAQUS-Inc., 2010), given as:

$$\left\{ \frac{< t_n >}{t_n^c} \right\}^2 + \left\{ \frac{t_{s1}}{t_{s1}^c} \right\}^2 + \left\{ \frac{t_{s2}}{t_{s2}^c} \right\}^2 = 1$$  \hspace{1cm} (4.6)

Where $t$ is the traction between the top and bottom faces of the element, and the superscript $c$ refers to the critical traction at the point of damage initiation. The Macaulay brackets, $<$, ensure that negative normal (compressive) traction does not damage the interface. This criterion accounts for the interaction between tensile and shear failure modes with differing initiation tractions. Damage evolution is controlled by a linear, energy based, damage evolution law. Interface fracture energies were calculated by Varna et al. (1997), for strong, tough interfaces in a glass fibre reinforced material for Mode I ($t_n$) and Mode II ($t_{s1}$) tractions and are presented in Table 4.3. Interfacial energies have been investigated numerically in Vaughan and McCarthy (2011a, b) for the composite under consideration herein, and the values used here are intermediate values from that study. The shear strengths and shear fracture energies of the interface are taken to be equal. Mode mixity was taken into account based on the Benzeggagh-Kenane (BK) criterion (Benzeggagh and Kenane, 1996):

$$G_c = G_n^C + \left( G_s^C - G_n^C \right) \left\{ \frac{G_S}{G_n + G_S} \right\}^\eta$$  \hspace{1cm} (4.7)

Where $G_c$ is the mixed mode fracture energy, $G_{s1} + G_{s2} = G_S$. The superscript $C$ refers to the critical fracture energies, given in Table 4.3. The interface parameters are illustrated on a traction separation diagram in Figure 4.3b. The unloading portion of the traction separation curve is controlled by the fracture energy. Since the area under the traction-separation curve is equal to the fracture energy, by assigning the fracture en-
4.3 Results

Figure 4.3: Cohesive Element details (a) Interface Local Orientations (b) Traction separation law

Energies, initial stiffnesses and critical tractions, the critical distances, $\delta_c^n$ and $\delta_c^s$, beyond which there is no traction across the interface, can be calculated. For any mode mix condition, Equation 4.6 and 4.7 provide the critical traction and fracture energy, from which the corresponding critical distance can be obtained. A value of $\eta = 1$ is used in the present work.

A viscous regularisation value of $2 \times 10^{-4}$ was necessary to promote convergence of the FE solution and was found to have a negligible effect on the accuracy of the results.

4.3. Results

The stress state considering only elastic deformations, prior to damage initiation, within the interface region around a fibre in a multi-fibre array is first compared to that around a single isolated fibre in a region of matrix, in order to demonstrate the influence of neighbouring fibres on the interfacial stress state. The RVE is subsequently subjected to three loading conditions, as shown in Figure 4.4. A parameter study of interface strengths under transverse tension and shear has been undertaken.
4.3 Results

![Figure 4.4: Loading conditions](image)

Figure 4.4: Loading conditions (a) Transverse tension (b) In-plane shear (c) Transverse shear

4.3.1. Interfacial Stress State

Radial plots of the stress distribution within the interface, following the approach in Hojo et al. (2009), are shown in Figure 4.5 and Figure 4.6. The plots have been arranged such that the highlighted fibre in Figure 4.6(a) has been isolated and compared to the single fibre case in Figure 4.5(a). The normal ($t_n$) and shear ($t_{s1}$) tractions in the interface of the fibre have been plotted radially. The edge of the fibre represents the zero axis for $t_n$ and $t_{s1}$. The absolute value of $t_{s1}$ has been plotted for clarity, as positive and negative shear contribute equally to the damage initiation criteria in Equation 4.6. Negative values of $t_n$, although shown, are not considered in the initiation criteria. It should be noted that under loading in the transverse plane, $t_{s2}$ was orders of magnitude lower than $t_n$ and $t_{s1}$, and is therefore not considered in these plots.

Single fibre RVE

A number of important interfacial stress distribution characteristics are visible in Figure 4.5 for the single fibre case. The most important, with relation to this study, is the relative magnitude of $t_n$ and $t_{s1}$, shown in Figure 4.5b and Figure 4.5c. Under transverse tension, $t_n$ is twice the magnitude of $t_{s1}$ and no compressive interfacial
4.3 Results

**Figure 4.5.** (a) Single Fibre RVE (b) the normal and shear interfacial tractions under transverse tension (c) normal and shear interfacial tractions under transverse shear

**Table 4.4.:** Interfacial strength values

<table>
<thead>
<tr>
<th>$t_n^c$ (MPa)</th>
<th>$t_{s1}^c$ (MPa)</th>
<th>$t_{s2}^c$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>85</td>
<td>125</td>
<td>125</td>
</tr>
</tbody>
</table>

stress was observed. An analytical solution for this case has been presented previously by Lesne et al. (1995), which produces distributions of $t_n$ and $t_{s1}$ which are almost indistinguishable from those shown here. Under transverse shear, the magnitude of $t_n$ and $t_{s1}$ are equal, with a large compressive $t_n$ stress.

**Multi-fibre RVE**

Figure 4.6 b and c show distributions of $t_n$ and $t_{s1}$ for the fibre in Figure 4.6a marked “A”, in a multi-fibre RVE, generated using the NNA of Vaughan and McCarthy (2010). Under transverse tension and shear, the effect of the neighbouring fibres on the distribution of $t_n$ around the fibre is illustrated by the irregular shape of $t_n$. Local maxima of $t_n$ are found to be located along lines connecting the centres of the nearest adjacent fibres and an amplification of $t_n$ relative to $t_{s1}$ was found in comparison to the single fibre case. It can be seen that the shape of the distribution of $t_{s1}$ is not as strongly influenced by the presence of the neighbouring fibres. It can be seen also that $t_n$ is now larger than $t_{s1}$ in both transverse tension and shear, where the magnitude of both were equal in the single fibre case under transverse shear.
4.3 Results

Figure 4.6: (a) Fibre in a multi-fibre array, marked “A” about which normal and shear interfacial tractions are shown in (b) under transverse tension and (c) under transverse shear

4.3.2. Overall response in transverse tension

The loading conditions for the transverse tensile load case are shown in Figure 4.4a. The highest stresses were found to occur between fibres aligned along the loading direction, as shown in Hojo et al. (2009). The elastic modulus under transverse tension ($E_{22}$) for the composite, was calculated as 10.8GPa, showing good correlation to the value experimentally determined by O’Higgins et al. (2011) of 10.1GPa.

The damage process was found to be consistent across the range of interface strengths studied. Plasticity initiated before the onset of debonding, in areas of high stress between closely packed fibres. Once the initiation criterion for damage in the cohesive elements was reached, the matrix and fibre separated from each other progressively and continuously along the fibre circumference. Debonding of the fibres was quite advanced when regions of matrix plasticity were seen to advance until a point was reached where adjacent plastic regions coalesced, linking each damaged interface to its closest damaged neighbour. The regions of matrix plasticity, combined with the debonded regions around the fibres, formed a “crack path” across the RVE, perpendicular to the loading axis, which is visible in Figure 4.7, where equivalent plastic strain (PEEQ) is plotted at the last increment of the solution. It should be noted that no damage mechanism was modelled in the matrix material.
4.3 Results

Figure 4.7.: Crack path under transverse tension indicated by high levels of equivalent plastic strain (PEEQ)

Influence of interface strength, transverse tension

The normal and shear interface strengths, $t_n^c$ and $t_{s1}^c$, have been varied, relative to the yield shear stress of the matrix, $\tau_m^c$. Either $t_n^c$ or $t_{s1}^c$ was held constant at $\tau_m^c$, and the other strength term was varied to higher and lower levels. The critical interface toughnesses, $G_n^C$ and $G_s^C$, were held constant. The overall stress-strain graphs obtained are presented in Figure 4.8. The results here are normalised with relation to $\tau_m^c$. Increases in $t_n^c$ were seen to correspond to an increase in the tensile strength of the composite in 4.8a, due to the ability of the interfaces to transfer load between the matrix and fibres to a higher applied global strain. This behaviour was shown experimentally through variation of fibre surface treatment in Hoecker et al. (1995) and experimentally and numerically in (de Kok and Peijs, 1999). This behaviour is not reproduced under variation of $t_{s1}^c$, where no significant increase in the strength of the composite was detected once the shear strength of the interface exceeded the normal strength, as seen in 4.8b. This is due to the high value of $t_n$, which dominates the damage process, unless the
interface shear strength is very low (see also 4.6b).

Table 4.4 contains the interface strengths, selected after consideration of the parameter studies in the previous and following sections, which provide the best match to the experimental data of O’Higgins et al. (2011). The stress-strain values in Figure 4.9 show good correlation to the strength data from O’Higgins et al. (2011). The ductile behaviour in comparison to the experimental data is necessary to provide a good match to experimental results under shear conditions, where matrix yielding is a critical factor.
4.3 Results

Figure 4.9.: Stress-strain results under transverse tension

4.3.3. In-plane Shear

The loading conditions for in-plane shear are illustrated in Figure 4.4b. The overall elastic shear modulus, $G_{23}$, was calculated as 5.6 GPa, compared to the value of 5.2 GPa, as determined through in-plane shear tests (O’Higgins et al., 2011).

**Strain Hardening Effects Under In-Plane Shear**

Although fibre-matrix debonding is a critical factor in the damage process at the microscale, the results of Totry et al. (2010) under in-plane shear indicate that interfacial debonding does not occur under in-plane shear until a high global shear strain was reached. The range of shear strains examined in this section has therefore been kept below the reported final, catastrophic failure strains, which allowed fibre debonding to be omitted.

Totry et al. (2010) showed, experimentally and numerically, a strain hardening effect occurring as the in-plane shear strain on the composite was increased. This has been attributed to fibre rotation, where the stiffer longitudinal axis of the fibre became progressively more aligned with the loading axis. Figure 4.10 illustrates the rotation of the fibres in pure shear. Initially, the fibres are aligned with the 1-axis, and lie at an angle of
4.3 Results

Figure 4.10: Pure shear state with relation to the fibre direction (not to scale)

\( \alpha = 45^\circ \) to the resultant of the combined shear load, \( F_{12} \), as shown by the dashed lines in Figure 4.10. This orientation is altered through the application of the shear strain, so that the angle between the fibre's longitudinal axis and \( F_{12} \) is reduced to \( \alpha' \) (this effect is exaggerated in the figure). Therefore the fibre's stiffer longitudinal direction is more closely aligned with the applied load.

To investigate the issue of fibre rotation, the four models, as described in Section 4.2.1, were used. The homogenised elastic shear modulus was the same for all four models, calculated as \( G_{12} = 5.09 \) G Pa, which is in close agreement to the published value of \( G_{12} = 5.2 \) GPa for HTA/6376 (Ireman, 1998, O'Higgins et al., 2011). The elastic model produced almost identical stress results from both the SDT and LDT analyses. Stress-strain results from the elastic-plastic model are shown in Figure 4.11. It can be seen that a strain hardening effect is obtained at higher strain levels from the LDT analysis, which is not obtained in the SDT results.

An investigation into the stress state in the fibres was undertaken, to isolate individual contributions to the hardening effect. An element was chosen from the centre of one of the fibres, where the edge effects, produced through interaction with the surrounding matrix, were negligible. The stress in the longitudinal direction of the fibre was non-existent, and showed no increase in the SDT analysis. However, from the LDT
Figure 4.11.: Stress strain results under in-plane shear, the hardening effect is visible at shear strains higher than approximately 4%.

analysis, the longitudinal stress in the fibre was seen to increase significantly, as shown in Figure 4.12, as the fibre aligned itself with the loading axis. The rate of increase of longitudinal stress within the element was greater in the elastic-plastic matrix case, as shown.

The shear stress in the same element was similarly investigated. The results from the elastic-plastic model are presented in Figure 4.13. The shear stress in the plastic model reflects the yielding behaviour of the surrounding matrix, however, a hardening effect is found here at high strains.

The fibre stress state remains almost homogeneous at the fibre centres and due to the linear elastic properties of the fibres, an increase in strain produces a corresponding increase in stress. This is not the case for the matrix material, as the perfectly plastic material properties of the matrix prevents hardening, as seen in the fibres, as an increase in the applied global strain produces an increase of plastic strain in the matrix, which is concentrated within a shear band.

The rotation of the fibres is illustrated in Figure 4.14, where the longitudinal axis of the fibre in the original orientation is compared with the final orientation of the fibre.
4.3 Results

**Figure 4.12.** Longitudinal stress in the fibre

**Figure 4.13.** Shear stress in the fibre
4.3 Results

The fibre rotation angle, $\theta$ was measured during the elastic and elastic-plastic simulations, and the results are presented in Figure 4.15. It can be seen that the rotation of the fibres was equal in both cases until $\gamma_{12} = 0.015$, at which point the rotation of the fibres in the plastic model begins to increase relative to the elastic model. At a strain of $\gamma_{12} = 0.1$, the rotation of the fibres in the plastic model was almost twice that of the fibres in the elastic model.

**General response under in-plane shear**

Analysis of the strain distribution of the RVE in Figure 4.16 shows that under in-plane shear, an intense band of plasticity forms across the RVE. Two separate sections of the RVE were created, one above, and one below, the plastic shear band, and no debonding was observed.

The corresponding stress-strain plot is presented in Figure 4.17, using the same material parameters as for the transverse tension case (see Table 4.4). If low values of interfacial shear strength are chosen, interfacial shear debonding is observed, and the predictions deviate from the experimental observations in (Totry et al., 2010). This implies that the interfacial shear strength value used represents a lower bound, as higher strengths could be seen to have negligible effects on the solution, while lower strengths
4.3 Results

**Figure 4.15.** Fibre Rotation, Elastic and Plastic models

**Figure 4.16.** In-plane Shear (Note: the outline of the deformed RVE has been extended in the fibre direction for clarity)
showed deviation from the experimental results.

### 4.3.4. Transverse Shear

The transverse shear loading conditions are illustrated in Figure 4.4c. The elastic model returned a transverse shear modulus $G_{23} = 3.8$ GPa, which provides a good match to the published value of $G_{23} = 3.9$ GPa (Ireman, 1998). Initiation of plastic yielding occurred between the fibres packed closest together prior to widespread debonding occurring across the RVE, which initiated under interfacial normal failure. Figure 4.18 shows equivalent plastic strain (PEEQ) after final failure, where a clear band of shear induced plasticity has formed vertically along the left hand side of the RVE and particle debonding is evident. Once the band traversed the RVE, further deformation was concentrated within the band of plasticity and the debonding process was halted.

**Influence of interface strength, transverse shear**

For this case, the interface strengths, $t_{n}^{c}$ and $t_{s1}^{c}$, have been varied under transverse shear, similar to the transverse tension case. The resultant stress-strain graphs are presented in Figure 4.19. The results are again normalised by $\tau_{m}^{c}$. 

Figure 4.17.: Stress strain results under in-plane shear
4.3 Results

The sequence of damage progression under transverse shear involved damage initiation at the interface, followed by matrix yielding under shear. It was shown in Figure 4.6 that during elastic deformation $t_n$ is slightly higher than $t_{s1}$ under transverse shear. Therefore, there is a slightly higher failure strength associated with high $t_n^c$ than a high $t_{s1}^c$. This can be seen in Figure 4.19, as the highest shear strength for the composite was found when a high interfacial normal strength was used. Under transverse shear, the maximum strength of the composite is approximately around 75-80% of the matrix shear strength, as shown in Figure 4.19. This can be attributed to the stress concentrations associated with the fibre arrangement inducing plastic yielding. This behaviour was confirmed by varying the yield stress of the matrix under shear, $\tau_m^c$, and varying the interface strength accordingly, where similar behaviour was found.

Similar to the transverse tension case, low interface strengths resulted in high levels of debonding around all fibres. For this case, the crack path at final failure occurred along a shear plane, orientated at approximately 45° to the applied loading axis.

Figure 4.18.: Plastic shear strain (PEEQ) band under transverse shear (Note: a deformation scale factor of 2 has been applied for this figure)
4.3 Results

No experimental data for the response of the material under transverse shear is currently available, as such tests are difficult to carry out in practice. A prediction for the shear strength of the composite of 57.5 MPa has been made using the data in Table 4.4, which has provided good correlation to experimental data in transverse tension and in-plane shear. In McCarthy et al. (2010) the strength of the material under transverse

Figure 4.19.: Stress-strain results under transverse shear (a) Variation of Mode I interfacial strength (b) Variation of Mode II interfacial strength
shear is assumed to be 50 MPa. The transverse shear strength is lower than the in-plane shear strength, as no debonding occurs under in-plane shear, while debonding under transverse shear reduces the shear load carried by the fibres, reducing the transverse shear strength.

4.4. Conclusions

Micromechanical models were used to investigate damage evolution in a high fibre volume fraction composite. A single fibre simulation was used to demonstrate that under transverse tension, the interfacial traction in the normal direction, $t_n$, was twice the shear traction, $t_{s1}$. Fibre interactions were seen to produce further amplification of $t_n$, relative to $t_{s1}$, according to the proximity of the neighbouring fibres in an RVE with multiple fibres.

Negligible increase in composite transverse tensile strength was produced through raising the interfacial shear strength, $t_{c1}^c$. Under transverse tension failure is dominated by debonding in the normal (Mode I) mode.

Under transverse shear loading, it was found that the magnitudes of $t_n$ at the interface, are slightly higher than the magnitudes of $t_{s1}$. Through variation of the interfacial strengths, the transverse shear strength of the composite material was seen to reach an upper limit at approximately 80% of the shear strength of the matrix material. The weak effect of the interfacial shear strength in the transverse plane has been identified.

The stiffening effect of the material under in-plane shear has been analysed. The results show that strain hardening can be attributed to the rotation of the fibres within the matrix of the RVE. The increase in shear stress and the increased longitudinal stress combined to produce the observed global strain hardening effect.

The observed failure response of the HTA/6376 composite material was reproduced. The interfacial strengths were tuned to provide good correlation with available experimental data under three different loadings, where different failure criteria are active. The novel aspect of this chapter is that it is shown that one set of interfacial strengths
4.4 Conclusions

can be used to replicate the failure behaviour of the composite material. The ability to accurately model the failure characteristics and strengths in three dimensions allows the current models to be applied to different geometries and load cases. Inter-laminar failure and adhesive joints require consideration of a three dimensional stress state to which the current models are very well suited.

The model developed here created a basis on which the following chapter is extended, to consider the loading case at the interface between a composite and an adhesive in a bonded joint.
5. Micromechanical modelling of damage processes at composite-adhesive interfaces

5.1. Introduction

In this chapter, a microscale model is developed to investigate damage processes at the interface between the adhesive and the composite in a composite joint under Mode I and Mode II loading.

Adhesive joint failure is most commonly approached using macroscale models, where the different damage processes are represented using cohesive zones or damage laws. Kim et al. (2008) focus on two failure modes, where damage of the adhesive includes separation of the interface between adhesive and composite, and secondly, delamination within the composite, using a damage indicator in the composite region and a plasticity law for the adhesive. Cohesive zone solutions to the problem include those of Li et al. (2006) where cohesive elements were introduced to the necessary regions during damage propagation and Sörensen et al. (2009), where cohesive zone parameters account for composite failure respectively.

In Chapter 2, examples of published microscale analyses of the deformation of adhesive layers were summarised. It is shown that these studies, which concentrate on

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1 The results of this chapter have been published in *Composites Science and Technology* (O’Dwyer et al., 2013a).
5.2 Model description

the heterogeneous nature of toughened adhesives, do not consider the microstructure of adjoining composite adherends. Despite this, good phenomenological microscale damage responses were produced. Figures from Salomonsson and Andersson (2008), using an epoxy adhesive with mineral filler is given as an illustrated example of these procedures in Chapter 2.

A micromechanical model of a composite material is described in Chapter 4, capable of producing accurate predictions under three different load cases, where differing failure mechanisms are active. The investigation has shown fibre-matrix debonding and plastic yielding of the matrix to be the critical damage processes in the composite ply.

In this chapter, the three dimensional microscale model, developed in Chapter 4 for the case of damage in a composite ply, is extended to consider an adjoining adhesive layer. A damageable interface between the composite and adhesive regions is included to investigate the microscale implications of varied bond strength on failure behaviour. The same adhesive and composite materials used in Chapter 3, are represented in the micromechanical model, and a visual comparison is made between the deformation results of the adhesive layer model and the experimental test results.

5.2. Model description

The FE model developed here consists of a microscale Representative Volume Element (RVE) of the interface between a composite ply and an adhesive layer, away from the edges of an adhesive joint. The two sections are separated by a damageable interface, referred to here as the adhesive-adherend interface. The model is shown in terms of an adhesive single-lap joint with composite adherends in Figure 5.1, where the axial load, F, on the joint induces Mode I and Mode II deformations at the composite-adhesive interface. The indentation technique used by Zheng and Ashcroft (2005) allowed stiffness measurements to be taken extremely close to the composite-adhesive boundary, and found no gradient of stiffness existed close to the boundary. This al-
5.2 Model description

Figure 5.1.: Adhesive layer model as applied to a composite joint, including local reference orientations. The dimensions L and x are the width of the RVE and the weakened central region of the adhesive-adherend interface respectively.

allows the assumption in this chapter, that no interphase zone exists, i.e. no gradient of material stiffness exists between the adhesive and the composite. A mesh sensitivity study was carried out to demonstrate the independence of the models to the number of elements, and is presented in section B.2. It was also ensured that the results are independent of the model size. The results of three different sized RVEs are presented in section B.1.

5.2.1. Adhesive layer properties

The adhesive used here, 3M 9323 B/A, is a high strength, two part epoxy adhesive (3M, 2010), as described in Chapter 3. This adhesive was examined in detail by Krenk et al. (1996), where the dependance of the initiation of plastic behaviour on hydrostatic stress was highlighted. The Mohr-Coulomb yield criterion is used here to capture this behaviour. The Mohr-Coulomb model has been successfully used to describe the microscale yield behaviour of a similar epoxy in a composite epoxy matrix system (O’Dwyer et al., 2012, Vaughan and McCarthy, 2011a,b, Krenk et al., 1996, González...
5.2 Model description

and Llorca, 2007, Totry et al., 2010). It is assumed that the adhesive under investigation is homogeneous at this scale. This is apparent from the in-situ and post failure SEM microscopy in Chapter 3, as no particles or voids are visible.

The details of the governing Mohr-Coulomb criterion are given in section 2.3.2 and in greater detail in section 4.2.3 and are not repeated here.

The material parameters, determined from the data of Krenk et al. (1996) are presented, along with the elastic properties of the material in Table 5.1. The value of the friction angle $\phi$ was approximated as $15^\circ$, an appropriate value for such a material suggested by Totry et al. (2010). As in (Vaughan and McCarthy, 2011a,b), non associated flow is assumed, and the angle of dilation used is zero.

<table>
<thead>
<tr>
<th>Table 5.1.: Material properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>HTA Fibre</td>
</tr>
<tr>
<td>$E_{11}$</td>
</tr>
<tr>
<td>$E_{22}$</td>
</tr>
<tr>
<td>$E_{33}$</td>
</tr>
<tr>
<td>$\nu_{12}$</td>
</tr>
<tr>
<td>$\nu_{23}$</td>
</tr>
<tr>
<td>$\nu_{31}$</td>
</tr>
<tr>
<td>$G_{12}$</td>
</tr>
<tr>
<td>$G_{23}$</td>
</tr>
<tr>
<td>$G_{31}$</td>
</tr>
<tr>
<td>$\phi$</td>
</tr>
<tr>
<td>$c$</td>
</tr>
</tbody>
</table>

The stress-strain plots for the matrix and the adhesive materials, as predicted using the Mohr-Coulomb criterion are presented in Figure 5.2. In each case, it can be seen that the material under compression is stronger than that under tension. The stiffness of the adhesive material is also seen to be much lower than that of the matrix.

5.2.2. Adhesive-adherend interface properties

The adhesive-adherend interface has been modelled using a layer of cohesive elements, the stiffness of which has been chosen to maintain stress continuity with the adjoining matrix. A traction-separation law has been employed to capture the damage
5.2 Model description

Figure 5.2: Comparison of the stress-strain behaviour of the adhesive and matrix materials.

behaviour of the adhesive-adherend interface. Damage initiation criterion is predicted by the quadratic stress criterion, as defined in Equation 5.1:

\[
\left( \frac{< t_n >}{t_{nc}^n} \right)^2 + \left( \frac{t_s}{t_{sc}^s} \right)^2 = 1
\]

(5.1)

where \( t \) is the traction across the cohesive element in the orientation designated by the subscript (n=normal, s=shear), and the superscript \( c \) refers to the critical damage initiation traction.

Once the damage initiation criterion is achieved, the stiffness of the interface is reduced from its value, \( k \), according to the value of a damage variable, \( D \). The damaged stiffness of the material, \( k^d \) (see Figure 5.3), is given by Equation 5.2.

\[
k^d = (1 - D)k
\]

(5.2)

where \( D \) increases monotonically from 0 to 1. The rate of increase of \( D \) is controlled by a mode independent, energy based, damage evolution law, where the critical fracture energy, \( G^c \), of the element is specified as the area under the traction separation curve,
Figure 5.3.: Adhesive-adherend interface traction-separation curve. Note: diagram not to scale.

shaded in Figure 5.3. The damage variable, $D$ is also subject to a viscosity parameter, $\mu$, which limits the rate of change of the damage variable. The implementation of the viscosity parameter is given in Equation 5.3, where $D$ is the damage variable to be used in Equation 5.2, $D_s$ is the value of the damage variable, if the traction-separation law was to be strictly implemented and $\dot{D}$ is the size of the current increment of the damage variable.

$$D = D_s - \mu \dot{D}$$

(5.3)

The initial traction-separation parameters are given in Table 5.2. The value of $G^c$ has been chosen to promote brittle behaviour. This energy value is intended to represent the fracture energy of the interface only, and therefore is lower than would be expected from the failure of an epoxy adhesive. Plastic yielding of the adhesive is included in the damage response of the model, therefore increasing the total fracture energy closer to an appropriate level for an adhesive bond. This is in accordance with the experimental SEM observations in Chapter 3, where crack growth was seen to propagate rapidly along the composite-adherend interface.
5.2 Model description

**Table 5.2.: Adhesive-adherend interface cohesive parameters**

<table>
<thead>
<tr>
<th>Adhesive-adherend interface</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>$k_n$</td>
<td>$3.64 \times 10^{13}$ N/m</td>
</tr>
<tr>
<td>$k_s$</td>
<td>$1.4 \times 10^{13}$ N/m</td>
</tr>
<tr>
<td>$G_c$</td>
<td>10 N/m</td>
</tr>
<tr>
<td>$\mu$</td>
<td>0.001 /s</td>
</tr>
</tbody>
</table>

5.2.3. Composite ply properties

The composite material used is HTA/6376, which has been microstructurally characterised in Chapter 4, in addition to (Vaughan and McCarthy, 2011a,b). The NNA has been adapted in this chapter to include complete fibres and 1 μm thick resin rich regions at the top and bottom of the RVE, as seen in Figure 5.4. This avoids stress concentrations due to partial fibres at the interface with the adhesive. The bonded surface between the adhesive and composite is assumed to be planar and lies parallel to the 1-3 plane, as shown in Figure 5.1.

The matrix material was modelled as an elastic-perfectly plastic material, with yielding determined by the Mohr-Coulomb yield criterion, described previously. The parameters for the Mohr-Coulomb model, based on properties determined experimentally in (Fiedler et al., 2005) are given in Table 5.1.

The carbon fibres are modelled as linear elastic and transversely isotropic. Experimental data from Hojo et al. (2009) are presented in Table 5.1 and refer to the orientations shown in Figure 5.1.

Damage has been incorporated in the composite through fibre-matrix debonding, which is controlled through a traction-separation law. The stiffness of the fibre-matrix interface is the same as that for the adhesive-adherend interface, given in Table 5.2. The critical tractions, under peel and shear at the interface, $t_n^c$ and $t_s^c$ respectively, and the critical fracture energies under peel and shear, $G_n^c$ and $G_s^c$ respectively, are presented in Table 5.3. This set of parameters, used in Chapter 4, were found to produce good correlation with experimental data. The Mode I and Mode II conditions applied here correspond to the transverse tension and in-plane shear cases investigated in
Table 5.3.: Fibre-matrix interface parameters, measured radially from the centre of each fibre.

<table>
<thead>
<tr>
<th>Fibre-matrix interface</th>
</tr>
</thead>
</table>
| $t^c_n$                | 85 MPa  
| $t^c_s$                | 125 MPa  
| $G^c_n$                | 10 N/m  
| $G^c_s$                | 25 N/m  

Figure 5.4.: Schematic of adhesive layer model

Chapter 3. Fibre rotation was not induced in the shear conditions as applied to the adhesive layer RVE.

5.2.4. Mesh and boundary conditions

The NNA was used to create three dimensional composite microstructures of width, $w = 100\ \mu m$, and depth, $d = 0.7\ \mu m$, similar to those in Figure 5.4. The height of the composite region, $h_c$, varied, depending on the fibre distribution in each individual RVE (typically $h_c > 45\ \mu m$). The mesh for the adhesive, fibre and matrix regions consisted of C3D8 elements with a limited number of C3D6 filler elements. The cohesive zones in the composite section consisted of COH3D8 elements. All cohesive elements were 0.1nm thick, thus assuming a negligible thickness (ABAQUS-Inc., 2010). The thickness
of the adhesive layer determined the number of elements in each model, and the total number ranged from approximately 116,000 to 165,000 elements.

The boundary conditions assume periodicity on model surfaces lying parallel to the 1 - 2 and 2 - 3 planes, in Figure 5.4. Similar conditions were used in Salomonsson and Andersson (2008), for an adhesive micro-model. To represent the full thickness of a composite ply, linear springs were applied to the bottom surface of the model, with an elastic stiffness equal to the through thickness stiffness of a composite ply. A constant displacement condition was applied to the top surface of the adhesive. The applied displacements were used to create Mode I (peel) and Mode II (shear) deformations.

To ensure the solution is not affected by RVE size, it was verified that the ratio of fibre diameter to RVE width was consistent with the data in Kulkarni et al. (2009). Normalisation of the data in Kulkarni et al. (2009), with relation to average particle diameter ($\phi$) gives independence from size of the RVE ($w_{RVE}$) above a value of $w_{RVE}/\phi = 6.25$. For the RVEs used here, the constitutive RVE dimensions give $w_{RVE}/\phi = 15.15$, where $w = 100\,\mu m$ and $\phi = 6.6\,\mu m$. This lies well above the value of 6.25, from Kulkarni et al. (2009), indicating that the size of the RVE is sufficient to produce accurate results.

In all cases, Python scripts were used to create geometries, which were submitted to the ABAQUS/Standard FE solver (ABAQUS-Inc., 2010).

5.3. Results

5.3.1. Model stiffness

The elastic stiffness of the model can be approximated analytically, using the homogenised elastic properties of the respective layers, as a validation of the elastic response of the adhesive layer model:

$$\frac{1}{k_T} = \frac{1}{k_a} + \frac{1}{k_c}$$

(5.4)

where $k_T$ refers to the total stiffness of the model, $k_a$ and $k_c$ are the stiffnesses of
the adhesive and composite layers respectively. The values for $k_a$ and $k_c$ are obtained using the standard spring stiffness equations for each layer:

$$k = \frac{EA}{h}$$  \hspace{1cm} (5.5)

where $E$ is the modulus of elasticity, $A$ is the area and $h$ is the thickness of the relevant layer. This calculation has been carried out for peel and shear conditions over a range of adhesive thicknesses. A corresponding FE analysis on models of varying adhesive thickness was undertaken. The models were deformed elastically, and homogenised elastic moduli were calculated in each case. The results are presented in Figure 5.5, showing the overall elastic modulus, normalised by the elastic modulus of the adhesive, as functions of adhesive thickness ($h_a$), normalised by the average fibre diameter ($d_f = 6.6$ μm). It can be seen that, as the thickness of the adhesive layer is increased, the modulus approaches that of the adhesive. Similarly, the modulus tends towards that of the composite section ($E_{comp} = 8.4$ GPa, $G_{comp} = 3.8$ GPa) as the thickness of the adhesive decreases. Under Mode I conditions, excellent correlation was achieved between Equation 5.4 and results from non-periodic models. Once periodicity is considered, Poisson contraction is restricted in the adhesive, causing the modulus of the models to be higher than the analytical result in Mode I. It can be seen that the FE results and Equation 5.4 are almost indistinguishable under Mode II loading.

### 5.3.2. Adhesive-adherend interface Strength Variation

In this section, the effect of adhesive-adherend interface strength on the response of the bonded composite joint is examined. The adhesive thickness was fixed at a value of 25μm ($h_a/d_f = 3.79$). Figures 5.6a and 5.6b show typical stress-strain results from models under Mode I and Mode II deformations respectively at low, intermediate and high adhesive-adherend interface strengths. The overall strength of the composite bond ($\sigma_{max}$ and $\tau_{max}$) are taken as the maximum stress value from these figures.
5.3 Results

![Graph](image)

(a) Correlation of elastic modulus, $E_{22}$, with analytical solution

(b) Correlation of elastic modulus, $G_{12}$, with analytical solution

**Figure 5.5.** Correlation of model elastic moduli with analytical solution.
5.3 Results

Figure 5.6: Stress-strain results

(a) Stress-strain results under Mode I deformation for high strength ($t_n^c = 100$ MPa), intermediate ($t_n^c = 70$ MPa) and low strength ($t_n^c = 50$ MPa) adhesive-adherend interfaces.

(b) Stress-strain results under Mode II deformation for high strength ($t_s^c = 40$ MPa), intermediate ($t_s^c = 25$ MPa) and low strength ($t_s^c = 15$ MPa) adhesive-adherend interfaces.
5.3 Results

**Low strength adhesive-adherend interface**

Contours of equivalent plastic strain are shown in Figure 5.7a, for the low strength adhesive-adherend interface \( t_n^c = 50 \text{MPa} \), after separation of the adhesive and composite regions under Mode I loading. The level of plastic deformation in the composite is low, and fibre-matrix debonding is not observed. It may be seen that plasticity remains localised in the region of the adhesive closest to the composite, while the damage process is confined to the adhesive-adherend interface. Damage initiated directly above fibres closest to the adhesive-adherend interface, as the non-uniform stress distribution in the matrix region propagated through the adhesive-adherend interface to the adhesive region, causing multiple damage initiation sites. The irregular distribution of plasticity within the adhesive layer, due to the non-uniform separation of the adhesive from the composite is noted. As shown in Figure 5.7b, the pattern of Mode II deformation is similar, with a clean separation of the adhesive and composite, and no plasticity or debonding occurred in the composite at low adhesive-adherend interface strengths.

**Intermediate strength adhesive-adherend interface**

Failure behaviour similar to that of a weak interface was found for intermediate adhesive-adherend interface strengths, where failure occurred in the adhesive-adherend interface. Plasticity was found to be more extensive, as the adhesive-adherend interface is able to transfer sufficient load between the adhesive and composite regions for plastic shear bands to form in the adhesive, under Mode I loading. Localised fibre-matrix debonding occurring between the most tightly packed fibres in the composite is noted under Mode I loading, accompanied by limited matrix plasticity as shown in Figure 5.8a.

A plot of equivalent plastic strain after failure under Mode II conditions is shown in Figure 5.8b for an intermediate strength adhesive-adherend interface \( t_n^c = 25 \text{MPa under Mode II load} \). At the indicated regions, the adhesive-adherend interface remained
5.3 Results

**Figure 5.7.**: (a) Mode I failure at a weak adhesive-adherend interface ($t_c^f = 50$ MPa) showing small amounts of plastic yielding in the adhesive only. (b) Mode II failure of a weak adhesive-adherend interface ($t_s^f = 15$ MPa), separation of the adhesive and composite occurred without significant plastic yielding.
5.3 Results

sufficiently intact to cause significant shear yielding of the matrix. No debonding was detected in the composite and regions of matrix plasticity were extremely small and localised near tightly packed fibres.
Figure 5.8: (a) Equivalent plastic strain after Mode I failure of an intermediate strength ($t_{ic} = 70$MPa) adhesive-adherend interface. Uneven failure of the adhesive-adherend interface, due to proximity of the surface fibres is visible. (b) Mode II failure of an intermediate strength ($t_{is} = 25$MPa) adhesive-adherend interface. Regions marked (A) are regions of high plasticity in the adhesive, due to uneven failure of the adhesive-adherend interface.
5.3 Results

High strength adhesive-adherend interface

Figure 5.9a illustrates plastic strain contours for a high strength interface \( t_n^c = 100 \) MPa) under Mode I loading. It may be seen that regions of intense matrix plasticity connect debonded fibres, to create a crack path through the composite region. Plastic yielding is also seen in the adhesive, where shear bands nucleate from areas adjacent to the fibres closest to the adhesive-adherend interface.

Mode II loading of a high strength adhesive-adherend interface results in yielding of the base of the adhesive layer, close to the composite region. As shown in Figure 5.9b, no damage was caused to the composite under Mode II deformations, as the yield point of the adhesive was such that plastic deformation occurred in the adhesive prior to any debonding within the composite. This matches the experimental results of Chapter 3, where no composite damage was found under Mode II conditions.
**Figure 5.9:** (a) Mode I failure at a strong adhesive-adherend interface ($t_{ic} = 100$MPa). The entire failure process is within the composite region. (b) Mode II deformation of a strong ($t_{is} = 40$ MPa) adhesive-adherend interface. Deformation was confined to the adhesive layer in the form of plastic yielding, and no debonding or damage at the adhesive-adherend interface was found.
5.3 Results

In Figure 5.10a, the strength of the composite joint is plotted against the adhesive-adherend interface strength \( t_c \). Linear data fits have been applied to the data points. It can be seen in Figure 5.10a, that under Mode I loading, bond strength increases linearly with respect to adhesive-adherend interface strength until a maximum is reached at an adhesive-adherend interface strength of approximately \( t_c = 77 \) MPa. Any further increase in adhesive-adherend interface strength produces no increase in model strength. This corresponds to the condition where damage processes transfers from the adhesive-adherend interface to the composite region, as the interfacial strength exceeds that of the composite. A similar trend was demonstrated experimentally by Kim et al. (2008), discussed previously in Chapter 2, where increasing adhesive strength led to composite failure, once the adhesive was stronger than the composite.

Similar trends are found under Mode II, in Figure 5.10b, where a plateau is reached once the shear strength of the interface exceeds \( t_s = 25 \) MPa. This corresponds to the point where the dominant deformation process transfers to the adhesive region from the interface. The critical adhesive-adherend interface strength is slightly lower than the yield point, under shear, of the adhesive. This can be attributed to stress concentrations, caused by the fibres, initiating premature adhesive yielding.
5.3 Results

(a) adhesive-adherend interface model strength variation under Mode I

(b) Bond shear strength variation under Mode II

**Figure 5.10.** Bond strength variation
5.3 Results

Table 5.4.: Mode I stress at failure and failure type for each model.

<table>
<thead>
<tr>
<th>w</th>
<th>S</th>
<th>$\sigma_{max}$ (MPa)</th>
<th>Fail type</th>
<th>$\sigma_{max}$ (MPa)</th>
<th>Fail type</th>
<th>$\sigma_{max}$ (MPa)</th>
<th>Fail type</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.95</td>
<td>79</td>
<td>Com</td>
<td>76</td>
<td>Com</td>
<td>80</td>
<td>Com</td>
</tr>
<tr>
<td>0.2</td>
<td>0.95</td>
<td>80</td>
<td>Adh</td>
<td>80</td>
<td>Mix</td>
<td>80</td>
<td>Mix</td>
</tr>
<tr>
<td>0.4</td>
<td>0.95</td>
<td>80</td>
<td>Adh</td>
<td>80*</td>
<td>Mix*</td>
<td>79</td>
<td>Mix</td>
</tr>
<tr>
<td>0.6</td>
<td>0.95</td>
<td>79</td>
<td>Mix</td>
<td>81</td>
<td>Adh</td>
<td>81</td>
<td>Adh</td>
</tr>
<tr>
<td>0.8</td>
<td>0.95</td>
<td>79</td>
<td>Mix</td>
<td>80</td>
<td>Adh</td>
<td>80</td>
<td>Adh</td>
</tr>
<tr>
<td>1</td>
<td>0.95</td>
<td>79</td>
<td>Adh</td>
<td>81</td>
<td>Adh</td>
<td>79</td>
<td>Adh</td>
</tr>
</tbody>
</table>

Com=composite failure, Adh=interface failure, Mix=mix of composite and interface failure

*Denotes the model shown in Figure 5.11.

5.3.3. Failure modes

Failure at the adhesive-adherend interface and within the composite region has been reported from tests on adhesively bonded composite joints (Feih and Shercliff, 2005, Kim et al., 2006). However, a mix of the two failure modes on a single failure surface is also a common result in published works, as shown in Chapter 2, and was also evident in the experimental work in Chapter 3. It has been shown that under Mode I conditions, the current models are capable of producing failure in the composite and the adhesive-adherend interface separately, depending on the adhesive-adherend interface strength but mixed failure has not been observed. In this section, a variation in adhesive-adherend interface strength is considered under Mode I (The same range of failure types was not observed in Mode II loading and therefore Mode II loading is not considered in this section). A central section of the adhesive-adherend interface of the adhesive layer microscale model is weakened in this section. The strong region is assigned a strength of $t_n^c = 78$MPa, (just exceeding the strength at which failure was entirely within the composite, see Figure 5.10a). A parameter $S$ is introduced as the strength of the weakened region, normalised by the strength of the strong region ($t_n^c$).
5.3 Results

Plastic strain

0.00
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.08
0.09
0.10
4.86

Figure 5.11.: Mixed failure under Mode I loading, with regions of composite and interfacial failure marked.

The length of the weakened region, \( x \) (see Figure 5.4), is varied with relation to the overall adhesive-adherend interface length, \( L \). The parameter \( w \) is introduced to characterise the weakened length as \( w = \frac{x}{L} \). Six different lengths of weakened regions were investigated, corresponding to \( w = 0, 0.2, 0.4, 0.6, 0.8, 1.0 \). \( w = 1 \) corresponds to an interface weakened along its entire length, while \( w = 0 \) corresponds to a full strength interface. Three RVEs were analysed at each value of \( w \) (Total number of RVEs=18), meaning that 18 realisations of the microscale model were created. Here, mixed failure is defined as failure occurring in the adhesive-adherend interface and the composite regions in the same RVE.

Investigations carried out corresponding to \( S = 0.8, 0.9 \) resulted in failure of the adhesive-adherend interface for all values of \( w \), as failure initiated and propagated in the adhesive-adherend interface without debonding occurring in the composite. A central region weakened to \( S = 0.95 \) (\( t_{nc}^f = 74 \) MPa) produced mixed failure in some cases. Mixed failure created the failure pattern shown in Figure 5.11. It can be seen that the debonded fibres are predominantly located beneath the section of adhesive-adherend interface which remained intact. The debonded fibres form the weak point in the loading path in this region, as the adhesive-adherend interface directly above remained intact. In the cen-
5.3 Results

tral region of the RVE, damage initiated at the weakened region of adhesive-adherend interface, preventing debonding of the adjacent fibres. This was common across all mixed failure models, however, some slight overlapping of debonding and interfacial damage was found to occur.

A comparison is made between the appearance of a failure surface from the bonded joint tests of Chapter 3, and the resultant failure appearance from the micromodels. An SEM micrograph from this chapter is shown in Figure 5.12a, and is compared to an example of modelled mixed failure in Figure 5.12b. In Figure 5.12b, the model from Figure 5.11 has been altered, as indicated, so that any region of composite beneath debonded fibres or failed adhesive-adherend interface has been removed and the resulting deformed shape has been patterned appropriately for clarity. In Figure 5.12a, failure in the composite is marked (A) and highly deformed adhesive surface is labelled (B). Fibre surfaces revealed through debonding provide a good correlation to similarly revealed fibres in the experimental results. Deformed matrix material also provides a good phenomenological match to the experimental figure. Therefore, it is reasonable to assume that the same experimental failure mechanisms are also active in the adhesive layer model.

Table 5.4 presents the joint strength and type of failure which was produced in the model. It can be seen that the joint strength remained relatively constant (within 7%) over all the models, irrespective of the failure type. Also to be noted from Table 5.4, is a general trend toward full adhesive failure is seen as the size of the weakened region is increased. These observations indicate that the presence of mixed failure was not entirely dependent on the presence of a weakened region. It can be seen that no relationship between the presence of mixed failure and the size of the weakened region exists. Since the only difference between the RVEs is the fibre distribution, it is possible to conclude that the fibre distribution of each RVE, combined with the presence of a weakened region of adhesive-adherend interface caused mixed failure behaviour.
5.3 Results

(a) Mixed failure of an adhesively bonded composite joint under Mode I dominant conditions. (A) is a region of fibre tear failure, region (B) is highly deformed adhesive.

(b) Mixed failure model, regions marked (A) and (B) are as in Figure 5.12a. Note: for clarity, the model has been repeated twice in the 3-direction and thirty times in the 1-direction.

Figure 5.12.: Qualitative comparison of experimental data and model results.
5.4 Conclusions

A microscale adhesive bond model has been developed, to expand on the composite microscale model of Chapter 4, and has been shown to be capable of reproducing the range of micromechanical failure mechanisms commonly found in experimental analyses of adhesive joints with composite adherends. Damage was assessed within the composite and at the adhesive-composite adhesive-adherend interface. Mode I and Mode II conditions were applied to the model, to investigate the microscale damage processes under each mode of deformation. The results show that damage in the adhesive-adherend interface and composite region dominated under Mode I loading, while adhesive yielding and adhesive-adherend interface damage was found under Mode II deformation.

A parameter study was undertaken, to examine the effect of variation of the adhesive-adherend interface strength on the strength and failure processes of the bond. This was carried out through altering the strength of the cohesive elements in the interface. Under Mode I loading, low interface strengths produced failure at the interface, and interfacial strengths exceeding the strength of the composite region produced composite failure. Under Mode II loading, damage was confined to the adhesive-adherend interface at low interface strengths and plastic yielding of the adhesive prevented damage in the composite at higher interface strengths. Plastic yielding of the adhesive provided the upper limit to the shear strength of the model, while the upper bound to the Mode I strength was provided by the strength of the composite ply.

Mixed failure behaviour was investigated, as experimental results often show a combination of failure mechanisms occurring in close proximity. Such behaviour was produced through weakening a central region of the composite-adhesive interface. No correlation between the size of the weakened region, and the presence of mixed failure was evident. It is concluded that the local fibre distribution, combined with the presence of a weakened region of adhesive-adherend interface is the cause of the mixed failure.
5.4 Conclusions

The failure behaviour of the model has been compared to an SEM micrograph of a failure surface under Mode I dominant conditions, produced in the experimental work of Chapter 3. A clear correlation was evident between the models and the experimental data. Bundles of fibres from the composite, remaining on the adhesive surface, accompanied by regions of high adhesive plastic strain shown in the experimental work was reproduced by the adhesive layer model.
6. Utilisation of Micromechanical Models to Predict Joint Failure

6.1. Introduction

This chapter creates a link between the experimental testing of adhesive joints in Chapter 3 and the microscale analysis of adhesive bond failure in Chapter 5. Obtaining a link between the failure of the joints at the macroscale and the microscale damage processes is difficult, as large computing requirements preclude damage modelling of a real composite component in its entirety at the microscale. Popular solutions to this issue are submodelling, embedded cell modelling and cohesive zone modelling. Determination of traction-separation laws for a global scale FE model was undertaken in Ivankovic et al. (2004) through analytical analysis of experimental tests. In this chapter, the traction-separation parameters are extracted from microscale models, and applied to the global scale.

The standard submodelling approach involves extraction of boundary conditions for a microscale model from a set of global macroscale results. Boundary conditions for submodels can be extracted from points of interest in a structure, in locations of known stress concentrations. This procedure, however, transfers boundary conditions from the macroscale to the microscale, where damage progression can be predicted, with no convenient method of obtaining stiffness feedback to the macroscale once damage initiates at the microscale. Lee et al. (1999) also highlight that the periodicity constraint at the microscale is unsuitable to transfer to the macroscale as it represents a region of
constant strain. Therefore, in a region of high strain gradient, the strain across the RVE may be required to change by a significant amount, which cannot be achieved using the periodicity assumption. An example of such a case exists here at the ends of the adhesive layer (see Figure 3.4 and Figure 3.5).

A coupled micro-macroscale analysis is a feature of the embedded cell approach. A small region of a global model is replaced by a micromodel, through replacement of global elements with a microscale model. The embedded model is tied to the surrounding mesh, which contains the homogenised material properties of the material. In the case of composite materials, this approach is limited by the small dimensions of the fibres, relative to component size. This means that an extremely small volume of the global model is represented at the microscale, and will not exert a great influence on the stiffness of the global model, thus limiting the effectiveness of the method. The approach was used in González and Llorca (2006), where failure in a composite sample was predicted for a composite sample under bending. Fibre rupture was the main source of damage and frictional sliding of the fibres along the matrix was also considered. It was concluded here that the fibre failure mechanism was reproduced accurately, and good predictions of the maximum load were made. At strains beyond the maximum load, the simulation results deviated from the experimental data, due to the matrix cracking mechanism dominating the failure process (which was not included in the model).

The extremely small dimensions of microscale models, in comparison to the global scale model, require severe mesh refinement in the region of the embedded cell, which rapidly increases the number of elements in the simulation. The mesh refinement is particularly severe in the case studied here, where a model of approximately 50mm would need a mesh refinement to sub micrometer scale in order to get adequate continuity between the global mesh and the embedded cell. This degree of refinement increases the amount of computational time needed and therefore the practicality of the embedded cell model is limited in this case.

The submodelling and embedded cell approaches provide good resolution of the
stress field at the microscale, but require microscale solutions at the microscale at frequent intervals or at every increment of a solution and therefore provide computationally expensive results.

The extraction of traction-separation parameters from a microscale RVE of a heterogeneous adhesive was shown for a homogenisation approach in Matouš et al. (2008). Kulkarni et al. (2009) extended this approach, investigating the case of the debonding process of toughening particles in an adhesive. Parameters of a cohesive zone model for use in a global model were obtained from the RVE analysis. However, the global model was not presented in the publication.

The approach used in this chapter to link the microscale and the macroscale is outlined in the three steps of Figure 6.1. The first step is the microscale modelling of the adhesive-composite interface region using the RVEs of Chapter 5. Traction-separation parameters are extracted in step two, from the adhesive layer RVEs using the homogenisation technique, thus representing the damage processes in the adhesive, first composite ply, and the interface between the two materials. Here, the traction is calculated through homogenisation of the appropriate stress components, and the displacements are calculated as the displacement of the nodes at the top of the RVE. The curves from the RVE models are approximated to a bi-linear traction-separation law, containing the damage initiation traction and fracture energy of the RVE model. This traction-separation data is used in step three in a global scale model, where the predicted location of the crack path is meshed using cohesive elements, behaving according to the previously determined traction-separation law.

In this chapter, a two-dimensional model of the three point bend experiment in Chapter 3 is used as the global model, into which the traction-separation law is inserted. The load-displacement results are compared to the corresponding experimental data of Chapter 3, against which the predictive capacity of the micromodelling approach is assessed.
Step 1: Microscale Modelling

Step 2: Homogenisation

Microscale results

Extract:

\[ t_{\text{max}} \]

\[ G^c \]

Apply to cohesive zone

Step 3: Macroscale Modelling

Figure 6.1.: Procedure outline, linking the micro and macro scale modelling
6.2 Interpretation of micromechanical results

In Chapter 5, models were developed to investigate damage progression at the composite adhesive interface. These models are used again in this chapter as Step 1 of the multiscale modelling process. It was seen that composite damage occurred once the adhesive-composite interface strength exceeded that of the composite region. Lower strengths produced failure at the bondline, with decreasing levels of plasticity in the adhesive as the interface strength was reduced. Here, the same models are used to provide the initiation traction values and fracture energies for a global scale cohesive zone.

The second step of the process was completed through the creation of Python scripts, which extracted traction-separation curves from the RVE models in Chapter 5. A bilinear, energy based, traction-separation law was used, incorporating the maximum traction and fracture energies of the RVEs. The displacements of the nodes at the top of the RVE are used directly as the separation parameter in all cases. Two bondline strengths were analysed, representing strong and weak bondlines, with bondline strengths of 80 MPa and 50 MPa, respectively. The strongest model results predict failure in the first ply of the composite region, while the result from the weak RVE reflects separation of the composite and adhesive along the adhesive-composite interface. Traction-separation curves, extracted from typical RVE analyses are presented in Figure 6.2. The area under these curves is the fracture energy, which is given for the two Mode I cases, accompanied by maximum tractions in the normal (peel) and shear directions, in Table 6.1. Failure has been assumed at 110% of the strain to maximum traction in the strong Mode I case (indicated by the dashed line in Figure 6.2), as total failure of the RVE was not achieved in this case (due to the absence of a damage model for the matrix material). In the case of the weak Mode I case, the Mode I fracture energy was calculated as the entire energy under the indicated curve in Figure 6.2, as complete separation of the composite and adhesive occurred in this case.

Mode II initiation of failure is not expected under three point bending. The traction-
6.2 Interpretation of micromechanical results

Figure 6.2.: Traction-separation curves, extracted from the models in chapter 5, for strong and weak bondlines under Mode I, and under Mode II deformation.

separation parameters under Mode II are extracted following the same procedure as was used to extract the Mode I parameters. The traction separation curve is included in Figure 6.2 and the resultant data is presented in Table 6.1. The failure strain in the traction-separation diagram is at 3% applied strain, indicated by the dotted line in Figure 6.2. The in-situ analysis in Chapter 3 showed large deformation of the adhesive layer prior to failure in all cases. The value of 3% is below the failure shear strain for the adhesive reported in Krenk et al. (1996), where a shear test did not show failure before 5% shear strain. However, the modelling work in Chapter 5 showed very high local shear strains in the adhesive near the interface with the composite. Therefore a shear failure at 3% applied shear strain was chosen, which corresponds to a displacement of 4.4 μm. This is indicated by the dotted line in Figure 6.2. The global initiation traction under Mode II was 60 MPa.

The constraint effects of the periodic boundary conditions have been assumed to be negligible here, following the approach of Kulkarni et al. (2009), who used the same homogenisation technique on a periodic RVE to extract traction-separation data from a periodic RVE of a particle-filled adhesive.
### 6.3 Joint model

#### Table 6.1: Cohesive parameters

<table>
<thead>
<tr>
<th></th>
<th>Traction</th>
<th>Energy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$t^c_n$</td>
<td>$t^c_s$</td>
</tr>
<tr>
<td>Strong</td>
<td>160 MPa</td>
<td>60 MPa</td>
</tr>
<tr>
<td>Weak</td>
<td>108 MPa</td>
<td>60 MPa</td>
</tr>
</tbody>
</table>

#### Figure 6.3: Model details. The adhesive layer thickness is exaggerated for clarity (actual thickness: $50 \times 10^{-3}$mm).

### 6.3. Joint model

The two dimensional FE model of a joint under three-point bending, as used in Chapter 3 to illustrate the stress distribution through the adhesive layer was adapted for the study in this chapter. A schematic of the model, including important dimensions and boundary conditions, is given in Figure 6.3. Plane strain was assumed, to reflect conditions far away from the edges of the joint and the region of the adhesive layer was meshed using a single layer of cohesive elements. The adherend regions were meshed using CPE4 elements, and the cohesive region was meshed with COH2D4 elements. A displacement boundary condition ($\Delta_{\text{applied}}$) was applied, as shown in Figure 6.3. The total number of elements used was just under 32,000. A mesh dependence study was carried out to verify the independence of the solution from the mesh size.

Elastic moduli for the transversely isotropic composite material, from Ireman (1998) are presented in Table 6.2, and refer to the orientations indicated in Figure 6.3. No damage or plasticity was considered in the composite adherends.
6.3 Joint model

Table 6.2.: Transversely isotropic elastic properties of the composite material.

<table>
<thead>
<tr>
<th>Composite</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_{11}$</td>
</tr>
<tr>
<td>$E_{22}$</td>
</tr>
<tr>
<td>$G_{12}$</td>
</tr>
<tr>
<td>$\nu_{12}$</td>
</tr>
</tbody>
</table>

6.3.1. Cohesive element parameters

The cohesive element behaviour in the joint model is defined by the element’s initial stiffness ($k$), the damage initiation traction ($t^c$) and the fracture energy ($G^c$), as described in Chapter 5. The values for $G^c$ and $t^c$ are taken directly from the microscale RVE, as shown previously. The resultant data is provided in Table 6.1.

Two methods for determining the initial stiffness of cohesive zones are used in the literature. The first approach considers the cohesive zone to be a negligibly thin interface, of extremely high stiffness (see Chapter 4 and Chapter 5) ensuring that the strain energy in the elements does not affect the solution prior to damage initiation. This approach is not suitable here as the adhesive layer is replaced entirely by the cohesive elements. Therefore, the elastic strain energy in the layer is non-negligible. During elastic deformation, the response of the cohesive zone is to be indistinguishable from the response of the elastic material it represents. This implies that the stiffness of the adhesive layer RVE model, which includes the stiffness of the first ply and the adhesive layer, is not suitable to apply directly to a joint model cohesive zone, which represents the adhesive layer of the joint model only. The initial stiffness of the cohesive zone must represent the stiffness of the adhesive only. Following the approach of Campilho et al. (2013), the initial stiffness of the cohesive layer, is given as $k = E/T$, where $E$ is the elastic modulus of the material and $T$ is the characteristic length of the element. In this case, $E$ is the modulus of the adhesive material (see Table 6.3), and $T$ is the thickness of the layer (50 μm), giving the initial stiffness in peel ($k_n$) and shear ($k_s$) in Table 6.3.

The bi-linear traction-separation curves, as applied to the cohesive zone of the two
### 6.3 Joint model

**Table 6.3.** Adhesive material elastic moduli and equivalent cohesive element properties.

<table>
<thead>
<tr>
<th></th>
<th>Adhesive material</th>
<th>Cohesive element</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Normal</strong></td>
<td>$E_n = 2.8$ GPa</td>
<td>$k_n = 5600$ GPa</td>
</tr>
<tr>
<td><strong>Shear</strong></td>
<td>$E_s = 1.014$ GPa</td>
<td>$k_s = 2030$ GPa</td>
</tr>
</tbody>
</table>

dimensional joint model are shown in Figure 6.4, for one strong and one weak Mode I case. The Mode II curve, which is common to both strong and weak cases is included in Figure 6.4 also. It can be seen that the Mode I behaviour is more brittle than the Mode II behaviour, to account for the yielding behaviour of the adhesive in Mode II deformation, as seen in the in-situ data of Chapter 3. Comparing the traction separation curves in Figure 6.4 to those in Figure 6.2, it can be seen that the modification to the initial stiffness does not have a large effect on the traction-separation curve. The maximum tractions are transferred directly to the cohesive zone and are therefore identical to the RVE results. The maximum displacements in the cohesive elements are slightly larger than the results of the RVE models, due to the non-linear shape of the curves extracted from the RVEs. This is most obvious in the strong Mode I data of Figure 6.2, where plasticity in the adhesive material causes a deviation from the linear elastic response, which increases the fracture energy, and causes an increase in final failure displacement from $2.75 \mu$m to $3.5 \mu$m in Figure 6.4 is found.

### 6.3.2. Results of joint model

The normal stress distribution ($\sigma_{22}$) in the central region of the joint, during elastic deformation is shown in Figure 6.5. The response of the strong model and the weak model was identical during elastic deformation and two stress concentrations are visible in Figure 6.5; one at either side of the lapped area.

The stress distribution in the model changes after the initiation of damage in the adhesive layer. This is shown in Figure 6.6, at a point in the unloading part of the simulation, where the force on the joint is 60% of the maximum reached. The stress concentrations, as seen previously in Figure 6.5, remain visible. However, the maximum
6.3 Joint model

Figure 6.4.: Traction separation curves, as applied to the joint model adhesive layer.

Figure 6.5.: Plot of $\sigma_{22}$ before damage initiation (Mode I stress - see orientations in Figure 6.3).
6.3 Joint model

![Diagram showing stress concentrations and damaged cohesive elements](image)

**Figure 6.6.** Plot of $\sigma_{22}$ after damage initiation (see orientations in Figure 6.3). Stress concentrations and damaged cohesive elements are highlighted.

Opening normal stress is at the middle of the overlapped region. This is due to the damaged cohesive elements being unable to transfer load between the adherends.

In Figure 6.7, the load-displacement curves resulting from the joint model are compared to the relevant data from the experimental work in Chapter 3. The stiffness of the models was slightly higher than the results of the experimental tests. A good match was found between the load-displacement data of the low strength model and the weak

![Graph showing load-displacement curves](image)

**Figure 6.7.** Comparison of 2D joint model and experimental results.
6.3 Joint model

joints. This indicates that the brittle interfacial failure mode is captured accurately in the micromodel.

The strong model represents the maximum strength attainable with properties extracted from the adhesive layer RVE model, and represents failure through the surface composite ply. Applied at the macroscale, these properties return a maximum applied load of 130 N. This is 57% of the average strength reported from the strong set of joints in Chapter 3 (average maximum load = 225 N). The joint which failed through delamination of the composite in Chapter 3, thus matching the predicted failure behaviour of the strong micromodel, resulted in a maximum load of 192 N, producing a slightly closer match to the joint model.

The good match of the experimental load-displacement data and the results of the joint model is not repeated for high strength data. This discrepancy is investigated in the following section in terms of the fracture energy of the adhesive layer.

**Adhesive layer fracture energy**

The effect of greater energy absorption in the adhesive layer on the maximum load on the joint was investigated, while the maximum traction was not changed. The model was rerun, using $G^c_n = 560\text{N/m}, G^c_s = 280\text{N/m}$ (twice the magnitude of the energy values in Table 6.1), and it was found that the maximum applied load increased to 170 N. Increasing the energy, to four times the magnitude resulting from the microscale analysis ($G^c_n = 1120\text{N/m}, G^c_s = 560\text{N/m}$), the resultant maximum load applied to the joint model increases to 227 N, which is the average strength of the strong set of joints. The plots of all three cases are shown in Figure 6.8, and it is clear that the high energy cases provide a good match to the experimental data.

The higher fracture energy in the cohesive elements increases the load carrying capacity of the cohesive elements, after damage initiation, which produces a more gradual evolution of damage. The length along the bondline over which the cohesive damage parameter ($D$) rises from its undamaged state, 0, to its fully damaged state, 1, is shown in Figure 6.9, at 60% of the maximum load, during unloading of the joints. In each case,
6.3 Joint model

Figure 6.8: Effect of increasing fracture energy of the cohesive zone in the joint model. The damage had progressed to the approximate centre of the lap. Figure 6.9 isolates the damage process zone in each case only and the distance on the x-axis \( x_d \) is measured from the start of each damage zone, for a clearer comparison. It is clear that there is a more gradual increase in the damage parameter associated with the high energy cohesive zones. The damage process zone resulting from the microscale parameters covered 0.074 mm (1.4% of the total bondline), while the highest energy parameter produced a process zone which covered 6.8% (0.36 mm) of the bondline.

It is clear that the predicted fracture toughness resulting from the RVE micromodel is an underestimation of the fracture energy of the joints in Chapter 3. Two possible sources of additional energy absorption in the adhesive layer micromodels are proposed here.

The adhesive layer micromodels use data taken from the literature. While utmost care was taken during the manufacturing of the adhesive, it is possible that the hand-mixing technique, used in Chapter 3, results in a more ductile adhesive than reported in the adhesive datasheet (3M, 2010). It is possible that the adhesive did not produce the expected stiffness and strength parameters. It has been shown that because of bet-
6.3 Joint model

Figure 6.9: Evolution of the damage parameter, D, measured from the start of the damage zone, along the bondline.

The second source of additional fracture energy is the consideration of microcracking of the matrix and epoxy materials, in the adhesive layer micromodel. This failure mode, omitted from the microscale models, is a possible source of energy absorption, which if included, could improve the correlation with the experimental data. Examples of damage models which incorporate this damage process are presented in Chapter 2. Post failure fractography in Chapter 3 showed that joint failure at high loads included damage occurring just beneath the adhesive and composite surfaces. Initiation of energy absorbing microcracks at the composite-adhesive interface, accompanied by a limited degree of matrix cracking at the composite surface could increase the predicted joint strength. This study is left for future work.
6.4 Conclusions

A method for creating a link between the microscale RVEs of Chapter 5 and the macroscale joints of Chapter 3 is presented in this chapter.

A two dimensional FE model of the joints tested in Chapter 3, was created and subjected to three-point bend loading. Linearly elastic composite adherends were separated by a thin cohesive zone, which incorporated the damage response of the adhesive layer micromodel while maintaining the elastic properties of the adhesive layer. Traction-separation curves, extracted from the microscale models in Chapter 5, provided the macroscale damage response of strong and weak bonds under Mode I conditions.

The resultant load-displacement curves matched well to the stiffness data of Chapter 3. The failure load of the weakened joints was matched using parameters from the weakest adhesive layer micromodel. The correlation between the numerical and experimental data for the strong joint model was poor, as the models underestimated the average strength of the joints by 43%. Correlation with the experiments improved slightly when the failure mode of the joints is considered. When the damage parameters extracted from the micromodel which predicted composite failure are compared to the strength of the joint which failed through composite delamination, the underestimation is reduced to 32%.

It is concluded that brittle failure at the adhesive-composite interface is reflected well in the microscale models. The discrepancy between the joint model and the strong joints was attributed to the underestimation of the fracture energy in the traction-separation law extracted from the microscale models. Two factors are suggested as sources of the error; the material properties of the adhesive used in the microscale models exceeding that of the joints manufactured in Chapter 3, and the absence of a damage model to capture microcracking in the adhesive and matrix materials in the microscale models. A fourfold increase in fracture energy (due to the possible combination of the factors listed here) was shown to provide sufficient energy absorption to produce an excellent match to the average failure load from the experimental data.
7. Conclusions, design recommendations and future work

7.1. Conclusions

This work has analysed the micromechanical damage processes that occur at the adhesive-composite interface in a bonded joint, using novel experimental and numerical methods. The following chapter provides a summary of the main points resulting from the work undertaken. Design recommendations and proposals for possible future work, and possible scenarios for further deployment of the methods developed here are proposed.

7.1.1. SEM testing

A testing regime using miniature composite-adhesive joint specimens was carried out to examine the damage progression at the microscale using an SEM. The test set up used here allowed higher resolution of the damage processes during failure in comparison with examples from the literature. Interfacial crack growth and yielding of the adhesive were the dominant identifiable damage processes under in-situ examination in Mode I loading. It was clear from in-situ footage of Mode II tests, that yielding of the adhesive was the main visible mode of deformation, until final failure occurred...
7.1 Conclusions

through crack growth at the composite-adhesive interface. It was shown that surface
preparation of the joints, prior to bonding has a direct effect on the strength of the
joints, and also the damage mechanisms which are activated during failure.

Post-failure microscopy was used to provide additional details on the failure mech-
anisms that occurred during failure of the joints. This technique is used because the
in-situ analysis only permits a view of the crack growth along the edge of the joint, and
failure processes which occur away from the edges of the joint are hidden during the
in-situ analysis. Examination of the failure surfaces showed complex failure processes,
and could incorporate damage at three locations; in the composite, in the adhesive
and at the interface between the composite and the adhesive. It was also shown that
the damage type can vary over a small distance, and images supporting this have been
presented. It is extremely important that the testing regime showed a range of damage
types, as this provided examples of each for comparison to numerical micromodels.

The experimental techniques using the miniature joint specimens have been shown
to be capable of producing the same range of failure mechanisms that are found in the
literature, using full scale joint specimens. The advantage of the SEM method over full
scale analyses is that it permits much higher resolution in assessing the damage pro-
gression during failure of the joints. The testing set up is not expensive from a materials
point of view as it is possible to make a large number of joints from a small composite
laminate. A drawback of the configuration is that defects which would be regarded as
negligible in a larger joint become significant at this scale, increasing the level of scat-
ter in the resulting data. This type of testing, however, is shown here to be capable of
reproducing the range of failure mechanisms found in large scale joints.

7.1.2. Micromechanical modelling of composite and adhesive
bond damage

A micromechanical model has been developed to analyse the failure behaviour of a
composite ply, extending a model developed at the University of Limerick. The model
has been extended to investigate the damage processes at the interface between an adhesive layer and a composite adherend. The experimental testing confirmed that the combination of materials was capable of producing the appropriate range of failure types and examples of these failure types are compared with modelling results.

**Composite ply microscale model**

An existing two-dimensional microscale model was extended into three dimensions to capture the failure behaviour of a composite ply (HTA/6376). The damage response of the RVE was examined under three different loadings to assess the capability of the model to produce accurate results under loading conditions where different damage mechanisms are active. It was necessary to ensure that a single set of model parameters was capable of producing accurate global stress-strain response, and an accurate representation of the appropriate damage process, under each loading. Using the Nearest Neighbour Algorithm (Vaughan and McCarthy, 2010), RVEs containing fibre distributions which are statistically equivalent to the fibre distributions found in an actual composite were generated. Loading in the transverse plane determined the relative influence of the Mode I and Mode II strengths at the fibre-matrix interface. While sensitive to the Mode I strength, it was shown that variation of the Mode II interfacial strength produced negligible effects on the stress-strain results of the models, under in-plane loading.

The response of the model under in-plane shear loading was investigated separately, as the stress-strain response of the material was most accurately reproduced when debonding of the fibre and matrix was inactive. Under in-plane shear, the relationship between shear stiffening of a laminate and fibre rotation was quantified.

The models were shown to be largely insensitive to the interfacial shear strength parameter in the transverse plane. The assumption was made that shear deformations at the fibre-matrix interface was equal, regardless of application in the hoop, or axial direction relative to the fibre. This allowed the adoption of high interfacial shear strength, sufficient to prevent debonding under in-plane shear. The interfacial normal
7.1 Conclusions

strength was subsequently chosen to provide the best match to stress-strain data under transverse tension. The set of interfacial strength parameters showed that despite different damage mechanisms dominating under each loading, the model was capable of providing accurate stress-strain response in each case.

**Composite-adhesive interface microscale model**

The three dimensional composite ply RVE was extended to form a micromechanical model of the interface between an adhesive layer and a composite material. A finite element RVE was developed consisting of a homogeneous adhesive layer, attached to the previously developed composite micromodel, with a layer of cohesive elements separating the two regions. Upon application of Mode I and Mode II conditions to the model, stress concentrations caused by the non-uniform fibre distribution in the composite were seen to propagate into the adhesive. Parameter studies were conducted, varying the strength of the composite-adherend interface. This parameter is analogous to changing bond quality through surface preparation. Similarly to the testing of Chapter 3, changing the interfacial strength directly affected the damage mechanisms which were activated during failure. Mode I and Mode II deformations applied to models with low interface strength produced separation of the adhesive and composite regions. Initiation of this damage type was found to occur directly above fibres which lay close to the adhesive-composite interface. It was also shown that increasing the interface strength caused damage to occur in the composite region under Mode I conditions, while under Mode II conditions, failure occurred in the form of plastic yielding, just inside the adhesive region.

The conditions which lead to a combination of failure mechanisms were investigated using the microscale model. It was found that through the introduction of a weakened region in the centre of a strong bondline, failure was seen to occur at the composite-adhesive interface, in addition to within the composite region, in the same model. Using three different fibre distributions, it was also shown that the presence of the mixed failure was dependent on the fibre distribution near the weakened region
7.1 Conclusions

of bondline. This finding demonstrated the importance of modelling individual fibres and their distribution in the composite adherend of an adhesive joint micromodel.

7.1.3. Linking of micro- and macro-scales

The adhesive joint micromodel was shown to be capable of producing a range of damage types, similar to the range seen in Chapter 3. To link the micro- and macro scales and to further validate the experimental work, a two dimensional model of the joint specimens used in the experimental work of Chapter 3 was created. Damage parameters (maximum traction and fracture energies), were extracted from the micromodels, and were assigned to a cohesive zone representing the adhesive region of the joint model.

Good correlation was shown between the set of joints prepared with poor surface treatment and the two dimensional joint model containing the parameters of the weak adhesive layer model. This allowed the conclusion that the brittle interfacial failure mechanism which was observed in the experimental work was accurately reproduced in the microscale model. The results of the joint model using the strong set of micromodel parameters did not match well to the experimental work. This was attributed to the absence of additional energy absorbing failure mechanisms in the micromodels. It was shown that increasing the fracture energy of the cohesive zone by a factor of four produced the required strength for comparison with the average failure load of the strong set of joints. Two sources of additional energy absorption are proposed; damage in the epoxy materials (adhesive and matrix), and the ductility of the adhesive. A more diffuse damage mechanism in the adhesive and matrix phases of the model would increase the fracture energy of the models. It is suggested that the manufacturing process for the adhesive produced a more ductile adhesive than the material data used in the micromodel, causing the micromodels to produce low fracture energy.
7.2 Design recommendations

Testing carried out as part of this work showed that the strongest joints were produced when the adherend surface was prepared using both mechanical abrasion and degreasing. The surface preparation increased the strength of the interface between the adhesive and composite, and caused the failure process to move from the interface into the adherend and adhesive. The microscale models showed that failure within the composite, while capable of providing high strength values causes extensive damage to the composite adherend. This is undesirable in the case of re-joining of adherends (often required after maintenance work), as subsurface damage of the composite would reduce the strength of the re-used joint.

The global joint model highlighted the importance of energy absorption during failure to the maximum strength of the joint. For maximum joint strength, energy absorption within the adhesive should be maximised. Increasing the degree of plastic yielding in the adhesive (e.g. through additive toughening particles or reduction in stiffness and strength of the adhesive) as an energy absorption technique should be considered. The adhesive choice should be based on an optimal level of adhesive yield stress, as yielding of the adhesive has the additional advantage of confining the damage process to the adhesive region.

When considering adhesively joined composite adherends, it is recommended that bond failure should occur through the adhesive layer only. The most desirable configuration is the use of an adhesive which displays strength sufficient to provide structural support, yet yields appropriately, to avoid composite damage and provide high toughness during failure. This ensures that failure occurs within the adhesive layer, with minimal damage to the composite region. In accordance with the findings regarding fracture energy of the two dimensional joint model, this joint configuration also produces high strength joint.
7.3. Recommended Future work

The methods developed in this thesis for micromechanical analysis of damage at the composite-adhesive interface have shown potential to become an accurate method of predicting joint failure behaviour. This section outlines recommendations for future work to further develop the method, and different applications to which such an approach is well suited.

7.3.1. Epoxy damage model

The most immediate area for further research with relation to the micromodel is the development of a damage model for the epoxy matrix and the adhesive materials. Such models have been developed in the literature to good effect (as shown in Chapter 2), to produce promising results. The pressure sensitivity of the material adds extra complication to the issue and cannot be ignored. Microcracking of both the matrix and the adhesive materials can increase the energy absorbing capacity during crack growth at the composite-adhesive interface. Such an energy absorbing mechanism would improve the correlation between the modelling and experimental results. In particular, a damage model for the adhesive material would be a significantly enhance a micromechanics-based design tool. By varying the damage parameters of the adhesive region in the micromodel, it would be possible to investigate the effect shown through experimental results (Katnam et al., 2013), where a strong but brittle adhesive produces a weaker joint than the same composite adherends bonded using a lower strength adhesive, with high ductility. Such an effect cannot be detected using the current adhesive layer model, as only plastic deformation of the adhesive is considered, and damage behaviour of the adhesive is required to capture such an effect.

7.3.2. Geometric alterations

A number of geometric extensions to the model are possible areas for future work. Two assumptions in relation to the surface of the composite region which require fur-
7.3 Recommended Future work

Figure 7.1.: RVEs showing (a) current planar interface and (b) proposed wavy interface.

Further investigation are; the thickness of the resin rich region between the top of the fibres and the adhesive layer, and the planar interface between the composite and adhesive regions.

The interface between the composite and the adhesive is likely to follow a path related to the surface fibres of the composite region rather than the planar surface assumed here. Mechanical abrasion during surface preparation will also cause fibre surfaces to be revealed and become bonded directly to the adhesive. A schematic of the proposed alterations to the model is shown in Figure 7.1, where the wavy interface and fibres bonded to the adhesive are marked. It is possible that the adhesion of the fibre to the adhesive is stronger than the bond between the matrix and adhesive. This will introduce the possibility of pull-out of single fibres from the composite during a bondline failure, which has been shown to be an active failure mechanism in the experimental work of Chapter 3. Such a mechanism was not allowed for in the damage response of the adhesive layer RVEs as a layer of resin rich material was included. A wavy composite-adhesive interface would also have the effect of increasing the length of an interfacial crack path relative to the width of the RVE, thereby slightly increasing the resultant fracture energy of the RVE.
7.3 Recommended Future work

7.3.3. Alternative adhesive types

Epoxy adhesives often contain additive rubber, mineral or glass particles, to increase the toughness of the epoxy (Segurado and Llorca, 2006, Reina-Romo and Sanz-Herrera, 2011). The deformation and debonding processes involving these particles absorbs more energy than in the case of unmodified epoxy, and this advantage is being exploited in industrial applications. Extension of the adhesive layer model developed in this work to incorporate toughening particles would provide an excellent platform on which to investigate the toughening mechanisms interaction with a heterogeneous adherend. Incorporation of additive particles would involve the adoption of an appropriate placement algorithm for the inclusions, and determination of the material properties of the inclusions in the adhesive. Nanoindentation of rubber-toughened epoxies provides a novel approach to the determination of material properties of the constituent phases of the epoxy. Indentation of the epoxy and toughening particles within the epoxy separately would provide accurate in-situ stiffness properties of the individual phases. Push in tests on each of the phases, accompanied by in-situ SEM tests could be used to demonstrate whether debonding of particles from the epoxy or yielding of the toughening particle dominates the energy absorption mechanisms of such particles, which would provide input data for a micromechanical characterisation of the material.

The major disadvantage of such a micromodel is the significant increase in mesh volume as the RVE is extended to include the three dimensional nature of the toughening particles, requiring a major increase in computational effort.

7.3.4. Use of microscale models to predict interlaminar Damage

The focus of this work is on the study of damage at the interface between the adhesive and unidirectional composite regions in an adhesive-composite bond. Delamination of composite plies is an underdeveloped area of micromechanical analyses. Such
7.3 Recommended Future work

A case was investigated by Ye and Chen (2011) for single fibres at different orientations, however, the case has of randomly distributed fibres has not been investigated. Interactions of the stress fields in plies orientated at different angles opens the possibility for premature yielding of the matrix and interfacial damage to occur. Introducing a second composite ply to the adhesive layer model, as shown in Figure 7.2a would provide an early indication of the feasibility and accuracy of such an approach. Three dimensional stress gradients would be induced during loading, and RVEs would be required to be sufficiently large to capture the gradients in their entirety. This model would therefore suffer from high computational costs.

Figure 7.2a shows a preliminary model of the interface between plies lying perpendicularly to each other, under loading in the transverse direction. The complicated stress state within the RVE is clearly visible in Figure 7.2b, where the first layers of fibres at either side of the interface between the plies are isolated. The stress at the fibre surface, which is critical to the debonding damage process, shows clear localisations along the fibres. Areas of stress relaxation caused by the highlighted resin rich region and stress concentrations caused by fibres in close proximity to each other are marked. Such distributions can be detected using a random distribution of fibres, as created by the NNA. Such stress distributions will affect the damage initiation point and therefore the interlaminar strength of a composite.

Development of such a model would provide a new insight into the causes of delamination of composite materials and damage progression during delamination. In addition to this, the model could be adapted to investigate the stress distributions related to different ply orientations within a laminate. Such a study could reveal whether stress distributions between certain ply orientations in a laminate cause more critical stress concentrations, and could aid in optimisation of ply orientations.
7.3 Recommended Future work

Figure 7.2.: Model of interply region within a laminate. (a) Shows stress in the through-thickness direction (b) examines the same stress contour over surface fibres, showing internal variations in the stress field.
7.3 Recommended Future work

7.3.5. In-situ SEM testing

DIC techniques, in tandem with in-situ SEM testing provide an opportunity to validate the crack path prediction of micromechanical models. A possible approach is to extract exact fibre positions at the start of an in-situ SEM video, and export these positions to a microscale FEA model. Validation of the crack path prediction of the microscale models can then be achieved through application of appropriate boundary conditions to the model, to correspond to the conditions applied in the test. Direct comparison of the experimental crack path and the path predicted at the microscale could be made, and the method could be used to further refine and tune the parameters of the micromodel. This area is particularly suited to the embedded cell approach, as a microstructural cell, inserted in a larger model consisting of homogenised properties could be used to improve the efficiency of a large model. Such an approach was as used by González and Llorca (2006), without support from in-situ SEM analysis. This approach could be employed in the case of specifically designed geometries possibly using a crack initiator in the form of a crack starter film or notch in order to improve predictions for the location of crack initiation.

In an extension of the technique above, tracking fibre positions at each frame in an in-situ SEM video would provide an approximate strain distribution at the microscale. The slow scan speed of the SEM during testing, and issues with maintaining constant brightness of the scans of the SEM during the test, make accurate DIC analysis of video results of the strain field between fibres extremely difficult. It is possible to view fibres “edge-on” through the SEM, as shown in Figure 7.3, and the good contrast between the fibres and the matrix would allow consistent markers for tracking with DIC. An approximation of the microscale strain distribution during loading and failure of a laminate would be an extremely novel application of the in-situ SEM analysis techniques, and could give a new insight into the crack initiation and propagation process at the microscale.
7.3.6. Investigation of joint test geometries

The failure analysis in this work concentrates on one specific combination of materials (composite: HTA6376; adhesive: 3M 9323), and data from tests on different combinations of materials should be gathered. The damage mechanisms produced through failure of different grades of adhesive (e.g. differing stiffness, yield stress) should be investigated. Composite layups can be varied to investigate if damage behaviour in the composite plies is dependent on the ply orientations.

The surface of the composite can be altered through the use of peel ply. Applied to the surface of the composite during the layup, the peel ply leaves a regular textured imprint on the laminate surface once it is peeled off after curing. Gude et al. (2011) showed that a peel ply surface on a laminate is an effective method for increasing the wetted bonding area over a flat surface. Investigation of changes to the microscale damage processes in laminates with a peel ply surface relative to laminates without peel ply is an ideal application of the in-situ SEM testing set up.

A range of different tests for examining failure of adhesive bonds are available, of which Ferraris et al. (2010) provide a good summary. Chief among the Mode I and Mode II tests are the DCB and ENF tests, respectively, which are the most common tests for separation of Mode I and Mode II conditions. Design of test fixtures to carry out such tests using in-situ SEM analysis would provide a comparison with the three-point bend and compression shear tests carried out here. Scatter in the experimental data here is an issue with testing of miniature bonded specimens. Testing on a range
of test geometries should be carried out to determine the configuration which causes least scatter in the results. This data can be used to determine a standard microscale test geometry for adhesively bonded joints can be determined and would be important as microscale testing increases in popularity.
Bibliography

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Chen and Han (1988), *Plasticity for Structural Engineers*, Springer-Verlag.


Keane, A. (2009), Multi-scale Modelling of Transverse and Shear Deformation in Unidirectional Composite Laminates, Unpublished thesis (M.Eng), University of Limerick.


A. Code Files

A.1. Periodic Constraint Equations

Three dimensional periodic constraints have been developed for the RVEs in this thesis. Python and MATLAB scripts (not included here) have been written to create nodesets containing the surface nodes of the RVEs in this work. The constraint equations follow in section A.1.1. Figure A.1 shows a generic cuboid shape, representative of RVEs in this work, on which the nodeset names are marked. Data is provided here for the

![Figure A.1: Nodesets, as applied to the composite ply RVE and the adhesive layer RVE](image)
A.1.1. RVE model

The following set of 36 equations are added to the assembly data of an RVE input file to maintains periodicity in the three-dimensional RVE model.

```
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SOUTH,1 , 1
N4,1 , 1
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N4,2 , 1
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SOUTH,3 , 1
N4,3 , 1
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3
FRONT,1 , −1
BACK,1 , 1
N5,1 , 1
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3
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BACK,2 , 1
N5,2 , 1
*EQUATION
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### A.1 Periodic Constraint Equations

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A.2. Python Scripts

The following section contains the necessary python scripts to produce homogenised results from the composite ply model.

A.2.1. Composite ply model: transverse tension

The following python script is used to homogenise results from the composite ply model, under transverse tensile conditions.

```python
from abaqus import *
from abaqusConstants import *
import visualization
import odbAccess
import os

#This section changes the working directory of the current ABAQUS session
currentViewport = session.viewports[session.currentViewportName]
odbFile = currentViewport.displayedObject
odbFileNameFull = odbFile.path
odbFileName = os.path.split(odbFileNameFull)[1]
workDir = os.path.split(odbFileNameFull)[0]
os.chdir(workDir)
odbPath=session.viewports[session.currentViewportName].displayedObject.path
myOdb=session.openOdb(odbFileName)
currentViewport.setValues(displayedObject=myOdb)

#This section assigns the Nodesets into appropriate containers
N8=myOdb.rootAssembly.nodeSets['N8'].nodes[-1][0].label-1
N7=myOdb.rootAssembly.nodeSets['N7'].nodes[-1][0].label-1
N6=myOdb.rootAssembly.nodeSets['N6'].nodes[-1][0].label-1
```
20 N5=myOdb.rootAssembly.nodeSets[ 'N5' ].nodes[−1][0].label−1
21 N4=myOdb.rootAssembly.nodeSets[ 'N4' ].nodes[−1][0].label−1
22 N3=myOdb.rootAssembly.nodeSets[ 'N3' ].nodes[−1][0].label−1
23 N2=myOdb.rootAssembly.nodeSets[ 'N2' ].nodes[−1][0].label−1
24 N1=myOdb.rootAssembly.nodeSets[ 'N1' ].nodes[−1][0].label−1
25 #This section saves the coordinates of the nodesets
26 N1loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N1].coordinates
27 N2loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N2].coordinates
28 N3loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N3].coordinates
29 N4loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N4].coordinates
30 N5loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N5].coordinates
31 N6loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N6].coordinates
32 N7loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N7].coordinates
33 N8loc=myOdb.rootAssembly.instances[ 'RVE_1' ].nodes[N8].coordinates
34 #This section calculates the areas of the faces of the RVE
35 ATOP=(N3loc[0]−N4loc[0])*(N8loc[2]−N4loc[2])
36 AFRNT=(N3loc[0]−N4loc[0])*(N8loc[1]−N5loc[1])
38 #This section extracts the appropriate field outputs for each
39 nodeset
40 RFU_N1=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL,
41 variable=(( 'RF',
42 NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'),(COMPONENT, 'RF3'
43 ), )), ('U', NODAL, ((
44 COMPONENT, 'U1'), (COMPONENT, 'U2'),(COMPONENT, 'U3'), )), ),
45 nodeSets=('N1', ))
46 RFU_N2=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL,
47 variable=(( 'RF',
48 NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'),(COMPONENT, 'RF3'
49 ), )), ('U', NODAL, ((
50 COMPONENT, 'U1'), (COMPONENT, 'U2'),(COMPONENT, 'U3'), )), ),
51 nodeSets=('N2', ))
A.2 Python Scripts

```
NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ), ('U', NODAL, ((COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'),)), ),
nodeSets=('N2', ))

RFU_N3=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL, variable=((RF,
    NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ), ('U', NODAL, ((COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'),)), ),
    nodeSets=('N3', ))

RFU_N4=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL, variable=((RF,
    NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ), ('U', NODAL, ((COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'),)), ),
    nodeSets=('N4', ))

RFU_N5=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL, variable=((RF,
    NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ), ('U', NODAL, ((COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'),)), ),
    nodeSets=('N5', ))

RFU_N6=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL, variable=((RF,
    NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ), ('U', NODAL, ((COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'),)), ),
    nodeSets=('N6', ))
```

RFU_N7=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL,
    variable=(( 'RF',
        NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ))), ('U', NODAL, ((
        COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'), ))), ),
    nodeSets=('N7', ))
RFU_N8=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL,
    variable=(( 'RF',
        NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'), (COMPONENT, 'RF3'), ))), ('U', NODAL, ((
        COMPONENT, 'U1'), (COMPONENT, 'U2'), (COMPONENT, 'U3'), ))), ),
    nodeSets=('N8', ))
framelen=len(myOdb.steps['Apply_Displacement'].frames)
S22=[[0] for _ in [0]*framelen]
e22=[[0] for _ in [0]*framelen]
#This section writes the homogenised stress and strain values to a
#file called S22.dat
S22file=open('S22.dat','w')
for i in range(0,framelen):
    S22[i]=(RFU_N3[1][i][1]+RFU_N4[1][i][1]+RFU_N7[1][i][1]+RFU_N8[1][i][1])/ATOP
    e22[i]=(RFU_N8[4][i][1])/(N8loc[1]-N5loc[1])
    S22file.write( '%f\t%f\t\n' % (e22[i],S22[i]))
S22file.close()
A.2.2. Composite ply model: transverse shear

The following python script is used to homogenise results from the composite ply model, under transverse shear conditions. The first 62 lines of this script are identical to that of the script in section A.2.1, to which the following lines are added.

```python
S23 =[[0] for _ in [0]*framelen]
e23 =[[0] for _ in [0]*framelen]
S23_file=open ( 'S23.dat', 'w')
for i in range(0,framelen):
    S23[i]=(((RFU_N2[1][i][1]+RFU_N3[1][i][1]+RFU_N6[1][i][1]+RFU_N7[1][i][1]) /ASIDE)+(RFU_N3[0][i][1]+RFU_N4[0][i][1]+RFU_N7[0][i][1]+RFU_N8[0][i][1]) /ATOP) /2
    e23[i]=((RFU_N7[4][i][1]/(N7loc[0]−N8loc[0]))+(RFU_N7[3][i][1]/(N7loc[1]−N6loc[1])))
S23_file.write( '%f	%f\n' % (e23[i], S23[i]))
S23_file.close()
```

A.2.3. Composite ply model: in-plane shear

The following truncated python script is used to homogenise results from the composite ply model, under in-plane shear conditions. The first 62 lines of this script are identical to that of the script in section A.2.1, to which the following lines are added.

```python
RFU_TOP=session.xyDataListFromField (odb=myOdb, outputPosition=NODAL, variable=((‘RF’,)
    NODAL, ((COMPONENT, ‘RF1’), (COMPONENT, ‘RF2’), (COMPONENT, ‘RF3’), ),) , (‘U’, NODAL, ((
    COMPONENT, ‘U1’), (COMPONENT, ‘U2’), (COMPONENT, ‘U3’), ), ),
    nodeSets=('TOP', ))
framelen=len (myOdb.steps[‘Apply Displacement’].frames)
```
A.2 Python Scripts

```python
S12=[[0] for _ in [0]*framelen]
e12=[[0] for _ in [0]*framelen]
S12file=open('S12.dat','w')
for i in range(0,framelen):
    S12[i] = ((RFU_N5[1][i][1]+RFU_N6[1][i][1]+RFU_N7[1][i][1]+RFU_N8[1][i][1])/AFRNT)+((RFU_N3[2][i][1]+RFU_N4[2][i][1]+RFU_N7[2][i][1]+RFU_N8[2][i][1])/ATOP))/2
e12[i] = ((RFU_N8[4][i][1]/(N8loc[2]-N4loc[2]))+(RFU_N4[5][i][1]/(N8loc[1]-N5loc[1])))
S12file.write('%f	%f
' % (e12[i],S12[i]))
S12file.close()
```

A.2.4. Adhesive layer model: transverse tension

The following truncated python script is used to homogenise results from the composite ply model, under transverse tensile conditions. The first 62 lines of this script are identical to that of the script in section A.2.1, to which the following lines are added.

```python
RFU_TOP=session.xyDataListFromField(odb=myOdb, outputPosition=NODAL, variable=(( 'RF',
    NODAL, ((COMPONENT, 'RF1'), (COMPONENT, 'RF2'),(COMPONENT, 'RF3'), )), ('U', NODAL, ((
    COMPONENT, 'U1'), (COMPONENT, 'U2'),(COMPONENT, 'U3'), )), ),
    nodeSets=('TOP', ))
framelen=len(myOdb.steps['Apply_Displacement'].frames)
S22=[[0] for _ in [0]*framelen]
e22=[[0] for _ in [0]*framelen]
S22file=open('S22.dat','w')
for i in range(0,framelen):
```
A.2 Python Scripts

```python
for p in range(len(RFU_TOP) / 6, len(RFU_TOP) / 6 * 2):
    S22[i] = S22[i] + [sum(RFU_TOP[p][i][1])]
    e22[i] = (RFU_N8[4][i][1]) / (N8loc[1] - (N5loc[1] + RFU_N5[4][i][1]))
    S22[i] = sum(S22[i] / ATOP)
    S22file.write('%f %t %f %t
' % (e22[i], S22[i]))
S22file.close()
```

A.2.5. Adhesive layer model: in-plane shear

The following truncated python script is used to homogenise results from the composite ply model, under in-plane shear conditions. The first 62 lines of this script are identical to that of the script in section A.2.1, to which the following lines are added.

```python
RFU_TOP = session.xyDataListFromField(odb=myOdb, outputPosition=NODAL,
    variable=(["RF",
        NODAL, ("COMPONENT", "RF1"), ("COMPONENT", "RF2"), ("COMPONENT", "RF3"),
        ),
        ("U", NODAL,
        ("COMPONENT", "U1"), ("COMPONENT", "U2"), ("COMPONENT", "U3"), )],
        nodeSets=(["TOP", ])
framenlen = len(myOdb.steps["Apply_Displacement"].frames)
S12 = [[0] for _ in [0]*framenlen]
e12 = [[0] for _ in [0]*framenlen]
S12file = open("S12.dat", "w")
for i in range(0, framenlen):
    S12[i] = (RFU_N5[1][i][1] + RFU_N6[1][i][1] + RFU_N7[1][i][1] + RFU_N8[1][i][1]) / AFRNT + (RFU_N3[2][i][1] + RFU_N4[2][i][1] + RFU_N7[2][i][1] + RFU_N8[2][i][1]) / ATOP) / 2
    e12[i] = (RFU_N8[4][i][1] / (N8loc[2] - N4loc[2]) + RFU_N4[5][i]...)
```

168
A.2 Python Scripts

\[
\frac{1}{(N8loc[1] - N5loc[1])}
\]

11 file.write('%f t %f t
\n' % (e12[i], S12[i]))

12

13 file.close()
B. Model Parameter Studies

This section contains a parameter study of model size and a mesh sensitivity study on the models used in this thesis.

B.1. Model Size Variation

An investigation was carried out on the size of the models, to ensure that the results are not dependent on the size of the model chosen.

Three models were generated, using different sized fibre distributions. The width of the RVEs was chosen as 30, 60 and 100 µm, and the height was maintained constant. The three different RVEs are shown in their damaged state in Figure B.1. Transverse tension was applied to the models, and the stress-strain results are presented in Figure B.2. It can be seen that the three different models produced stress-strain plots which are...
B.2 Mesh Sensitivity

Figure B.2.: Stress-strain plots of three different sized adhesive layer models almost superimposed on each other, indicating that the strength results presented in this thesis are independent of model size.

B.2. Mesh Sensitivity

A mesh sensitivity study was carried out on an adhesive layer RVE. A single random fibre distribution was used (Dimensions: 100 \( \mu \)m \( \times \) 40 \( \mu \)m \( \times \) 2 elements deep) and a 30 \( \mu \)m adhesive layer was used, shown in Figure B.3 was created. The geometry was meshed four times, creating four models of increasing mesh density. The range of
B.2 Mesh Sensitivity

number of elements was 85985 to 226414. The stress-strain plots were almost indistinguishable from each other, and the damage initiated at the same fibres in all cases. Figure B.4 shows a comparison plot of the resultant strength of the model in each case against the number of elements in the RVE. It can be seen that no change in strength was visible over the range of mesh sizes investigated.

Figure B.4: Relationship between the mesh size and resultant strength of the RVE.