

**Bio-inspired designs
for enhanced damage resistance
of composite structures:
Bouligand, Herringbone and
multi-tailored bone-inspired strategies**

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Abstract

This thesis presents three novel bio-inspired microstructural concepts, for point-by-point tailorable composite solutions, that exploit the ability to diffuse damage in a sub-critical state; these aim at enhancing the damage resistance and the energy dissipation capability of conventional composite structures. To this end, we investigated three bio-inspired concepts: *Bouligand*, *Herringbone-Bouligand* and *multi-tailored bone-inspired*.

The *Bouligand* concept, inspired by the impact-resistant periodic region of the mantis shrimp's dactyl club and consisting of a helicoidal lay-up with very small inter-ply (pitch) angles, was used to address the inherent low performance to through-the-thickness loading (static and dynamic) of CFRPs. We designed, manufactured, tested and analysed several ultra-thin-ply CFRP Bouligand microstructures, with the smallest pitch angle (2.5°) ever reached in the literature. Experimental studies, as well as detailed analytical and numerical modelling showed that tailored Bouligand microstructures achieved enhanced damage resistance via: smooth double-helicoidal evolution of damage, reduced delamination areas and sub-critical Bouligand cracks. Quasi-static indentation test showed that, compared to conventional CFRPs, tailored Bouligand microstructures achieved a simultaneous 92% increase in load-bearing capability, 74% delay in catastrophic failure and 97% increase in total dissipated energy.

The *Herringbone-Bouligand* concept, inspired by the high-damage-resistant features of the mantis shrimp's dactyl club impact region and impact surface, was used to improve further the damage resistance of Bouligand-inspired CFRPs. We designed, prototyped, tested and analysed the first high-performance Herringbone-Bouligand microstructure in the literature. We also devised the first prototyping procedure to manufacture point-by-point tailorable Herringbone-Bouligand CFRPs. Quasi-static indentation tests showed that, compared to standard Bouligand solutions, the Herringbone-Bouligand microstructure delays onset of delaminations, reduces the in-plane spreading of damage (71%), increases the energy dissipation capability (13%) and contains damage within the tailored Herringbone-Bouligand region.

The *multi-tailored bone-inspired* concept, based on the introduction of patterns of discontinuities in the form of laser-cuts across the load-carrying fibres of FRPs, was adopted to devise a versatile design strategy that can be used to locally-tailor different damage resistance

requirements at different locations within the same structure. Specifically, for a SRPP/CFPP hybrid composite, we demonstrate that patterns of discontinuities can be successfully tailored to create high-energy dissipation paths through which damage is stabilised and diffused at sub-critical levels. This results in a great increase (90%) in energy dissipation with respect to a non-engineered structure, i.e. a similar SRPP/CFPP hybrid structure without discontinuities. We demonstrate that discontinuities can be successfully tailored to enhance impact damage resistance, achieved with increased energy dissipation at sub-critical levels and delayed catastrophic failure.

Declaration of originality

The work presented hereafter is based on research carried out by the author at the Department of Aeronautics of Imperial College London and it is all the author's own work except where otherwise acknowledged. No part of the present work has been submitted elsewhere for another degree or qualification.

Lorenzo Mencattelli

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List of publications & dissemination

Journal publications

- L. Mencattelli and S. T. Pinho, “Herringbone-Bouligand CFRP structures: a new tailorable damage-tolerant solution for damage containment and reduced delaminations”, *Composites Science and Technology*, 108047, 2020. <https://doi.org/10.1016/j.compscitech.2020.108047>
- L. Mencattelli and S. T. Pinho, “Ultra-thin-ply CFRP Bouligand bio-inspired structures with enhanced load-bearing capacity, delayed catastrophic failure and high energy dissipation capability”, *Composites Part A: Applied Science and Manufacturing*, 105655, 2019. <https://doi.org/10.1016/j.compositesa.2019.105655>
- L. Mencattelli and S. T. Pinho, “Realising bio-inspired impact damage tolerant thin-ply CFRP Bouligand structures via promoting diffused sub-critical helicoidal damage”, *Composites Science and Technology*, vol. 182, 107684, 2019. <https://doi.org/10.1016/j.compscitech.2019.107684>
- L. Mencattelli, J. Tang, Y. Swolfs, L. Gorbatikh and S. T. Pinho, “Bio-inspired design for enhanced damage tolerance of self-reinforced polypropylene/carbon fibre polypropylene hybrid composites”, *Composites Part A: Applied Science and Manufacturing*, vol. 121, pp. 341-352, 2019. <https://doi.org/10.1016/j.compositesa.2019.03.028>
- J. Tang, Y. Swolfs, A. Aslani, L. Mencattelli, G. Bullegas, S. T. Pinho, S. V. Lomov and L. Gorbatikh, “Engineering tensile behavior of hybrid carbon fibre/self-reinforced polypropylene composites by bio-inspired fibre discontinuities”, *Composites Part B: En-*

gineering, vol. 178, 107502, 2019. <https://doi.org/10.1016/j.compositesb.2019.107502>

Conference publications

Conference papers - oral presentation

- L. Mencattelli, J. Tang, Y. Swolfs, L. Gorbatikh and S. T. Pinho, “Enhancing the damage tolerance of SRPP/CFPP hybrid composites via a bio-inspired design”, *ICCM22 - 22nd International Conference on Composite Materials*, Melbourne, Australia, 11th–16th August 2019.
- L. Mencattelli and S. T. Pinho, “Low velocity impact and compression after impact of thin-ply CFRP Bouligand structures”, *ECCM18 - 18th European Conference on Composite Materials*, Athens, Greece, 24th–28th June 2018.

Conference abstracts - oral presentation

- L. Mencattelli and S. T. Pinho, “Damage analysis of Bouligand and Herringbone/Bouligand thin-ply CFRP structures”, *Composites2019 - 7th ECCOMAS Thematic Conference on the Mechanical Response of Composites*, Girona, Spain, 18nd–20th September 2019.
- S. T. Pinho, A. Willems, B. Chen, L. Gigliotti, G. Bullegas, L. Gigliotti, F. Narducci, R. Hasa, R. S. Costa, J. A. Pascoe and L. Mencattelli, “Thinking creatively about microstructures and numerical methods for composites design”, *Composites2019 - 7th ECCOMAS Thematic Conference on the Mechanical Response of Composites*, Girona, Spain, 18nd–20th September 2019.
- L. Mencattelli and S. T. Pinho, “Realising impact damage tolerant Bouligand and Herringbone/Bouligand thin-ply CFRP structures”, *CompTest2019 - 9th International Conference on Composites Testing and Model Identification*, Lulea, Sweden, 27nd–29th May 2019.

- L. Mencattelli and S. T. Pinho, “Thin-ply CFRP Bouligand architectures for enhanced damage tolerance”, *New Generations of Composites - 15th European-Japanese Symposium on Composite Materials*, London, UK, 24nd–26th September 2017.

Posters

- L. Mencattelli and S. T. Pinho, “On the impact damage tolerance of thin-ply Bouligand structures”, *Composites Research Showcase - Imperial College London*, London, UK, 24th September 2018.

Workshops

- S. T. Pinho, G. Bullegas, F. Narducci, R. Hasa and L. Mencattelli, “Mechanics of composites”, “Novel composite microstructures for increased energy absorption”, *Workshop on crash behaviour of composites*, Gothenburg, Sweden, September 11, 2018.

Seminars

- L. Mencattelli, “Damage tolerant composite structures: challenges, design strategies and applications”, *FiBreMoD School&Conference*, Leuven, Belgium, 9th–12th December 2019, www.fibremodproject.eu/schoolandconference.

Outreach

- “The science of breaking stuff”, Interactive science exhibition at the Imperial Festival 2018, London, United kingdom, Apr 28-29, 2018
- “The science of breaking stuff”, Interactive science exhibition at the Imperial Festival 2019, London, United kingdom, Jun 28-30, 2019

Awards and Grants

- “CompTest2019 Best Student Presentation Award”
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bone/Bouligand thin-ply CFRP structures”

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Acronyms & abbreviations

FE, FEM	Finite element, Finite element method
CFRP	Carbon fibre reinforced plastic
FRTF	Fibre reinforced thermoplastic
FRP	Fibre reinforced plastic
GFRP	Glass fibre reinforced plastic
CFPP,C	Carbon fibre polypropylene, Carbon fibre polypropylene ply
SRPP, S	Self-reinforced polypropylene, Self-reinforced polypropylene ply
PP	Polypropylene
F	Film of polypropylene
QI	Quasi-isotropic
CP	Cross-ply
UD	Unidirectional
NS	Non symmetrical
S	Symmetrical
SO	Symmetric odd
DP	Double ply
VP	Variable pitch angle
QSI	Quasi static indentation
LVI	Low velocity impact
CAI	Compression after impact
DEN-T	Double edge notched tensile
CT	Compact tension
CNT	Centre notched tension
EWf	Essential work of fracture

SWF	Specific work of fracture
3PB	Three-point bending
DIC	Digital image correlation
SEM	Scanning electron microscope
CF	Critical failure
FP	Full penetration
DPSS	Diode pumped solid state
STD	Standard deviation
2D	Two-dimensional
3D	Three-dimensional

Notation & nomenclature

Fonts convention

a, A scalar variable

$\mathbf{A}$ matrix or tensor

Symbols

$\Delta\theta$ pitch angle

ψ twist angle in Bouligand cracks

α^* kink angle in Bouligand cracks

t_{ply} ply thickness

l ligament length

t laminate thickness

g, α, h parameters defining the pattern of staggered cuts tailored for impact samples

p p-value

m, n, o slopes of trendlines

R^2 R-squared

Γ span-to-thickness ratio of QSI and 3PB samples

A amplitude of Herringbone pattern

λ wavelength of Herringbone pattern

s_i^k stack- i of level- k

s final stack

r radius

ϕ	diameter
ϕ_f	fibre diameter
ρ	density
ρ_m	matrix density
ρ_f	fibre density
E	Young's modulus
E_{c1}	longitudinal tensile modulus of the composite
E_{c2}	transverse (in-plane) modulus of the composite
E_{c3}	transverse (out-of-plane) modulus of the composite
E_{1f}	longitudinal tensile modulus of the fibre
E_{2f}	longitudinal compressive modulus of the fibre
ν	Poisson's ratio
ν_{f12}	Poisson's ratios of the fibre
$\nu_{12,13,23}$	Poisson's ratios of the composite (ply-level)
G	shear modulus, energy release rate of twisting crack
G_0	energy release rate of a flat crack
G_m	shear modulus of the matrix
$G_{12,13,23}$	shear moduli of the composite (ply-level)
G_{f12}	shear modulus of the fibre
$\mathbf{B}$	in-plane/bending coupling matrix
ε	normal strain
ε_f^*	tensile failure strain of fibre
τ	shear stress
ply – i	i^{th} ply of a Bouligand layup
τ_{13}^i	intra-laminar shear stress (13 in material coordinates) on ply- i
τ_{23}^i	intra-laminar shear stress (23 in material coordinates) on ply- i
$\max \tau_{13}^i $	maximum value of intra-laminar shear stress 13 (material coordinates) on ply- i
$\max \tau_{23}^i $	maximum value of intra-laminar shear stress 23 (material coordinates) on ply- i
$\max \tau_{13}^{\Delta\theta} $	through-the-thickness distribution of the maximum values of intra-laminar shear stress (13 in material coordinates) of a laminate with pitch angle $\Delta\theta$

$\max \tau_{23}^{\Delta\theta} $	through-the-thickness distribution of the maximum values of intra-laminar shear stress (23 in material coordinates) of a laminate with pitch angle $\Delta\theta$
$k''_{I,II,III}$	local stress intensity factors at the front of a Bouligand crack in Mode I, II and III loading conditions
$K_{I,II,III}$	global stress intensity factors in Mode I, II and III loading conditions
C_{0°	refers to a 0° carbon fibre polypropylene ply
C_{90°	refers to a 90° carbon fibre polypropylene ply
δ	displacement at final failure
δ_{peak}	displacement at peak load
P_{peak}	peak load
W_{fracture}	total work of fracture
$W_{\text{essential}}$	essential work of fracture
W_{plastic}	plastic work of fracture
$W_{\text{plastic,PP}}$	plastic work of fracture due to failure of PP tapes
W_{pullout}	plastic work of fracture due to carbon fibre pull-outs
w_{fracture}	specific total work of fracture
$w_{\text{essential}}$	specific essential work of fracture
w_{plastic}	specific plastic work of fracture
β	shape factor of the plastic area
A_{ultimate}	area of the plastic region at final failure
A_{damage}	area of the plastic region at a generic instant during test
N_{cuts}	number of laser-cuts involved in the failure process

Chapter 1

Introduction

1.1 Background and Motivation

Laminated composites are widely used in a variety of industrial applications. Traditionally used in aeronautical and aerospace structures, composite materials have spread to several mass production markets, ranging from automotive and civil engineering to home appliance and leisure. Composite materials offer higher specific strength and stiffness than other conventional materials. The possibility of creating lightweight structures capable of withstanding elevated loads has widened the utilisation of Fibre Reinforced Plastics FRPs (specifically Carbon Fibre Reinforced Plastics CFRPs) in load-bearing applications. For instance, in the latest aircraft models, CFRPs have reached values of up to 50% of the entire airframe [1].

In mass production markets, the hybridisation of highly ductile polymers with high-stiffness and high-strength reinforcements, such as carbon fibres, has allowed the use of low-stiffness plastics in load-bearing applications. Some carbon fibre reinforced thermoplastic composites are in fact particularly suitable to mass productions being characterised by high recyclability, low production costs, excellent formability and high performances.

Although the aforementioned properties justify the efficiency of composite solutions with respect to classical ones (i.e. metallic and pure polymer blends), the inherent brittleness of high-performance reinforcements, such as carbon fibres, typically leads to damage localisation eventually resulting in abrupt and catastrophic failures with limited damage diffusion, relatively

poor energy dissipation capability and thus, relatively poor damage tolerance and damage resistance. The relatively poor damage tolerance and damage resistance, in addition to a lack of knowledge related to the mechanisms of failure of composite structures, often lead to over-designing the majority of composite components.

Historically, ‘damage resistance’ refers to the ability of a structure to resist damage initiation and/or growth [2]. This definition does not make any distinction between different damage types and the way these propagate in the structure. In this thesis, we will refer to ‘damage resistance’ as the ability of a microstructure to resist only those damage types that lead to the catastrophic failure of the structure (e.g. localised delamination and fibre failure) and/or to the reduction of certain metrics of mechanical performance (such as load-bearing capability, stiffness, energy dissipation etc.).

The sensitivity of composites to impacts has also been one of the historical limitations to the widespread use of laminated solutions. FRPs can absorb energy under dynamic loading only by elastic deformation and activation of several mechanisms of failure. In particular, their high heterogeneity and anisotropy allows for three main modes of failure to activate and interact between them under transverse loading:

1. *matrix mode*: cracking parallel to the fibres;
2. *delamination mode*: produced by interlaminar stresses in combination with the low interlaminar strength of the interfaces between layers;
3. *fibre mode*: fibre break in tension and micro-buckling/kinking in compression.

Depending on the specific function of the component, priority should be given to limit the most relevant failure modes from the list above. For instance, if the material must sustain loads after impact, which is the case in many structural applications, limiting fibre breaks and delaminations is imperative in order to maintain high values of residual strengths.

Researchers have developed many different strategies to increase the damage tolerance and damage resistance of FRPs. From these, biological materials and natural microstructures have been recently considered as an effective resource to better understand and improve human-made composite solutions. Not surprisingly, features such as discontinuities, hierarchy, fractality and

helicoidality are extremely recurrent in many bio-materials and are possibly responsible for some of their outstanding damage-resistance properties.

Among the recurrent features in naturally-occurring layered microstructures which have developed to withstand impacts and, in general, through-the-thickness loads, helicoidal architectures stand out. Helicoidal (also known as Bouligand [3]) fibre arrangements, consist of periodic helicoidal stack-ups of fibrous layers with small inter-ply (pitch) angles. Under impact, Bouligand architectures promote the nesting of sub-critical helicoidal matrix cracks, thus preventing fibres from catastrophic failure [4]. Recently, a study on the biological microstructure of the mantis shrimp's dactyl club has shown that the combination of helicoidal arrangements with non-planar (wavy) ply-interfaces (Herringbone-Bouligand microstructures) can potentially provide a superior damage diffusion capability compared to classical Bouligand microstructures, thus resulting in a formidable enhancement in resistance to through-the-thickness loads [5]. Therefore, understanding how these features promote the toughening mechanisms observed in nature and, hence, mimicking them in human-made composite solutions offers a great opportunity to overcome one the most relevant limitations of composite materials: their sensitivity to through-the-thickness loads.

The presence of staggered arrangements of discontinuous stiff inclusions embedded in a soft matrix is another recurrent feature in several biomaterials, such as bone and nacre-like microstructures. In biomaterials, this versatile feature is tailored to achieve a wide variety of damage-resistance properties not typically encountered in synthetic composite materials. These include the ability to diffuse sub-critical damage, the high energy dissipation capability, the high resistance to through-the-thickness loads and the ability to stabilise failure [6–9]. Therefore, the development of a design strategy based on the use of staggered discontinuities could potentially be used to tailor, point-by-point, the damage resistance of composite structures by mimicking the same toughening mechanisms observed in nature. Such multi-tailored design concept could greatly expand the design space of composite structures and thus, achieve significant industrial impact.

Although Bouligand, Herringbone-Bouligand and bone-like biological microstructures present distinctive traits and distinctive toughening mechanisms, they all share the ability of diffusing damage at a sub-critical level. This delays the occurrence of catastrophic failure and controls the failure process through stable highly-dissipative crack paths, resulting in an

outstanding damage resistance. Therefore, mimicking the different features and toughening mechanisms of the bespoke microstructures holds the potential to show that the capability for enhanced damage diffusion at sub-critical level can be exploited to enhance the damage resistance and the mechanical performance of various carbon fibre composite materials under various loading conditions.

In this context, this thesis therefore focuses on investigating and exploiting, with various carbon fibre composites, the ability to diffuse damage at a sub-critical state for the bio-inspired concepts of Bouligand, Herringbone-Bouligand, as well as bone-inspired tailored discontinuities.

1.2 Aims and Objectives

Based on the bio-inspired opportunities identified in § 1.1, in this PhD, three bio-inspired strategies will be investigated to improve the damage resistance of composite structures reinforced with carbon fibres, with the aims of:

- (i) realising the potential of Bouligand structures with CFRP via a deep understanding of their failure mechanisms;
- (ii) realising the potential of Herringbone-Bouligand architectures with CFRPs via combining helicoidally-layered structures with interlocking ply-interfaces; and
- (iii) devising a multi-tailored composite design strategy based on tailored patterns of discontinuities to enhance multiple damage resistance properties of a Self-Reinforced Polypropylene/Carbon Fibre Polypropylene (SRPP/CFPP) hybrid structure.

Aim (i) will be achieved with the following more detailed objectives:

1. to develop a manufacturing process for Bouligand composites that is effective, accurate and reproducible, allowing to explore Bouligand microstructures with features such as pitch angle and Bouligand repetitions never achieved before;
2. to plan experimental campaigns in order to verify the mechanical performances of the devised microstructure under through-the-thickness loads;

3. to develop analytical and numerical tools for investigating the response of Bouligand layups to understand how the characteristic features of Bouligand microstructures can lead to the toughening mechanisms experimentally observed; and
4. to conduct parametric studies with the developed analytical and numerical tools in order to assess the effect of changing the features of the microstructure on the mechanical and damage resistance properties of CFRP Bouligand composites and, consequently, to design tailored Bouligand configurations with improved damage resistance.

Aim (ii) will be achieved with the following more detailed objectives:

1. to develop the first manufacturing process for Herringbone-Bouligand CFRP composites, allowing to explore the first high-performance Herringbone-Bouligand microstructure;
2. to demonstrate that Herringbone-Bouligand architectures can be tailored point-by-point in the structure;
3. to plan experimental campaigns in order to verify the mechanical performances of the devised microstructure under through-the-thickness loads; and
4. to investigate the failure mechanisms and damage resistance performances of Herringbone-Bouligand CFRP structures, demonstrating their superior damage resistance and structural integrity with respect to ‘classical’ Bouligand architectures.

Aim (iii) will be achieved with the following more detailed objectives:

1. to define the manufacturing technologies suitable to design staggered patterns of discontinuities in a SRPP/CFPP hybrid composite;
2. to define a tailoring strategy and identify the microstructural parameters driving the design of the patterns of discontinuities;
3. to plan experimental campaigns in order to verify the mechanical performances of the devised microstructure under notched tension and impact loading;
4. to demonstrate how patterns of discontinuities can potentially be tailored to meet different damage resistance requirements at different points of a component, using the same technique to engineer the microstructure and the same material system; and

5. to use analytical tools and experimental observations to understand how the activation of specific toughening mechanisms can lead to the achieved damage resistance enhancement.

1.3 Structure of the thesis

In this PhD thesis, the aims and the objectives expressed in § 1.2 will be addressed consecutively. Considering that the microstructural concepts adopted in this thesis present distinctive traits, distinctive toughening mechanisms and aim at enhancing different aspects of the damage resistance of FRPs, the literature review relevant to each microstructural concept will be presented contextually within the respective chapter. The thesis is structured as follows.

Chapter 2 The impact damage resistance of ultra-thin-ply CFRP Bouligand structures: diffused sub-critical helicoidal damage

In this chapter, we present the design, manufacturing and testing of several bio-inspired ultra-thin-ply CFRP laminates, mimicking the helicoidal architecture of the mantis shrimp's dactyl club periodic region, with the smallest inter-ply (pitch) angle in the literature (2.5°), thus better mimicking the actual micro-structure of the dactyl club. We conducted Low Velocity Impact (LVI) tests on a wide range of pitch angles (2.5° , 5° , 10° , 20° , 45°), thus demonstrating that decreasing the pitch angle leads to a progressively smoother double helicoidal evolution of damage, reduces delamination areas, diffuses sub-critical damage, and enhances damage resistance. We then conducted Compression After Impact (CAI) tests, thereby demonstrating that the residual strength and failure strain are preserved as the pitch angle is reduced, even though there is a steep decrease in the proportion of 0° -plies (plies aligned with the loading direction) as the pitch angle decreases. Via detailed numerical and analytical modelling, we developed and proposed an explanation for why reducing the pitch angle leads to a progressively better defined through-the-thickness helicoidal distribution of damage. Additionally, we analysed how the pitch angle and the ply thickness affect the resistance of Bouligand CFRP composites to growing twisting Bouligand-like cracks forming at the back face of the laminate (LVI tests) due to the tensile stresses originating from bending.

Chapter 3 Towards ultra-damage-resistant thin-ply CFRP Bouligand structures: progressive failure analyses

In this chapter, based on the knowledge developed in [Chapter 2](#), we present an original study to demonstrate that the inherent low performance of thin-ply composites to transverse loading can be greatly improved via a tailored bio-mimetic Bouligand design. To this end, we performed several in-situ damage analyses on ultra-thin-ply Bouligand CFRP configurations similar to the ones developed in [Chapter 2](#). Quasi-Static Indentation (QSI) full-penetration tests allowed for the first time to explore the full-penetration behaviour (and the full spectrum of the energy dissipation capability) of ultra-thin-ply CFRP Bouligand structures. We revealed how the sequence of activation of different failure mechanisms depends on the pitch angle and how this affects the performances to out-of-plane loading of ultra-thin-ply Bouligand structures at the sub-critical (sub-peak), catastrophic (peak) and penetration (post-peak) stages of failure. Three-Point Bending (3PB) tests results, conducted under a Scanning Electron Microscope (SEM), along with a detailed modelling of the growth of Bouligand matrix cracks presented in [Chapter 2](#), revealed that twisting Bouligand cracks, crack branching, cracks dispersion and stable ply-fragmentation have allowed small pitch angles laminates to dissipate energy at sub-critical levels, preserving most of their load-bearing capability and delaying catastrophic failure. Overall, we demonstrate the extraordinary capability of ultra-thin-ply CFRP Bouligand structures in dissipating energy at sub-critical levels leading to a significant simultaneous increase in (i) maximum load-bearing capability (92%), (ii) delay in catastrophic failure (74%) and (iii) energy dissipation (97%).

Chapter 4 Herringbone-Bouligand CFRP structures: a new tailorable damage-resistant solution

In this chapter, we present the design, prototyping, testing and analysis of the first high-performance Herringbone-Bouligand microstructure, with CFRP, inspired by the full microstructure of the high-impact-resistant mantis shrimp's dactyl club. To this end, we devised the first prototyping procedure to manufacture point-by-point tailorable Herringbone-Bouligand CFRP microstructures; this was based on the micro-moulding of uncured CFRP prepreg, and led to mimicking features of the club microstructure never achieved before with CFRPs. We investigated the damage resistance of the pro-

totyped Herringbone-Bouligand CFRP laminates, compared against ‘classical’ Bouligand and CFRP laminates, using QSI full-penetration tests. Our test results show that the Herringbone-Bouligand microstructure resulted in delayed onset of delaminations, reduced in-plane spreading of damage, increased energy dissipation capability, and in the containment of damage within the tailored Herringbone-Bouligand region. We conclude that Herringbone-Bouligand CFRP microstructures offer an excellent tailorable damage-resistant solution with great potential for composite applications where resistance to through-the-thickness loads is paramount.

Chapter 5 An original multi-tailored bone-inspired design strategy: enhancing the damage resistance of SRPP/CFPP hybrid structures

In this chapter, we investigate the toughness of an inter-layer SRPP/CFPP cross-ply hybrid composite and devise strategies to improve two aspects of its damage resistance: (i) energy dissipation capability and (ii) enhancing the impact damage resistance. To this end, we locally engineered the microstructure of the composite via introducing discontinuities in the form of laser-cuts across the fibres of the CFPP plies. We tailored two patterns of laser-cuts to meet each specific requirement. We designed baseline specimens and specimens with engineered microstructures to conduct Double Edge Notched Tensile (DEN-T) and low velocity drop-weight penetration impact tests. We used the Essential Work of Fracture (EWF) method to provide a measure of fracture toughness and energy dissipation performances of the hybrid composites. The DEN-T tests show that hybridising polypropylene tapes with continuous carbon fibres results in a tough material — 213 kJ/m². Engineering the microstructure of DEN-T samples has successfully delocalised strain concentrations and extensively diffused damage. The activation of dissipative mechanisms, such as pull-outs of carbon fibre bundles, has led to a great increase in energy dissipation capability of the structure — 90% increase in the slope of the Specific Work of Fracture (SWF) with respect to the baseline. Engineering the microstructure of impact samples has led to enhanced impact resistance, with a more ductile impact response, increased energy dissipation at a sub-critical level and delayed critical failure.

Chapter 6 Conclusions

In this chapter, we report the key message and overall conclusions of this PhD thesis; we highlight the main achievements and the original contributions. Additionally, we provide insights and recommendations for future work.

Chapter 2

The impact damage resistance of ultra-thin-ply CFRP Bouligand structures: diffused sub-critical helicoidal damage

2.1 Introduction

Non-standard lamination sequences inspired by naturally-occurring impact-resistant micro-structures, such as Bouligand [3] structures (Fig. 2.1a), have been investigated in the past years in order to improve the resistance of Fibre Reinforced Plastic FRP (and specifically Carbon Fibre Reinforced Plastic CFRP) laminates to out-of-plane loads [10–21]. Significant progress has been made in our understanding of Bouligand structures [10–21]; however, we have not yet been able to fully exploit this type of highly damage resistant micro-structure in engineering solutions.

Bouligand-featured structures can be found in a variety of animals, including the mantis shrimp (*Odontodactylus scyllarus* in Fig. 2.1b [22]). The so called periodic region of the mantis shrimp's dactyl club micro-structure consists of several repetitions of the Bouligand unit,

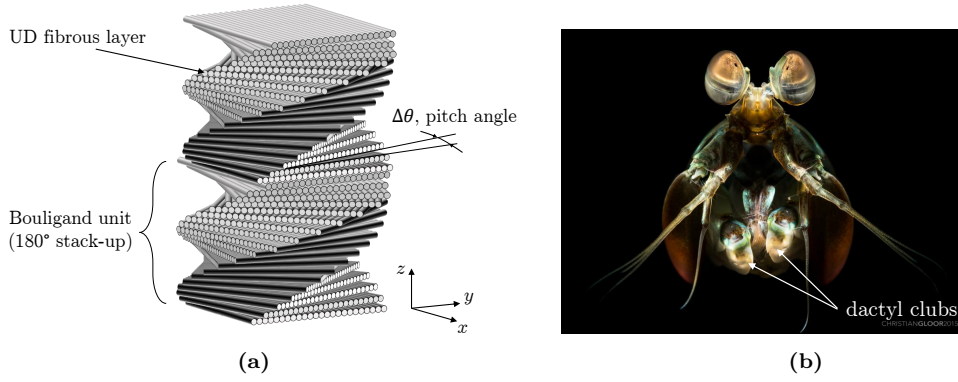


Figure 2.1: (a) Schematic of Bouligand micro-structure; (b) photograph of the mantis shrimp (*Odontodactylus scyllarus*) [22].

stacked up along the thickness direction [4]. Each Bouligand unit (Figure 2.1a) contains a helicoidal layup with very small inter-ply (pitch) angles $\Delta\theta$ (from 6.2° to 1.6° [12]) for a total rotation of 180° inside each unit.

By analysing the coronal cross section of an impacted club (Figure 2.2), we observe an interesting mechanism of failure under impact: several cracks nest inside each unit, growing in a helicoidal pattern, remaining parallel to the local fibre orientation mainly via cracking matrix, leaving fibres mostly undamaged [4]. The accumulation of the highly diffused sub-critical damage (visible in Figure 2.2) allows biological Bouligand structures to withstand several impacts while retaining more of their undamaged mechanical properties.

The attempts to exploit the enhanced impact damage tolerance and damage resistance of biological Bouligand arrangements in CFRP and Glass Fibre Reinforced Plastic (GFRP) structures led to significant progress [10–19]. In some studies (see Table 2.1), FRP Bouligand laminates outperformed conventional laminates under out of plane loadings [10–12, 14, 15].

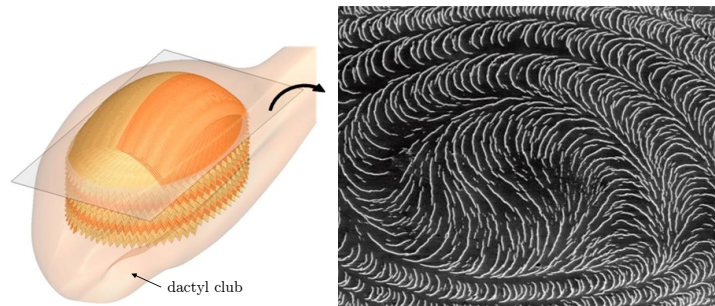


Figure 2.2: SEM micrograph of a coronal cross section of the dactyl club showing cracks (in white) nesting in the Bouligand units of the periodic region of the club (adapted from Weaver et al. [4]) and Suksangpanya et al. [20] .

Table 2.1: Experimental tests conducted in previous studies on Bouligand FRP composites [10–19].

Author(s)	Baseline material	Configurations and pitch angles $\Delta\theta$	Sample thickness [mm]	Periodicity	Test(s)
Apichattrabrut et al. [10]	CFRP (NCT304-1)	UD	6.35 (40 layers)	2	Tension, Bending, Ballistic
		$\pm 45^\circ$			
		10° (S)			
Cheng et al. [11]	GFRP (DA409U/S2-glass)	QI	4.5-5 (24 layers)	2	Short and long beam bending
		7.8° (S & NS)			
		16.4° (S)			
Grunenfelder et al. [12]	CFRP (CYCOMS320-1)	UD	6.5 (48 layers)	2	High energy Impact (100J), CAI
		QI			
		7.8° (S)			
		16.3° (S)		4	
		25.7° (S)		6	
Shang et al. [13]	CFRP (T700/2510)	CP	0.88-1.52 (11 & 19 layers)	1	Quasi-static indentation
		10° (NS)			
		18° (NS)		1	
Pinto et al. [14]	CFRP (T800/M21)	QI	26 layers	2	Tension, LVI (30 J), CAI
		15° (S)			
		15° (NS)		2	
Ginzburg et al. [15]	CFRP (T800/M21)	CP	4 (32 layers)	1	LVI (40-80 J), CAI
		QI			
		6° (NS)			
		12° (NS)		2	
		12° (S)		2	
Liu et al. [16]	CFRP (T700/2510)	CP	1.52 (19 layers)	1	Quasi-static indentation
		10° (NS)			
Liu et al. [17]	CFRP (T700/2510)	CP	1.52 (19 layers)	1	Quasi-static indentation
		10° (NS)		2	
		20° (NS)		3	
		30° (NS)		4	
		40° (NS)		1	
		CP	2.96 (37 layers)	1	
		5° (NS)		2	
		10° (NS)		4	
		20° (NS)		1	
		CP	5.84 (73 layers)	1	
		2.5° (NS)		2	
		5° (NS)		4	
		10° (NS)		8	
		20° (NS)		1	
		5° (NS,DP)		2	
		10° (NS,DP)			
Liu et al. [18]	CFRP (T700/2510)	$(5^\circ, 10^\circ)$ (VP,NS)	5.84 (73 layers)	1	Quasi-static indentation
		$(10^\circ, 5^\circ)$ (VP,NS)		1	
		$(10^\circ, 20^\circ)$ (VP,NS)		1	
Abir et al. [19]	CFRP (T700/2510)	QI (NS)	2.96 (37 layers)	1	Ballistic
		CP (S)			
		5° (NS)			
UD - Unidirectional	CP - Cross ply	QI - Quasi-isotropic	NS - Non Symmetrical		
S - Symmetrical	DP - Double Ply	VP - Variable Pitch angle			

In other studies, [13, 16–18] (Table 2.1), CFRP Bouligand laminates achieved enhanced load bearing capability, albeit with a more brittle response and different failure mechanisms than

those observed in the biological Bouligand micro-structures. The latter has been also observed in other studies on CFRP Bouligand laminates [12, 15]. Finally, in the study conducted by Ginzburg et al. [15] (Table 2.1), the residual compressive properties due to high impact energies (80 J) of CFRP Bouligand microstructures was found to be better than the corresponding quasi-isotropic; however, the impact performances at lower impact energies (40 J) of CFRP Bouligand laminates fell behind those of conventional configurations.

One of the challenges that needs overcoming in order to achieve enhanced impact damage resistance in Bouligand CFRP structures is achieving small pitch angles $\Delta\theta$ and large number of Bouligand unit repetitions through the thickness of the laminate, given the current standard pre-preg thicknesses (~ 0.1 mm). For instance, the smallest pitch angle $\Delta\theta$ published to date in the literature on CFRP Bouligand structures in a symmetrical configuration is 7.8° [11, 12] (Table 2.1), while in natural Bouligand micro-structures pitch angles $\Delta\theta$ of 1.6° have been observed [12]). Small pitch angles ($\Delta\theta < 7.8^\circ$) have been so far manufactured only in non-symmetrical configurations [13, 15–19] possibly resulting in a detrimental effect on the performance of the laminate due to residual thermal stresses [14]. Moreover, while biological Bouligand structures contain many repetitions of the Bouligand unit with small pitch angles $\Delta\theta$, engineered Bouligand structures with small pitch angles ($\Delta\theta < 10^\circ$) in the literature have never exceeded two repetitions [10–19]).

In view of this, we devised an original experimental and modelling study to investigate ultra-thin-ply Bouligand structures under Low Velocity Impact (LVI) and Compression After Impact (CAI).

This chapter contains the following original contributions:

- (i) ultra-thin-ply CFRP pre-preg with a thickness down to $24\text{ }\mu\text{m}$ are used to manufacture Bouligand CFRP laminates;
- (ii) CFRP laminates are manufactured with very small pitch angles $\Delta\theta = 2.5^\circ$ for the first time in a symmetrical and balanced configuration, while in the literature such a small pitch angle has been explored only in non-symmetrical laminates. The small $\Delta\theta = 2.5^\circ$ pitch angle is in the range of the pitch angles observed in naturally-occurring Bouligand structures;

- (iii) a wide range of pitch angles ($\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45°) are explored and compared, where all laminates are symmetric, with the same nominal thickness and loaded under the same loading conditions;
- (iv) trends are drawn on the effect of the pitch angle $\Delta\theta$ on the mechanical and damage properties of ultra-thin-ply CFRP Bouligand structures under LVI and CAI tests;
- (v) it is shown that is possible to achieve enhanced impact damage resistance with ultra-thin-ply Bouligand CFRP structures and small pitch angles $\Delta\theta = 2.5^\circ$;
- (vi) an understanding is developed, via a detailed analysis that uses numerically-obtained stress fields, on the role of the pitch angle $\Delta\theta$ on the LVI performances of ultra-thin-ply CFRP Bouligand structures; and
- (vii) an understanding is developed on the role of the pitch angle $\Delta\theta$ on the propagation of matrix cracks forming at the back face of ultra-thin-ply CFRP Bouligand laminates under impact.

2.2 Materials and methods

2.2.1 Manufacturing

We designed five different Bouligand configurations, with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° . All configurations have the same nominal thickness with a total of 146 plies, and are all symmetric with respect their mid-plane. We decided to study symmetric laminates in order to avoid the in-plane/bending coupling of an asymmetric lay-up ($\mathbf{B} \neq 0$), which depends on the pitch angle. Therefore, by using a symmetric laminate, we can better relate the findings of this work to the pitch angle $\Delta\theta$ directly. Table 2.2 shows the stacking sequence of each configuration. We considered the $\Delta\theta = 45^\circ$ configuration as the reference configuration in this study since it most closely matches a quasi-isotropic laminate used in engineering practice.

We manufactured samples using Skyflex USN20A, a unidirectional ultra-thin-ply pre-preg (20 gsm). The very low thickness of Skyflex USN20A has allowed to reach, for the first time in the literature in a symmetrical and balanced configuration, extremely low pitch angles $\Delta\theta = 2.5^\circ$ yet maintaining reasonable laminate thicknesses (~ 3.5 mm). The ply-level and the

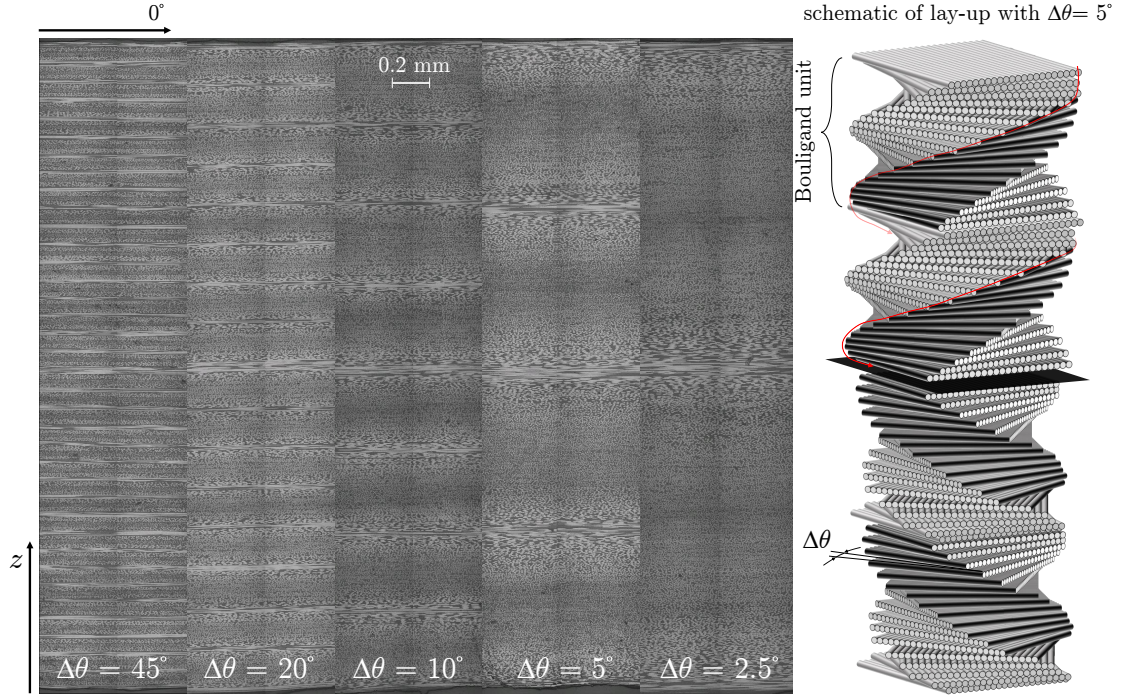


Figure 2.3: Micrographs of ultra-thin-ply CFRP Bouligand composites with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° .

constituents properties of Skyflex USN20A are reported in Tables 2.3 and 2.4, respectively. We used an automatic cutting table to cut each ply at the desired angle. We ensured excellent alignment during the lay-up procedure by means of an alignment mould with pin holes [23]. We cured the samples in the same autoclave cycle according to the pre-preg manufacturer's recommendations. After curing, we cut the plates to their final dimensions (150 mm long and 100 mm wide [24, 25]) using a water-jet cutter. We cut the samples such that the 0° -layers were aligned along the length of the samples. Finally, we scanned each sample in an immersion tank with a ultrasonic C-scan in order to verify that there were no defects. Figure 2.3 shows

Table 2.2: Samples specifications for LVI and CAI tests (Mean $\pm$ STD).

$\Delta\theta$	Stacking sequence	n°of samples	thickness [mm]	n°of plies	Energy [J]	Impact height [mm]
2.5°	$[0^\circ/2.5^\circ/5^\circ/\dots/180^\circ]_s$	3	3.56 ± 0.02	146	11.94 ± 0.08	221.29 ± 1.78
5°	$[0^\circ/5^\circ/10^\circ/\dots/360^\circ]_s$	1	3.57	146	11.96	221.66
10°	$[0^\circ/10^\circ/20^\circ/\dots/720^\circ]_s$	1	3.60	146	12.06	223.51
20°	$[0^\circ/20^\circ/40^\circ/\dots/1440^\circ]_s$	1	3.52	146	11.79	218.51
45°	$[0^\circ/45^\circ/90^\circ/\dots/3240^\circ]_s$	1	3.51	146	11.75	217.77

micrographs of the 5 microstructures. The details of the manufactured samples are reported in Table 2.2.

Our goals were to:

- (i) characterize the $\Delta\theta=2.5^\circ$ configuration because it is the one that better mimics the features of naturally-occurring Bouligand microstructures, and
- (ii) investigate the effect of pitch angle $\Delta\theta$ on various characteristics of the response, i.e. to understand how quantities such as projected delamination area during impact correlate with pitch angle.

For goal (i), we manufactured 3 identical samples with pitch angle $\Delta\theta=2.5^\circ$ for impact and compression after impact testing. For goal (ii), we manufactured 4 further samples, with pitch angles $\Delta\theta=5^\circ$, 10° , 20° and 45° respectively, also for impact and compression after impact testing.

With regards to goal (ii), note that we do not have specimen repetitions for $\Delta\theta=5^\circ$, 10° , 20° and 45° and hence we will not be able to characterize the scatter for these configurations. However, we have seven specimens whose test results can be used to carry out statistical hypothesis tests on whether correlations between different measurements and pitch angle exist, which is our goal in this chapter.

2.2.2 Testing methods: LVI and CAI

We conducted LVI tests using a drop-weight tower (Instron CEAST 9350) following the testing procedure ASTM D7136/D7136M [24]. We kept the impactor mass (5.5 kg) and geometry (hemispherical tip, 8 mm radius) constant in each test. Plates (150 mm long and 100 mm wide, with the 0° -layers aligned along the length of the samples) were simply supported by four rubber pins over a rectangular opening of 125 mm x 75 mm, i.e. testing window of the fixture [24]. To achieve a reasonable amount of damage, we impacted each sample with an impact energy per unit thickness of 3.35 J/mm (Table 2.2). The impactor reached the laminates with an average velocity of 2.08 m/s. We quantified the dissipated energy of each sample by computing the area under the load vs. displacement curve, where the displacement represents the displacement of the impactor. We conducted post-impact damage analysis of the samples by

Table 2.3: Elastic properties of the Skyflex USN20A plies [26] and its constituent.

E_{c1}	$E_{c2} = E_{c3}$	$\nu_{c12} = \nu_{c13}$	$G_{c12} = G_{c13}$
101.7 GPa	6.0 GPa	0.3	2.4 GPa

Table 2.4: Constituents properties of Skyflex USN20A [26, 27].

Mitsubishi Rayon TR30S 3K		SK Chemicals K50	
Fibre diameter, (ϕ_f)	6.8 μm	Young's modulus, E_m	3.35 GPa
Tensile modulus, (E_{f1})	235 GPa	Shear modulus, G_m	1.21 GPa
Compression modulus, (E_{f2})	13 GPa	Poisson's ratio, ν_m	0.38
Shear modulus, (G_{f12})	15 GPa	Density (cured), ρ_m	1.30 g/cm ³
Poisson's ratio, (ν_{f12})	0.2		
Fibre density, (ρ_f)	1.79 g/cm ³		
Tensile failure strain, (ε_f^*)	1.9 %		

means of visual inspections and ultrasonic C-scans to investigate the distribution of damage, the delamination extent and the projected delamination area. The latter was evaluated by post-processing the C-scan images. The image analysis comprises the following steps: (i) grey scale transformation and binarisation of the C-scan images (the threshold for discretisation of black (no-damage) and white (damage) pixels was the same for each sample); (ii) scaling of the pixel dimension to millimetre; and (iii) counting of the white pixels and calculation of the projected delamination area. Additionally, for each sample, we performed visual inspection of the back face to provide a measure of the extent of back face damage. This was calculated as the diameter ϕ of the smallest circle that encloses the back face damage detectable by visual inspection.

We performed CAI tests following the testing procedure ASTM D7137/D7137M [25]. Samples were loaded with a constant displacement rate of 1.25 mm/min using a 250 kN load cell Instron machine, with the long side of the samples, i.e. 0°-layers, aligned to the loading direction. We instrumented each sample with four strain gauges [25], two on the impacted side and two on the back face in order to monitor strains during the test, to verify that the initial alignment of the sample was within acceptable bounds [25] and that global buckling did not occur. In addition, we used 3D digital image correlation on the impacted surface to verify that global buckling did not occur during CAI.

Table 2.5: Peak load, displacement at peak load, and dissipated energy of LVI tests. Results for $\Delta\theta = 2.5^\circ$ are presented in terms of Mean $\pm$ STD.

$\Delta\theta$	Peak load [kN]	Displacement at peak load [mm]	Dissipated energy [J]
2.5°	5.62 ± 0.092	4.13 ± 0.25	7.1 ± 1.03
5°	4.72	3.26	7.3
10°	5.37	3.16	6.8
20°	5.35	3.23	8.0
45°	4.96	3.06	7.9

2.3 Results: LVI and CAI

For the LVI tests, Table 2.5 shows the peak load, corresponding displacement and dissipated energy for each pitch angle $\Delta\theta$. Figures 2.4a and 2.4b respectively show the load vs time and load vs displacement curves for all the samples tested.

The $\Delta\theta = 2.5^\circ$ response (Figures 2.4a and 2.4b) shows a region with a progressive reduction in stiffness until just before the load drop. For $\Delta\theta = 5^\circ, 10^\circ, 20^\circ$ and 45° , this region does not exist and the linear-elastic behavior is followed by sudden load-drops of increasing magnitude as the pitch angle $\Delta\theta$ increases. Based on the samples tested in this work, i.e. 3 samples for $\Delta\theta = 2.5^\circ$ and one sample for each one of the other configurations, the $\Delta\theta = 2.5^\circ$ configuration showed a 13.3% higher peak load and a 34.9% higher peak displacement than the $\Delta\theta = 45^\circ$ configuration.

Figure 2.5 illustrates the three-dimensional representation of the C-scan images of the impacted samples, representing the delamination damage for each configuration $\Delta\theta$. The coloured regions represent the delamination areas with different colours associated to different ply-interfaces. Figure 2.5 shows that reducing the pitch angle $\Delta\theta$ leads to a progressively smoother double helicoidal distribution of damage.

Figure 2.5f shows that, while in the $\Delta\theta = 2.5^\circ$ configuration the through-the-thickness distribution of delaminations is approximately uniform, in the $\Delta\theta = 45^\circ$ configuration they are distributed in a ‘step-wise’ manner (with big jumps occurring only at specific locations across the thickness).

We used a least-square linear regression model to calculate trendlines for the effect of the pitch angle $\Delta\theta$ on the total projected delamination area, delamination extent and back face damage (Figures 2.6a-2.6c). We performed a calculation of the p -values associated to the slope m of each trendline in order to assess their statistical significance. The value of p corresponds to the probability of the trendline to have a zero slope ($m = 0$) given the experimental data measured. As is common in the literature, we set the threshold value $p \leq 0.05$ to conclude that a certain slope is non-zero with sufficient statistical significance.

Based on the samples tested in this work, i.e. 3 samples for $\Delta\theta = 2.5^\circ$ and one sample for each one of the other configurations, Figure 2.6 shows that reducing the pitch angle $\Delta\theta$ leads to a statistically-significant decrease in total projected delamination area (with a global reduction of 29.4% between $\Delta\theta = 2.5^\circ$ and $\Delta\theta = 45^\circ$), in delamination extent (35.8% decrease) and in extent of back-face damage (46.8% decrease).

Figure 2.7a and 2.7b respectively show the CAI strength and failure strain of all the different configurations. The pitch angle $\Delta\theta$ did not show a statistically-significant effect on the residual properties after impact ($p > 0.05$) and all configurations showed similar values of residual CAI strength and failure strain.

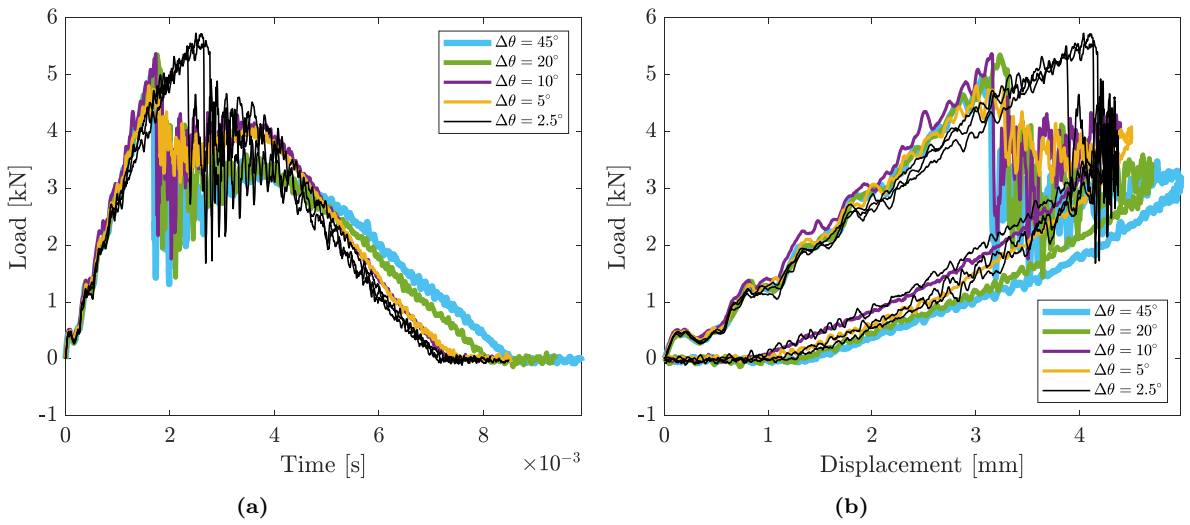


Figure 2.4: LVI (a) load vs time and (b) load vs displacement for $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° .

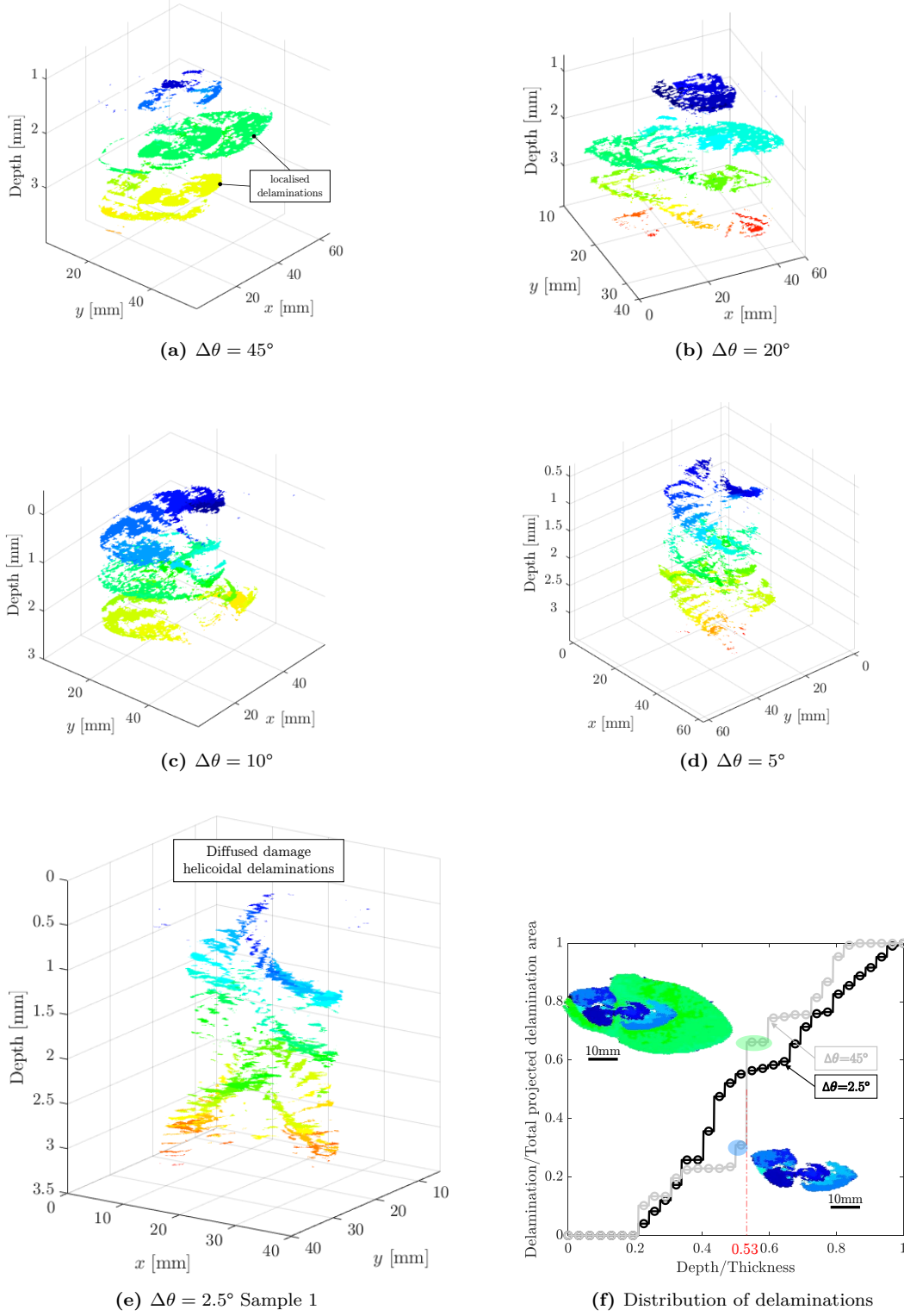


Figure 2.5: (a-e) Three-dimensional representation of ultrasonic C-scans for configurations with different pitch angles $\Delta\theta$. (f) Through-the-thickness distribution of delaminations in the $\Delta\theta = 2.5^\circ$ (Sample 1) and $\Delta\theta = 45^\circ$ configurations.

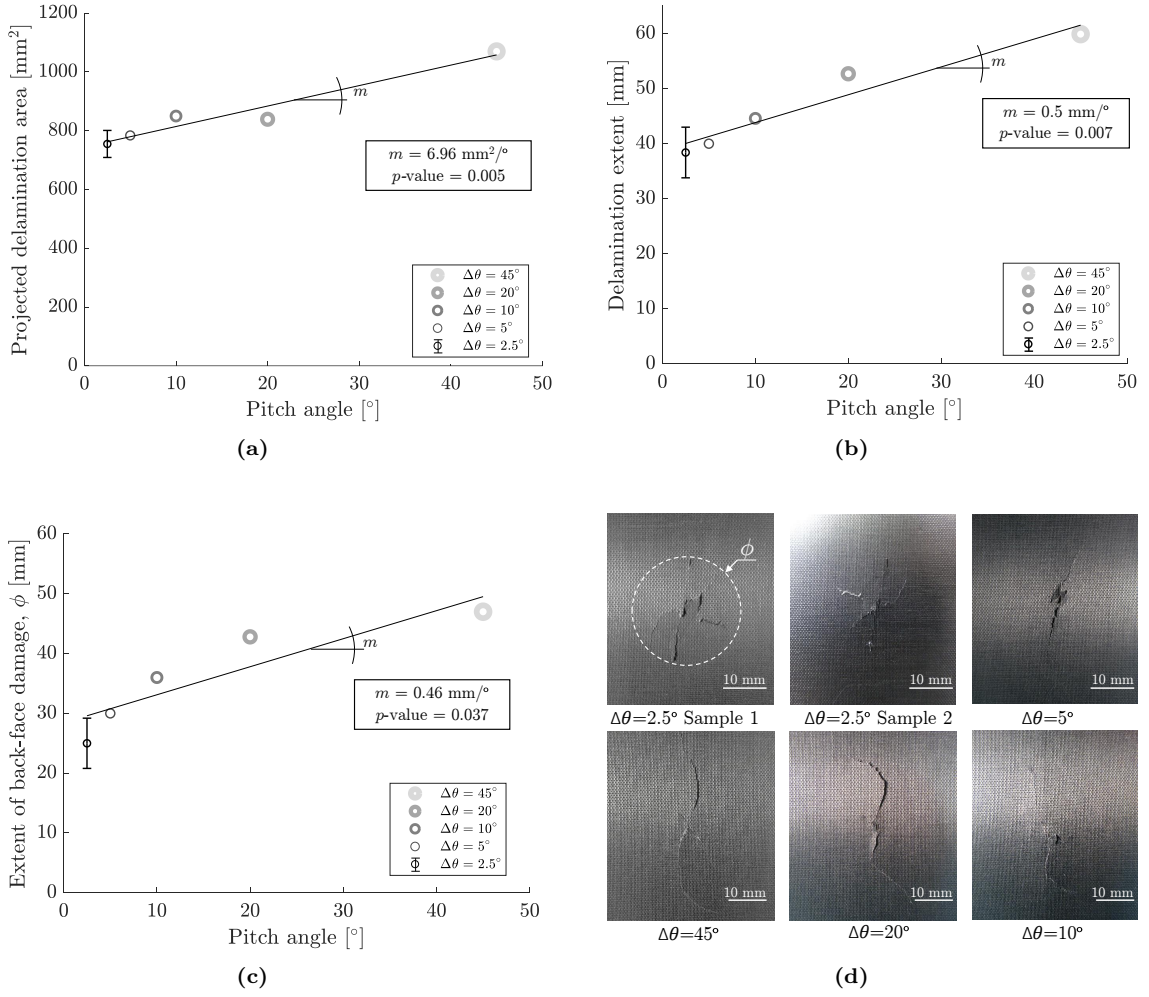


Figure 2.6: Effect of the pitch angle $\Delta\theta$ on (a) projected delamination area, (b) delamination extent and (c) extent of back-face damage, ϕ (LVI). (d) Damage at the distal face, showing fibre failures and splitting.

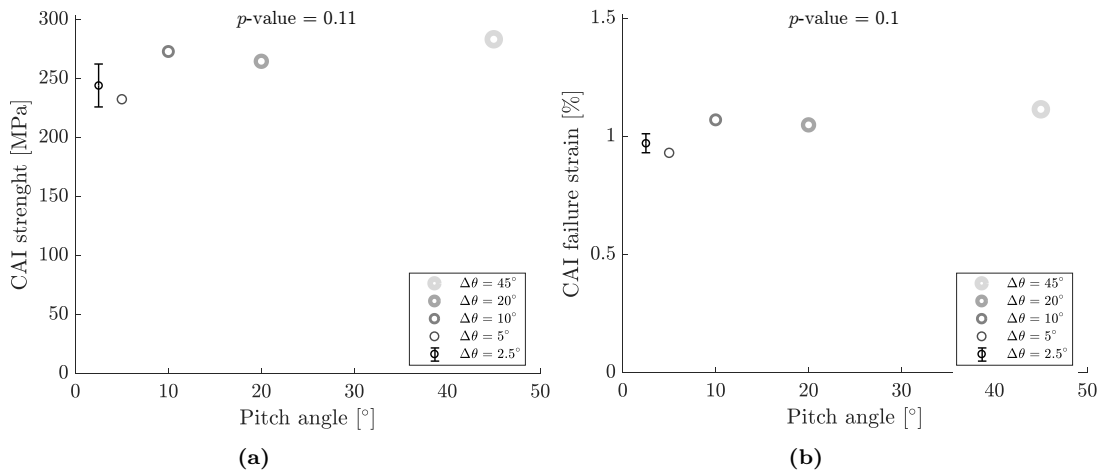


Figure 2.7: (a) CAI strength and (b) CAI failure strain for all pitch angles $\Delta\theta$ showing no statistically-significant trends.

2.4 Discussion

2.4.1 The enhanced impact damage resistance of ultra-thin-ply CFRPs Bouligand laminates

As the pitch angle $\Delta\theta$ decreases, the damage due to impact follows an increasingly better-defined double helicoidal (Figure 2.5), and is more equally-spread across the thickness (with smaller delaminations at more interfaces, Figure 2.5f). The smooth and highly diffused double helicoidal delamination pattern in the $\Delta\theta = 2.5^\circ$ (Figure 2.5e) correlates with a substantial increase in displacement at peak load (Figure 2.4b) and in the energy dissipated prior to the peak load (related to the non-linear region before the peak load in Figure 2.4b). Therefore, reducing the pitch angle to very small values, down to $\Delta\theta = 2.5^\circ$, leads to higher impact resistance via a significant sub-critical accumulation of damage.

For $\Delta\theta = 2.5^\circ$, Figure 2.4a shows sub-critical damage accumulation before the peak load. The low impact energy used in the tests is not likely to have caused fibre failure in the bulk of the laminate at this stage of the test; fibre failure did occur at the distal face (Figure 2.6d) later in the test, and is related to the load drop visible in Figures 2.4a and 2.4b. Therefore, we conclude that the sub-critical damage is the result of the double helicoidal matrix crack and delamination pattern.

Regarding the load drop due to the fibre failure at the distal face mentioned, Figure 2.4b shows that this occurs at larger displacements for $\Delta\theta = 2.5^\circ$. Therefore, there is also a correlation between the formation of the double helicoidal damage pattern and the delay in fibre failure at the distal face.

Table 2.5 shows that decreasing the pitch angle $\Delta\theta$ leads to similar dissipated energies. This was achieved via an increasing amount of diffused sub-critical helicoidal damage as the pitch angle decreases. Therefore, by decreasing the pitch angle, it is possible to preserve the energy dissipation capability of the structure via dissipating at a sub-critical level rather than via the activation of catastrophic failure mechanisms (such as the large localised delaminations and extensive fibre breaks observed in $\Delta\theta = 20^\circ$ and 45° , see Figures 2.5 and 2.6).

2.4.2 The effect of the pitch angle $\Delta\theta$ on the distribution of sub-critical damage: delaminations and shearing matrix cracks

2.4.2.1 Role of τ_{13} and τ_{23} on the failure mechanisms in LVI tests

In § 2.4.1, we observed that smaller pitch angles lead to an increasingly better-defined double helicoidal damage pattern (Figure 2.5) and a more homogeneous distribution of delaminations (Figure 2.5f). In this section we explore the reasons why this may happen.

We know that, during an impact on a composite laminate, the first damage to form is typically matrix cracking; matrix cracks may start in the bulk of the laminate due to shear stresses (shearing cracks), and from the distal face due to bending tensile stresses (bending cracks) [28]. In thin-ply CFRP laminates, shearing cracks and bending cracks do not interact until after fibre failure at the distal face has occurred [29,30]. In addition, the helicoidal damage experimentally observed in Figure 2.5 expands around the mid-plane of the laminate, where shearing cracks are expected. Using a combined experimental and analytical approach, Choi et al. [28] showed that the shear stress distributions τ_{13} and τ_{23} play a major role in the failure of CFRP laminated composites subjected to low velocity impact. Therefore, we will focus here on how the conditions for initiation of shearing cracks may depend on the pitch angle $\Delta\theta$.

In each ply- i , shearing cracks are caused by the τ_{23}^i shear stress component (in material coordinates, see Figure 2.8). Therefore, in each ply- i , if shearing cracks are to form, we expect them to be located at the point(s) corresponding to maxima of $|\tau_{23}^i|$ (it will be shown later that there are two per ply). Additionally, we know that dynamic effects are not essential for determining damage initiation in LVI [31]. Therefore, the effect of the pitch angle $\Delta\theta$ on the expected locus for the shearing matrix cracks can be inferred from the locus of the maxima of $|\tau_{23}^i|$ in a suitable static linear elastic finite element model.

At the interface between two generic plies $(i-1)$ and i , Choi et al. [28] showed that delamination formation starting from shearing cracks in LVI loading conditions is due to $|\tau_{23}^i|$ in the ply- i (ply above) and $|\tau_{13}^{i-1}|$ in the ply- $(i-1)$ (ply below) (Figure 2.8). Delaminations then grow aligned with the fibre direction of the ply below [28, 31–33] as schematically illustrated in Figure 2.8, and hence we know that $|\tau_{13}^{i-1}|$ plays a significant role in delamination growth for the interface between plies $(i-1)$ and i . Therefore, the effect of the pitch angle $\Delta\theta$ on the expected locus for delamination at the interface between plies $(i-1)$ and i can in principle be

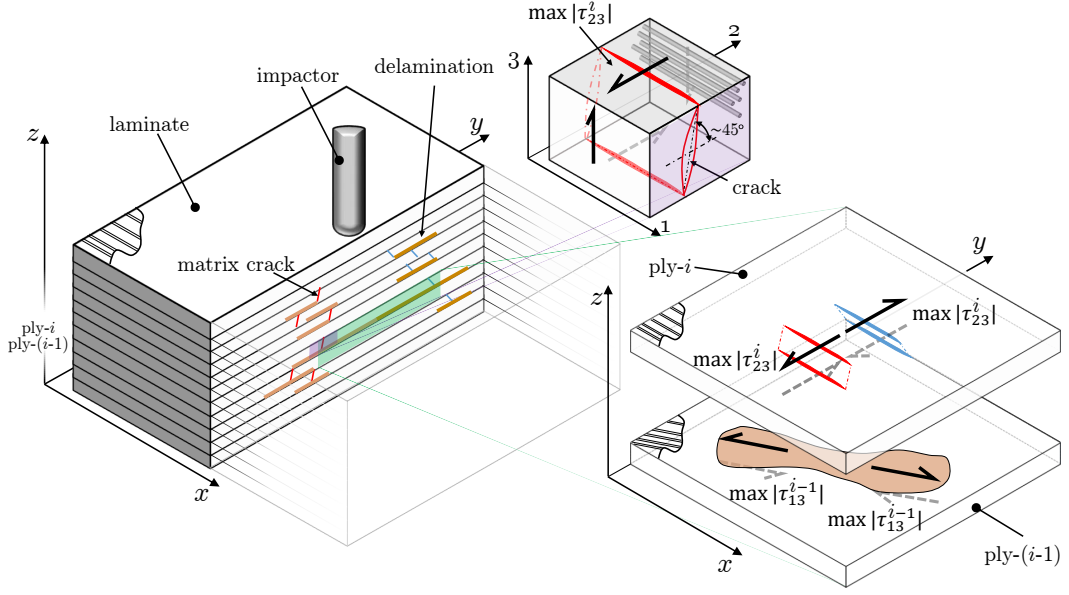


Figure 2.8: Schematic of the failure mechanisms taking place in the bulk of a laminate composite under out-of-plane loading. Firstly, shearing matrix cracks will form in a generic ply- i due to the intra-laminar τ_{23}^i shear stress. Subsequently, a delamination at a given interface between two plies, $(i-1)$ and i , can form from two cracks driven by the intra-laminar τ_{23}^i shear stress of the ply above and the intra-laminar τ_{13}^{i-1} shear stress of the ply below the interface [28].

inferred from the maxima of $|\tau_{23}^i|$ and $|\tau_{13}^{i-1}|$ in a suitable static linear elastic finite element model.

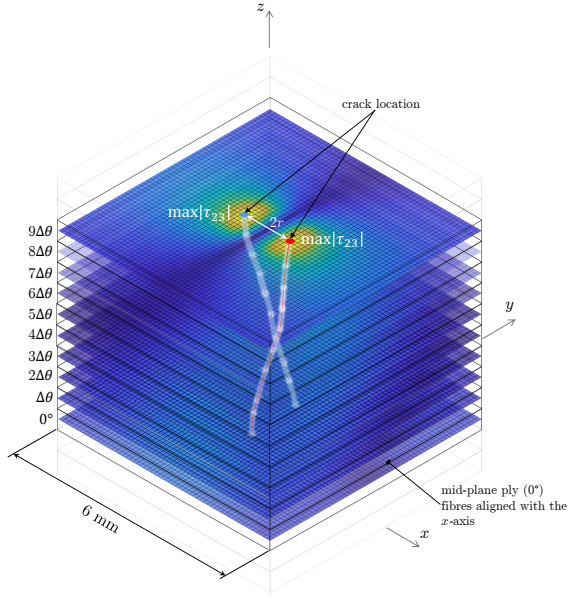
2.4.2.2 FE model

We created five static linear elastic models, one for each pitch angle $\Delta\theta$, using the FE software Abaqus [34]. Both geometries of the laminate (150 mm x 100 mm) and the indenter (hemispherical with a 8 mm tip radius) were the same as the ones used in actual testing conditions. The material properties assigned to each lamina are reported in Table 2.3. Note that being the lamina transversely isotropic, $G_{c12} = G_{c13} \neq G_{c23}$ and $\nu_{c12} = \nu_{c13} \neq \nu_{c23}$. In the FE models, we assumed $G_{c23} = G_{c12} (= G_{c13})$ and $\nu_{c23} = \nu_{c12} (= \nu_{c13})$. The elastic properties were commonly assigned to each FE model therefore, qualitative comparisons between the different model-results still hold true. While we assumed a linear elastic material behaviour, the effect of material non-linearities on the shear stress distributions was not investigated in this work. Since we used a linear elastic material behaviour, with no damage being modelled, we used conventional quadratic shell elements (S8R) to model the laminate (ply-by-ply using lamination theory) and an analytical surface for the indenter. We conducted a mesh convergence

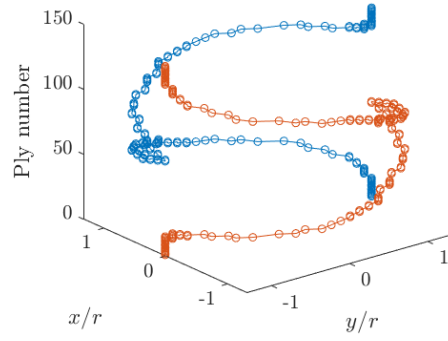
study in order to guarantee a good representation of the elastic response to transverse load of the sample tested. To this end, we used four parameters in order to determine the minimum element size in the impact region: (i) distribution of maximum τ_{13} , (ii) distribution of maximum τ_{23} , (iii) location of points with maximum τ_{23} and (iv) location of points with maximum τ_{23} . The mesh of the impact region converged at a minimum element size of 0.06 mm. We then discretised the laminate with a bias mesh. Samples were simply supported along a rectangular perimeter (125 mm x 75 mm) centred to the plate, resulting in boundary conditions similar to the one provided by the LVI test fixture (ASTM D7136/D7136M [24]). A vertical downward displacement of 2 mm was applied to the indenter.

2.4.2.3 FE results and analysis

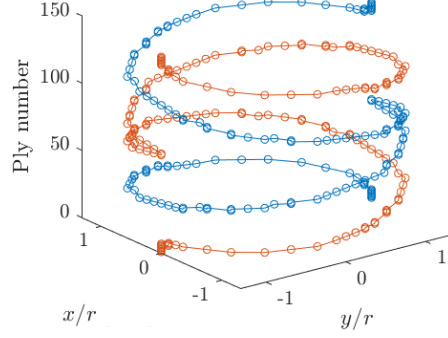
For each ply- i , Figure 2.9 shows the likely location of shearing cracks, i.e. points with maximum $|\tau_{23}^i|$. In each ply- i , $|\tau_{23}^i|$ exhibits two identical maximum values (Figure 2.9a). The locus of points with maximum $|\tau_{23}^i|$ form a double helix, which can be observed in Figure 2.9b to 2.9f for $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° respectively. The double helices are symmetric about the laminate symmetry plane and are better defined for smaller $\Delta\theta$. When observed perpendicularly to the plane of the laminate, all helices corresponding to different $\Delta\theta$ collapse on the same circle, as shown in Figure 2.9g.



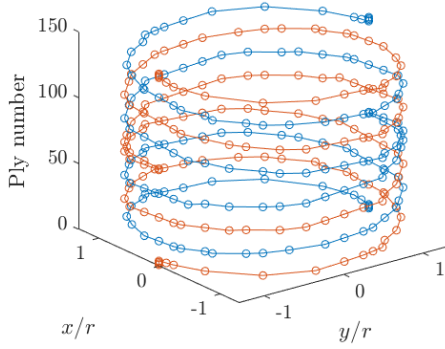
(a) Zoom of the FE model near the impact region, highlighting the double helicoidal distribution in the laminate of the points with $\max|\tau_{23}^i|$ corresponding to the locations of shearing matrix cracks



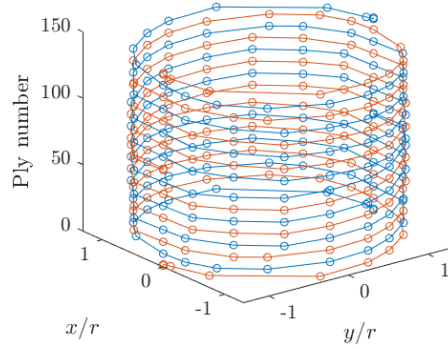
(b) $\Delta\theta = 2.5^\circ$ locus of $\max|\tau_{23}^i|$



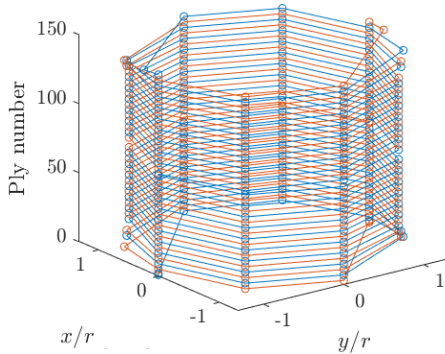
(c) $\Delta\theta = 5^\circ$ locus of $\max|\tau_{23}^i|$



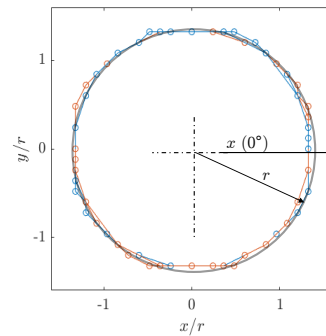
(d) $\Delta\theta = 10^\circ$ locus of $\max|\tau_{23}^i|$



(e) $\Delta\theta = 20^\circ$ locus of $\max|\tau_{23}^i|$



(f) $\Delta\theta = 45^\circ$ locus of $\max|\tau_{23}^i|$



(g) $(x - y)$ projection of $\max|\tau_{23}^i|$ loci (all $\Delta\theta$)

Figure 2.9: Loci of likely shearing matrix cracks, forming in each ply- i at the locations with $\max|\tau_{23}^i|$, exhibiting a double helicoidal pattern of radius r as $\Delta\theta$ decreases.

Figure 2.10a shows the through-the-thickness distributions of maximum $|\tau_{23}|$ for laminates with different pitch angles $\Delta\theta$. We normalised each distribution to the highest value of the distribution of maximum $|\tau_{23}^{45^\circ}|$ in order to highlight the differences between the configurations modelled. Figure 2.10a shows that for the $\Delta\theta = 2.5^\circ$ configuration, the maximum value of $|\tau_{23}|$ is higher than for any other configurations. This suggests that sub-critical damage (in the form of matrix cracks) should occur earlier for the $\Delta\theta = 2.5^\circ$ configuration; this is consistent with the experimental observation of early loss of linearity observed in the $\Delta\theta = 2.5^\circ$ laminates (see Figure 2.4a).

Delaminations can potentially initiate from the predicted shearing cracks (see § 2.4.2.1). The analysis of the synergistic effect between τ_{23} and τ_{13} in promoting delaminations in configurations with different pitch angles $\Delta\theta$ can be reduced to the study of two consecutive plies, $(i-1)$ and i , and their interface. To this effect, Figure 2.11a shows a generic ply- i , together with ply- $(i-1)$ at an angle $-\Delta\theta$. For ply- i , Figure 2.11a shows the $|\tau_{23}^i|$ field, and highlights the two locations where $|\tau_{23}^i|$ has a maximum and hence shearing cracks are expected to occur. For ply $(i-1)$, Figure 2.11a shows the $|\tau_{13}^{i-1}|$ field specifically for $\Delta\theta = 2.5^\circ$, and highlights (for various possible values of $\Delta\theta$) the two locations where $|\tau_{13}^{i-1}|$ has a maximum and hence where the conditions for delamination growth from a shearing crack are ideal.

Figure 2.11b shows a projection of Figure 2.11a on the plane of the laminate, showing that the locus of maximum $|\tau_{13}|$ projects to the same circle corresponding to the projection of maximum $|\tau_{23}|$ shown in Figure 2.9g. Importantly, Figure 2.11b shows that the distance between

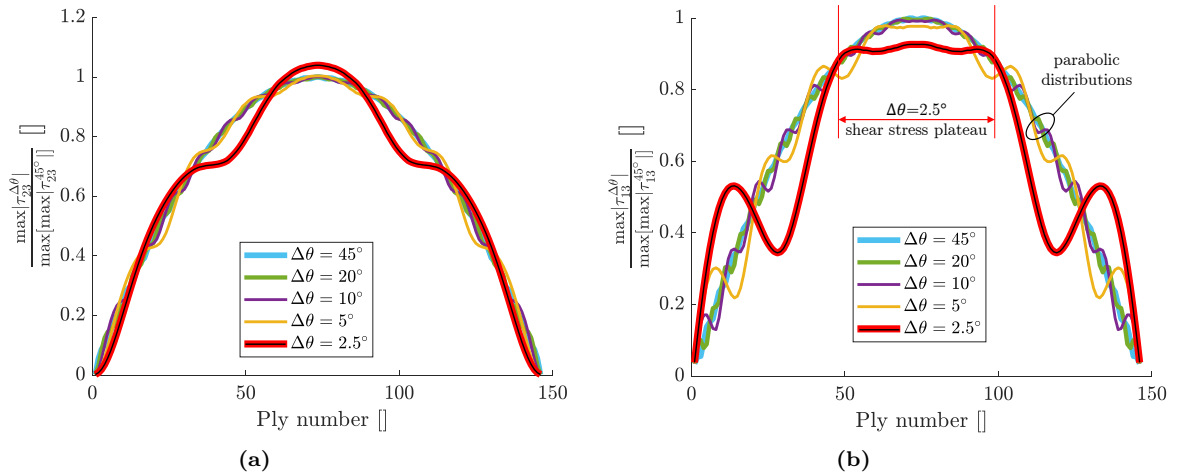


Figure 2.10: Through-the-thickness distribution of (a) maximum $|\tau_{23}|$ and (b) maximum $|\tau_{13}|$ for different pitch angles $\Delta\theta$.

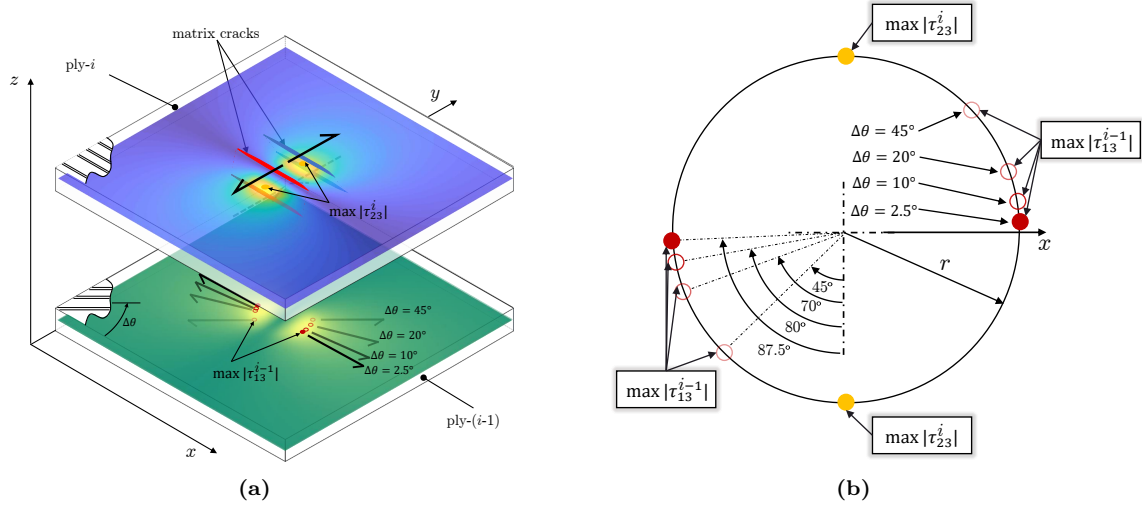


Figure 2.11: Synergistic effect between the maximum values of $|\tau_{23}^i|$ in the ply- i above, and $|\tau_{13}^{i-1}|$ in the ply- $(i-1)$ below a given interface for $\Delta\theta = 2.5^\circ, 10^\circ, 20^\circ$ and 45° . (a) Schematic of the two consecutive plies i and $(i-1)$. Two shearing cracks are illustrated in the ply i , located at the points with maximum $|\tau_{23}^i|$ (yellow dots). The red dots in the ply $(i-1)$ correspond to the location of maximum $|\tau_{13}^{i-1}|$ for different pitch angles $\Delta\theta$. (b) Projection of the points illustrated in Figure 2.11a in the plane of the laminate.

the maximum $|\tau_{13}^{i-1}|$ and maximum $|\tau_{23}^i|$ decreases as the pitch angle $\Delta\theta$ increases. Therefore, increasing the pitch angle $\Delta\theta$ is expected to promote the growth of delaminations from shearing cracks.

Figure 2.10b shows the thorough-the-thickness distribution of maximum $|\tau_{13}|$ for laminates with different pitch angles $\Delta\theta$. We normalised each distribution to the highest value of the distribution of maximum $|\tau_{13}^{45^\circ}|$ in order to highlight the differences between the configurations modelled. While the curves for most values of $\Delta\theta$ exhibit a parabolic, it can be observed that, for $\Delta\theta = 2.5^\circ$, there is a substantial plateau. Importantly, the existence of this plateau means that, for $\Delta\theta = 2.5^\circ$, several interfaces will undergo similar maximum shear stresses. This correlates well with the diffused delaminations through the thickness of the laminates with pitch angle $\Delta\theta = 2.5^\circ$ observed experimentally (Figures 2.5e and 2.5f).

Additionally, for laminates with large pitch angles, such as $\Delta\theta = 45^\circ$, delaminations are expected to localize near the few interfaces with maximum $|\tau_{13}|$ and therefore, firstly at the mid-plane of the laminate. This correlates well with the large delamination at the mid-plane of the laminate observed experimentally (Figures 2.5a). Furthermore, once this central delamination has grown significantly, given the high periodicity of configurations with large pitch angles $\Delta\theta$, a similar analysis for each half of the laminate leads to the expectation of delaminations at

the one- and three- quarter height of the laminate, and this is also observed experimentally (Figures 2.5a).

As mentioned in § 2.4.2.2, we modelled each laminate using a linear elastic material behaviour. In this work, we did not investigate the effect of a non-linear material behaviour on the distributions of maximum $|\tau_{13}|$ and maximum $|\tau_{23}|$. This is likely to lead to a slight flattening of the stress distributions reported in Figure 2.10, potentially resulting in: (i) a slightly less severe localisation of delamination damage in configurations with large pitch angles and (ii) a slightly larger shear stress plateau (see Figure 2.10b) in the $\Delta\theta = 2.5^\circ$ configuration, thus further promoting a homogeneous through-the-thickness distribution of delaminations.

2.4.3 The effect of the pitch angle $\Delta\theta$ on the distribution of sub-critical damage: twisting Bouligand cracks

In Sections 2.4.2.1-2.4.2.3 we have investigated the role of pitch angle $\Delta\theta$ on the formation of shearing matrix cracks and delaminations in Bouligand CFRP ultra-thin-ply laminates subjected to low velocity impact. In this section, we will discuss the role of the pitch angle $\Delta\theta$ and ply thickness t_{ply} on the energy release rate for the growth of twisting Bouligand-like cracks forming at the back face of the laminate (LVI tests) due to the tensile stresses originating from bending.

In this context, twisting crack models [35] can be particularly helpful, and therefore we implemented the recent model developed by Suksangpanya et al. [20, 21]. This model analyses local stress intensity factors along the crack front of a continuously twisting crack in a Bouligand medium, starting from an initially flat crack, see Figure 2.12. In our case, this flat crack represents a matrix split at the back face due to the tensile stresses originating from bending. The crack front is initially aligned with the coordinate system (x, y, z) , with the origin of the coordinate system located at the crack front, and where the z -axis defines the initial crack growth direction and the x -axis is normal to the initial crack plane. In our problem, the y -axis therefore coincides with the fibre direction at the initial crack front. As the crack grows, kinks and twists, see Figure 2.12, the local coordinate system of the crack front at any given time is (x'', y'', z'') , while the original coordinate system translated to a generic point along the new crack front is (x', y', z') . Again, in our problem, the y'' -axis therefore coincides with the local fibre direction at the current crack front. Figure 2.12 shows that the twisting of the crack front

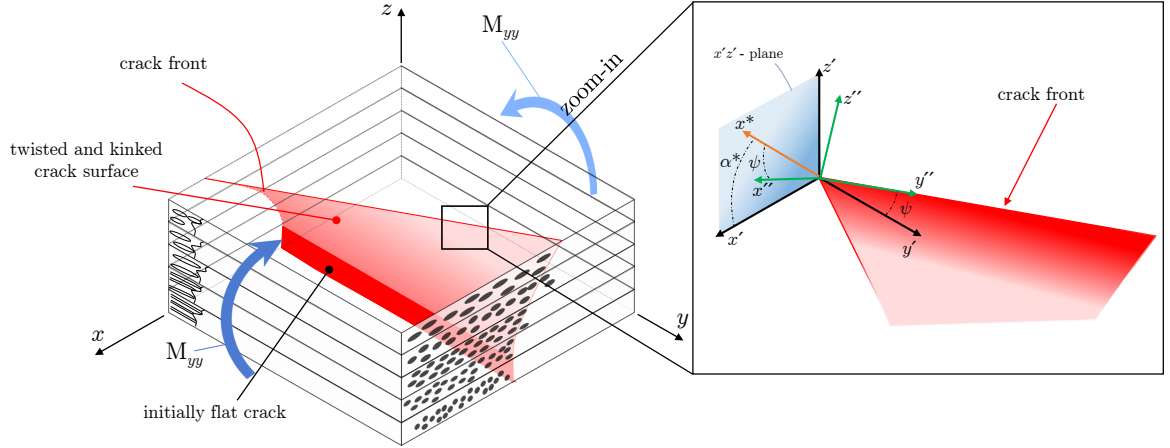


Figure 2.12: Schematic of a twisting crack in a Bouligand structure initiating from a flat crack. During impact, the bending moment M_{yy} results in tensile bending stresses at the bottom of the laminate that tend to open the initially flat matrix split in Mode I.

can be defined by the angle ψ between y' and y'' , and, for a fibre-reinforced laminate with individual ply thickness t_{ply} , it is given by

$$\psi = z \left(\frac{\Delta\theta}{t_{\text{ply}}} \right). \quad (2.1)$$

In addition, the kinking of the crack is defined by the effective kink angle α^* , defined as the angle between x^* and x' , where x^* is obtained by rotating x'' about z' by an angle $-\psi$ (Figure 2.12). For a fibre-reinforced laminate with individual ply thickness t_{ply} , it can be expressed as (based on [20]):

$$\alpha^* = \cos^{-1} \left\{ \frac{\cos \psi [1 + \tan^2 \psi]}{\sqrt{[(y\Delta\theta/t_{\text{ply}}) \sec^2 \psi]^2 + 1 + \tan^2 \psi}} \right\}. \quad (2.2)$$

To obtain a solution to the energy release rate for the problem described, Suksangpanya et al. [20] assume the following: (i) the helicoidal crack grows in an isotropic elastic medium with isotropic fracture toughness (which is an approximation in our case); (ii) the crack front along the twisted path remains straight; (iii) other competing failure mechanisms such as delaminations and fibre breaks do not occur (which in our case neglects interaction with shearing cracks starting in the bulk of the laminate); and (iv) the flat crack is loaded in Mode I with a stress intensity factor K_I . The bending moment M_{yy} reported in Figure 2.12 generates tensile stresses at the back-face of the laminate, hence introducing a Mode I opening to a matrix split

(flat crack in Figure 2.12) forming parallel to the fibres of the bottom ply. Therefore, a Mode I opening is representative of the loading condition for bending matrix cracks forming at the back face of the laminate under LVI tests.

Based on this, the energy release rate G at each point on the twisting crack front, normalised to the energy release rate if the crack were to remain flat (G_0), is [20]

$$\frac{G}{G_0} = \left(\frac{k_I''}{K_I} \right)^2 + \left(\frac{k_{II}''}{K_I} \right)^2 + \frac{1}{1-\nu} \left(\frac{k_{III}''}{K_I} \right)^2, \quad (2.3)$$

where ν is the Poisson's ratio of the isotropic medium, and the local stress intensity factors along the crack front k_I'' , k_{II}'' and k_{III}'' are expressed as [20]

$$\frac{k_I''}{K_I} = \cos \left(\frac{\alpha^*}{2} \right) \left[\cos^2 \left(\frac{\alpha^*}{2} \right) \cos^2 \psi + 2\nu \sin^2 \psi \right], \quad (2.4a)$$

$$\frac{k_{II}''}{K_I} = \sin \left(\frac{\alpha^*}{2} \right) \cos^2 \left(\frac{\alpha^*}{2} \right) \cos \psi, \quad (2.4b)$$

$$\frac{k_{III}''}{K_I} = \cos \left(\frac{\alpha^*}{2} \right) \sin \psi \cos \psi \left[\cos^2 \left(\frac{\alpha^*}{2} \right) - 2\nu \right]. \quad (2.4c)$$

For the design of Bouligand CFRP laminates, the parameters used to tailor the microstructure are the pitch angle $\Delta\theta$ and the ply thickness t_{ply} . For each of the five configurations used in this study, Figures 2.13a-2.13b show the energy release rate during crack growth, where it can be seen that the energy release rate for a twisting crack decreases with increasing pitch angle $\Delta\theta$. This result suggests that bending cracks should propagate more easily towards the bulk of the laminate for small pitch angles than for large pitch angles. This is in good agreement with the experimental observations conducted in this work, where the smallest pitch angle $\Delta\theta = 2.5^\circ$ showed helicoidal damage forming at sub-critical levels diffused throughout the entire thickness of the laminate (Figure 2.5e) and delayed catastrophic failure (load-drop in Figure 2.4).

Additionally, Figure 2.13c shows that for decreasing pitch angles $\Delta\theta$, the first local minimum of G/G_0 (circles in Figure 2.13c), corresponding to a twisting crack front perpendicular to the initially flat crack, is delayed to larger values of z . This also suggests that smaller pitch angles

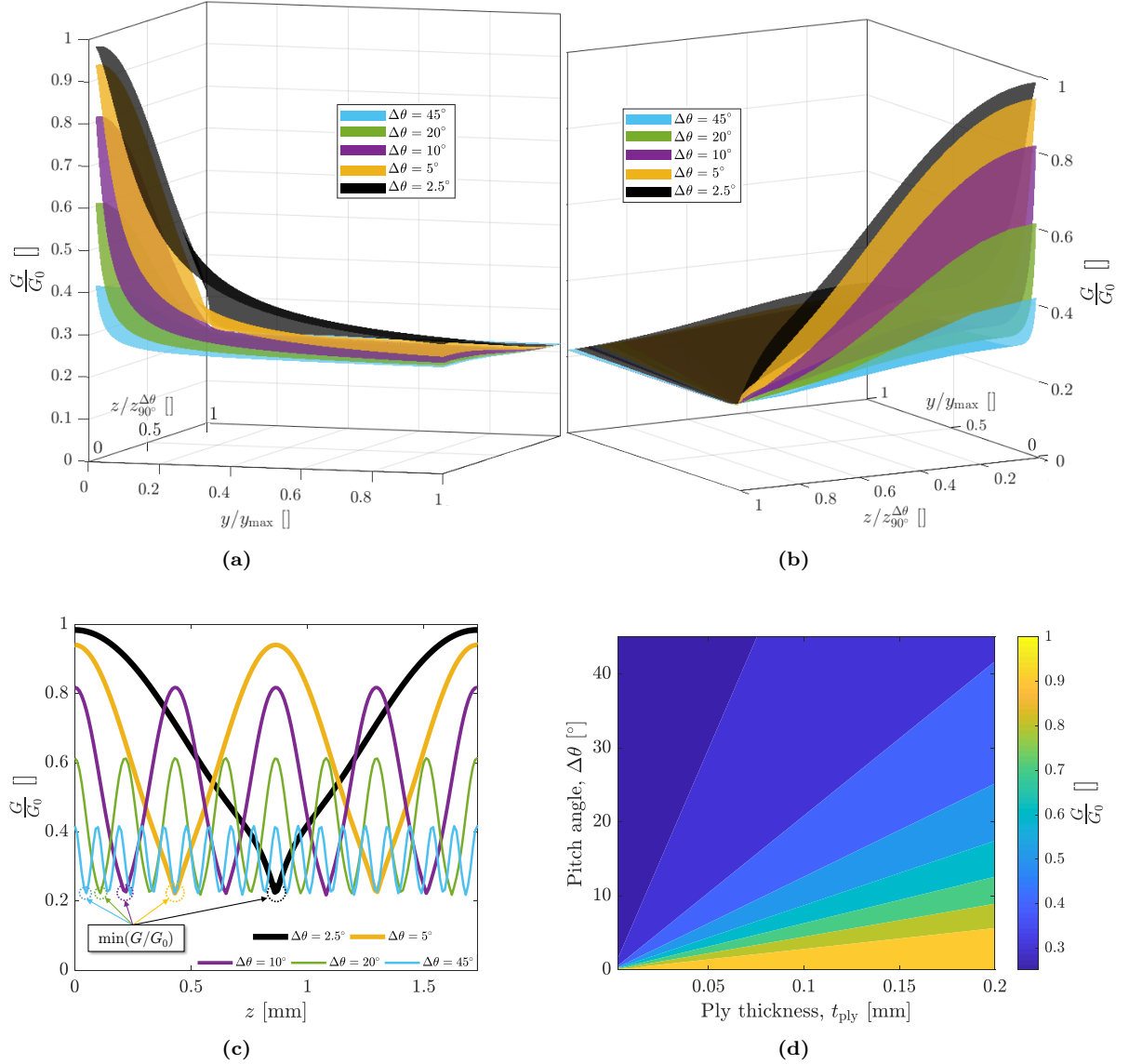


Figure 2.13: (a-b) Energy release rate maps (G/G_0) describing the crack growth of twisting cracks at each point (z, y) of the twisting crack path for the pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ, 45^\circ$ and $t_{ply} = 0.024$ mm. For each pitch angle $\Delta\theta$, z has been normalised to the value of z with minimum G/G_0 . y has been normalised to half of the crack length. (c) Energy release rate (G/G_0) vs. z evaluated at $y = 0.1$ mm for the pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ, 45^\circ$ and $t_{ply} = 0.024$ mm. (d) Distribution of energy release rate (G/G_0) at $(z, y) = (0, 1)$ as a function of pitch angle $\Delta\theta$ and ply thickness t_{ply} .

would more easily propagate towards the bulk of the laminate diffusing sub-critical damage at the back face of the laminate, as observed experimentally (Figure 2.5e).

The lower energy release rate for twisting cracks, i.e. higher twisting-crack resistance, shown by larger pitch angles (Figure 2.13a-2.13b) potentially leads to bending cracks which do not propagate towards the bulk of the laminate, leading to the anticipated activation of more

catastrophic mechanisms of failure such as delaminations and fibre breaks. This is in good agreement with the experimental observations conducted in this work where the configurations with pitch angles $\Delta\theta = 20^\circ$ and 45° showed localised large delaminations (Figures 2.5a and 2.5b), extensive back-face damage (Figure 2.6c and 2.6d) and anticipated catastrophic failure (load-drop in Figure 2.4).

Figure 2.13d shows G/G_0 as a function of $\Delta\theta$ and t_{ply} with $z = 0$ and $y = 1$ mm (the curves are qualitatively similar for other points along the crack path). The figure also shows that the energy release rate for twisting cracks increases with ply thickness (for a constant pitch angle $\Delta\theta$). This is in good agreement with other works in the literature on LVI tests of quasi-isotropic laminates (similar to the $\Delta\theta = 45^\circ$ configuration tested in this work) [29,30]; in these works, it is shown that, as t_{ply} increases (with interply angle and total laminate thickness kept constant), the damage in the bulk of the laminate and the one forming at the back face start interacting.

Based on these observations, we conclude that helicoidal cracks should propagate more easily from matrix splits at the distal face for lower pitch angles (as verified in the experiments) and for larger ply thicknesses.

2.4.4 The role of the pitch angle $\Delta\theta$ and the percentage of 0° -plies on the CAI residual properties

Figures 2.7a and 2.7b show that, by reducing the pitch angle $\Delta\theta$, similar residual compressive strength and failure strain can be achieved with a decreasing number of 0° -plies (i.e. with fewer plies aligned with the loading direction).

It is known that the CAI strength increases with the proportion of 0° -plies (i.e. with the proportion of plies aligned with the loading direction) [36,37]. However, Figures 2.7a and 2.7b show that, by reducing the pitch angle $\Delta\theta$, similar residual compressive strength and failure strain can be achieved with a decreasing number of 0° -plies (i.e. with fewer plies aligned with the loading direction). Specifically, we were able to preserve a similar CAI strength and failure strain while reducing the proportion of 0° -plies very significantly from 26% to 2.7%.

2.5 Conclusions

We investigated several bio-inspired Bouligand ultra-thin-ply CFRPs laminates with pitch angles ranging from $\Delta\theta = 2.5^\circ$ to 45° . We conducted LVI and CAI tests to study the influence of the pitch angle $\Delta\theta$ on the damage morphology and mechanical response of the laminate. The main conclusions of this study are here summarized.

Amongst the configurations with different pitch angles $\Delta\theta$ investigated in this work, the $\Delta\theta = 2.5^\circ$ configuration better mimicked the damage evolution of the Bouligand microstructure of the mantis shrimp's dactyl club, showing:

- enhanced impact resistance obtained via a significant diffused sub-critical damage accumulation before the peak load;
- a smooth and diffused double helicoidal pattern of delaminations;
- a delay in the occurrence of fibre failure at the distal face. This delay may be related to the formation of the double helicoidal delamination pattern; and
- a 13.3% higher peak load and a 34.9% larger displacement at load drop during impact with respect to the single $\Delta\theta = 45^\circ$ test.

In our tests, reducing the pitch angle from $\Delta\theta = 45^\circ$ to $\Delta\theta = 2.5^\circ$ leads to:

- a progressively smoother double helicoidal evolution of damage during impact;
- a more homogeneous distribution of delamination area through most interfaces during impact;
- a statistically-significant reduction of impact damage in the form of delamination, with a 29.4% decrease in total projected delamination area (p -value = 0.005), a 35.8% decrease in delamination extent (p -value = 0.007) and a 46.8% decrease in extent of back-face damage (p -value = 0.037); and
- similar residual properties both in terms of CAI strength (p -value = 0.11) and CAI failure strain (p -value = 0.1), achieved with a progressively smaller amount of 0° -plies (26% of plies aligned with the loading direction for $\Delta\theta = 45^\circ$ while only 2.7% for $\Delta\theta = 2.5^\circ$).

We created FE models of the biomimetic ultra-thin-ply CFRP laminates to analyse the role of the pitch angle $\Delta\theta$ on triggering different damage mechanisms, as observed experimentally. For this, the analysis of the intra-laminar shear stress τ_{23} allowed us to infer the distribution of shearing matrix cracks in laminates with different pitch angles $\Delta\theta$. Subsequently, we analysed the conditions for the formation of delaminations initiating from the matrix cracks focusing on the intra-laminar τ_{13} and τ_{23} shear stresses. The main conclusions are the following:

- the points in the laminate where shearing matrix cracks are predicted to initiate are distributed in a double helicoidal progressively better defined as the pitch angle $\Delta\theta$ decreases;
- the $\Delta\theta = 2.5^\circ$ configuration shows a higher maximum value of τ_{23} than any of the other configurations. This might have led to an anticipated formation of sub-critical damage in the $\Delta\theta = 2.5^\circ$ configuration (in the form of matrix cracks) resulting in the early loss of linearity observed in the experimental mechanical response;
- the synergistic effect between τ_{13} and τ_{23} in promoting delaminations decreases as the pitch angle decreases. This will have contributed to delaying delamination initiation for $\Delta\theta = 2.5^\circ$; and
- the $\Delta\theta = 2.5^\circ$ configuration shows similar maximum τ_{13} values for a large number of plies. This is expected to promote the failure of several interfaces, thereby generating diffused delamination damage (as experimentally observed in $\Delta\theta = 2.5^\circ$ laminates).

We analysed how the pitch angle $\Delta\theta$ and the ply thickness t_{ply} affect the resistance of Bouligand CFRP composites to growing twisting Bouligand-like cracks forming at the back face of the laminate (LVI tests) due to the tensile stresses originating from bending. The main conclusions are the following:

- by reducing the pitch angle $\Delta\theta$, the crack resistance (under dominant Mode I opening) to growing Bouligand cracks decreases, facilitating the propagation of sub-critical matrix cracks forming at the back-face towards the bulk of the laminate. This is in good agreement with the largely diffused sub-critical helicoidal damage observed in the $\Delta\theta = 2.5^\circ$ configuration;
- by increasing the pitch angle $\Delta\theta$, the crack resistance (under dominant Mode I opening) to growing Bouligand cracks increases, promoting the occurrence of catastrophic mech-

anisms of failure, such as delaminations and fibre breaks. This is in good agreement with the localised large delaminations, extensive fibre failure and the anticipated catastrophic failure observed in configurations with large pitch angles such as $\Delta\theta = 20^\circ$ and 45° .

In summary, in this work, we have developed a strong knowledge on the role of the pitch angle on the failure mechanisms of Bouligand microstructures. We have demonstrated that, with a tailored Bouligand design, it is possible to achieve an enhancement in damage resistance to impact via delayed fibre failure and increased load-bearing capability. While this enhancement was demonstrated only for a low impact energy, the full damage resistance capability to through-the-thickness loads of ultra-thin-ply Bouligand CFRPs has yet to be explored. Detailed progressive failure analyses of ultra-thin-ply Bouligand CFRPs aiming at:

- exploring the full-penetration behaviour (and thus the full spectrum of energy dissipation capability); and
- investigating the sequence of activation, the interaction and the growth of the failure mechanisms responsible for the increase in impact resistance,

is paramount to unlock the full potential of Bouligand microstructures in industrial applications. This will be addressed in [Chapter 3](#).

Chapter 3

Towards ultra-damage-resistant thin-ply CFRP Bouligand structures: progressive failure analyses

3.1 Introduction

The use of thin-ply Carbon Fibre Reinforced Plastic (CFRP) materials is of growing interest due to their high resistance to form matrix cracks and delaminations as well as their excellent in-plane mechanical properties [29, 30, 38–40]. However, they are very susceptible to through-the-thickness loading.

Yokozeki et al. [29, 40] compared Quasi-Static Indentation (QSI) tests of standard-ply-thickness (145 μm) quasi-isotropic CFRP laminates against similar thin-ply laminates (75 μm) showing that the thin-ply laminates were able to reduce the in-plane extent of delaminations but this in turn resulted in an anticipated fibre failure and in-plane localisation of damage. Wagih et al. [30] also observed premature fibre failure in QSI tests of thin-ply (40 μm) CFRP laminates which led to anticipated catastrophic failure compared to similar quasi-isotropic laminates with

a ply thickness of 80 μm . More recently, Garcia et al. [41] confirmed similar trends showing, with a full 3D tomographic investigation of damage obtained from impact and QSI tests, that while standard-ply-thickness (135 μm) CFRP laminates developed more delaminations and delayed fibre failure, in thin-ply (70 μm) laminates fibre failure was anticipated. The premature fibre failure led to a lower load-bearing capability and lower residual properties of the thin-ply laminates compared to the standard-ply-thickness ones. Therefore, research efforts towards improving the resistance to out-of-plane loading of thin-ply CFRP structures are of fundamental importance to fully exploit the potential of thin-ply materials for structural applications.

The investigation presented in [Chapter 2](#) showed that, for a low impact energy, a bio-inspired design based on Bouligand architectures with small pitch angles ($\Delta\theta$ down to 2.5°) can improve the resistance to through-the-thickness loads of thin-ply CFRP structures commonly used in engineering, resulting in a delay in the occurrence of fibre failure and in a slightly increased load-bearing capability.

However, three major aspects ought to be addressed in order to unlock the full potential of Bouligand microstructures for use in industrial applications as a solution to the high sensitivity to through-the-thickness loads of thin-ply CFRPs:

- demonstrate that a tailored thin-ply CFRP Bouligand composite can be capable of a simultaneous increase in performances in terms of maximum transverse load-bearing capability, delay in catastrophic fibre failure, and energy dissipation;
- explore the full-penetration behaviour (and the full spectrum of the energy dissipation capability) of thin-ply Bouligand CFRP structures; and
- provide an analysis that allows for the detailed observation of the sequence of activation, the interaction and the growth of the failure mechanisms responsible for the resistance to through-the-thickness loads of thin-ply Bouligand CFRP structures.

To this end, in this chapter we devised an original study to demonstrate that by reducing the pitch angle $\Delta\theta$ in ultra-thin-ply Bouligand CFRP structures to values mimicking the periodic region of the mantis shrimp's dactyl club, we are able to obtain such a simultaneous increase in structural performances. In particular, we demonstrate that the inherent low performance of thin-ply composites to transverse loading can be greatly improved via a tailored bio-mimetic

Bouligand design therefore greatly expanding the potential of thin-ply CFRP materials for structural applications.

To this end, we performed several in-situ damage analyses of the five ultra-thin-ply Bouligand and CFRP configurations analysed in [Chapter 2](#), under progressive QSI full-penetration tests and Three-Point Bending (3PB) tests conducted under a Scanning Electron Microscope (SEM). We revealed how the sequence of activation of different failure mechanisms depends on the pitch angle $\Delta\theta$ and how this affects the performances to out-of-plane loading of ultra-thin-ply Bouligand structures at the sub-critical (sub-peak), catastrophic (peak) and penetration (post-peak) stages of failure.

This chapter contains the following original contributions:

- (i) we show how, by reducing the pitch angle $\Delta\theta$ to values mimicking the natural microstructure, it is possible to achieve simultaneously a large increase in total dissipated energy, maximum load-bearing capability and displacement at fibre failure with respect to standard quasi-isotropic laminates;
- (ii) we propose a detailed description of the full penetration behaviour of ultra-thin-ply Bouligand laminates under transverse loading along with a discussion on the structural integrity of ultra-thin-ply CFRP Bouligand composites;
- (iii) we demonstrate the nesting and propagation of sub-critical Bouligand matrix cracks from the back face of ultra-thin-ply CFRP laminates and hence explain one of the most important failure mechanisms responsible for the high resistance to through-the-thickness loads of Bouligand microstructures with small pitch angles;
- (iv) using experimental observations in combination with an analytical model, we define an energy release rate threshold for the growth and arrest of sub-critical Bouligand matrix cracks; and
- (v) we show that the failure process progressively shifts from being dominated by fibre failure in configurations with large pitch angles to being characterised by extensive diffused sub-critical damage in configurations with small pitch angles.

3.2 Materials and methods

3.2.1 Manufacturing

The Bouligand-inspired samples used in this chapter were manufactured using the same materials and procedures described in §2.2.1. For clarity, we briefly report the steps of the manufacturing procedure in this section.

We used Skyflex USN20A, a unidirectional ultra-thin-ply prepreg tape (20gsm) to manufacture five different Bouligand configurations, with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° . The prepreg uses Mitsubishi Rayon TR30S 3K as reinforcement and SK Chemicals K50 as resin system. The physical properties of the constituents can be found in §2.2.1 (Table 2.3 and Table 2.4, respectively). Table 3.1 shows the lay-up sequences of the five configurations.

The manufacturing of the samples can be divided in the following steps: (i) we cut plies already at the specific angle using an automatic cutting table; (ii) we laid-up the plies with the support of an alignment mould (this ensured excellent alignment and repeatability); (iii) we cured all the samples in the same autoclave cycle following the material manufacturer's recommendations; (iv) we cut the cured plates to their final dimensions, i.e. 60 mm x 60 mm for QSI and 50 mm x 10 mm for 3PB, using a disc saw; and (v) we verified the quality of the samples using ultrasonic C-scans in an immersion tank.

Regarding the 3PB samples, we polished and gold-sputtered the lateral surfaces for better visualisation in the SEM. We cut the 3PB samples such that the top and bottom plies fibre direction, i.e. 0° -plies, were oriented along the width of the sample (orthogonal to the span

Table 3.1: Samples specifications for QSI and 3PB tests.

$\Delta\theta$	Stacking sequence	n°of samples	thickness [mm]	n°of plies
45°	$[0^\circ/45^\circ/90^\circ/\dots/3240^\circ]_s$	2	3.51 ± 0.01	146
20°	$[0^\circ/20^\circ/40^\circ/\dots/1440^\circ]_s$	2	3.52 ± 0.02	146
10°	$[0^\circ/10^\circ/20^\circ/\dots/720^\circ]_s$	2	3.60 ± 0.04	146
5°	$[0^\circ/5^\circ/10^\circ/\dots/360^\circ]_s$	2	3.57 ± 0.03	146
2.5°	$[0^\circ/2.5^\circ/5^\circ/\dots/180^\circ]_s$	3	3.56 ± 0.02	146

direction). This was necessary to investigate the effect of the pitch angle $\Delta\theta$ on the formation of sub-critical Bouligand matrix cracks from the back face of the laminates.

We manufactured a total of 11 samples per test type. For each test, we manufactured 3 nominally equivalent samples for the $\Delta\theta = 2.5^\circ$ and 2 nominally equivalent samples for the remaining configurations. We chose 3 samples for the $\Delta\theta = 2.5^\circ$ because it is the configuration that better mimics natural Bouligand microstructures and the most sensitive to misalignment errors in the pitch angle $\Delta\theta$.

3.2.2 Testing methods: QSI and 3PB

We conducted full-penetration QSI tests using a test rig which design was based upon the ISO 6603-2 standard [42]. Each square sample, 60 mm side and ≈ 3.5 mm thick, was clamped between two steel plates with a central circular hole 40 mm in diameter. The samples and test-rig geometries were designed to have a very similar span-to-thickness ratio (Γ) to the samples tested in 3PB ($\Gamma_{\text{QSI}} \approx 11.4$, $\Gamma_{\text{3PB}} \approx 10.2$). Therefore, it was possible to directly correlate the findings of the two tests. Additionally, we preferred a circular hole to other hole geometries to avoid introducing any preferential directionality in the test.

The QSI samples were loaded with a 50 kN load cell Instron machine at 1 mm/min displacement rate using an indenter with hemispherical tip 20 mm in diameter [42]. We set the acquisition time to 100 ms and used an Imetrum camera to record the displacement at the indenter tip, so that our measurements are not affected by the machine compliance. Two samples for $\Delta\theta = 2.5^\circ$ and one sample for each one of the other configurations were loaded continuously until full-penetration. One sample per configuration was tested using three step-loading/unloading cycles. The load was stopped (i) before the knee region of the load vs displacement curve (linear-elastic region), (ii) at the end of the knee region, i.e. after the first non-linear region, when the load increased again at roughly a constant rate, and (iii) at catastrophic failure (peak-load). Between each loading-unloading cycle, we analysed the damage using ultrasonic C-scans and visual inspections of the impact and back faces of the laminates. For all samples, we interrupted the test when the load was below the load-threshold of 1 kN.

We conducted 3PB tests in a SEM environment to progressively capture damage evolution during testing. The span between the two supporting pins was set to 36 mm. We used a

5 kN micro-tester and we set the loading rate and acquisition time to 0.2 mm/min and 500 ms, respectively. During the initial linear elastic loading, we interrupted the tests at equal load increments of 50 N. After onset of damage, we interrupted the tests at any relevant damage evolution.

3.3 Results

3.3.1 QSI test results

For the QSI tests, Table 3.2 shows the load at the knee point, the peak load, the penetration load, the displacement at the the peak load, the energy dissipated before the peak load and the total dissipated energy for each pitch angle $\Delta\theta$. Figure 3.1a shows the load vs displacement curves for all the samples tested under QSI. Figures 3.1b-3.1d respectively show the correlation between the pitch angle $\Delta\theta$ and the load at three specific instants during the test (knee point, peak and penetration), the displacement at the peak load and the dissipated energy (before the peak-load and total).

Specifically, for each pitch angle $\Delta\theta$ the knee point indicates the beginning of the knee region (non-linear region in the load vs displacement curves of Figure 3.1a) which corresponds

Table 3.2: Mean and max/min values of displacement at peak load, dissipated energy before the peak load, total dissipated energy, load at knee point, peak load and load at penetration for the samples tested under QSI.

$\Delta\theta$	Displacement at peak load, δ_{peak} [mm]	Dissipated energy before peak load [J]	Total dissipated energy [J]
45°	$1.92^{+0.118}_{-0.118}$	$5.80^{+0.492}_{-0.492}$	$27.83^{+0.875}_{-0.875}$
20°	$2.56^{+0.080}_{-0.080}$	$9.38^{+0.266}_{-0.266}$	$37.37^{+1.560}_{-1.560}$
10°	$2.97^{+0.141}_{-0.141}$	$12.46^{+1.230}_{-1.230}$	$45.74^{+0.743}_{-0.743}$
5°	$3.21^{+0.028}_{-0.028}$	$13.54^{+0.317}_{-0.317}$	$48.86^{+0.688}_{-0.688}$
2.5°	$3.35^{+0.071}_{-0.114}$	$15.36^{+1.870}_{-1.779}$	$54.91^{+4.049}_{-3.923}$
$\Delta\theta$	Knee load [kN]	Peak load [kN]	Penetration load [kN]
45°	$3.984^{+0.043}_{-0.043}$	$4.683^{+0.044}_{-0.044}$	$3.631^{+0.107}_{-0.107}$
20°	$3.524^{+0.022}_{-0.022}$	$6.405^{+0.258}_{-0.258}$	$3.856^{+0.031}_{-0.031}$
10°	$3.325^{+0.048}_{-0.048}$	$7.588^{+0.916}_{-0.916}$	$3.841^{+0.177}_{-0.177}$
5°	$2.924^{+0.030}_{-0.030}$	$8.690^{+0.206}_{-0.206}$	$4.915^{+0.016}_{-0.016}$
2.5°	$2.550^{+0.060}_{-0.030}$	$8.970^{+0.498}_{-0.384}$	$5.257^{+0.053}_{-0.331}$

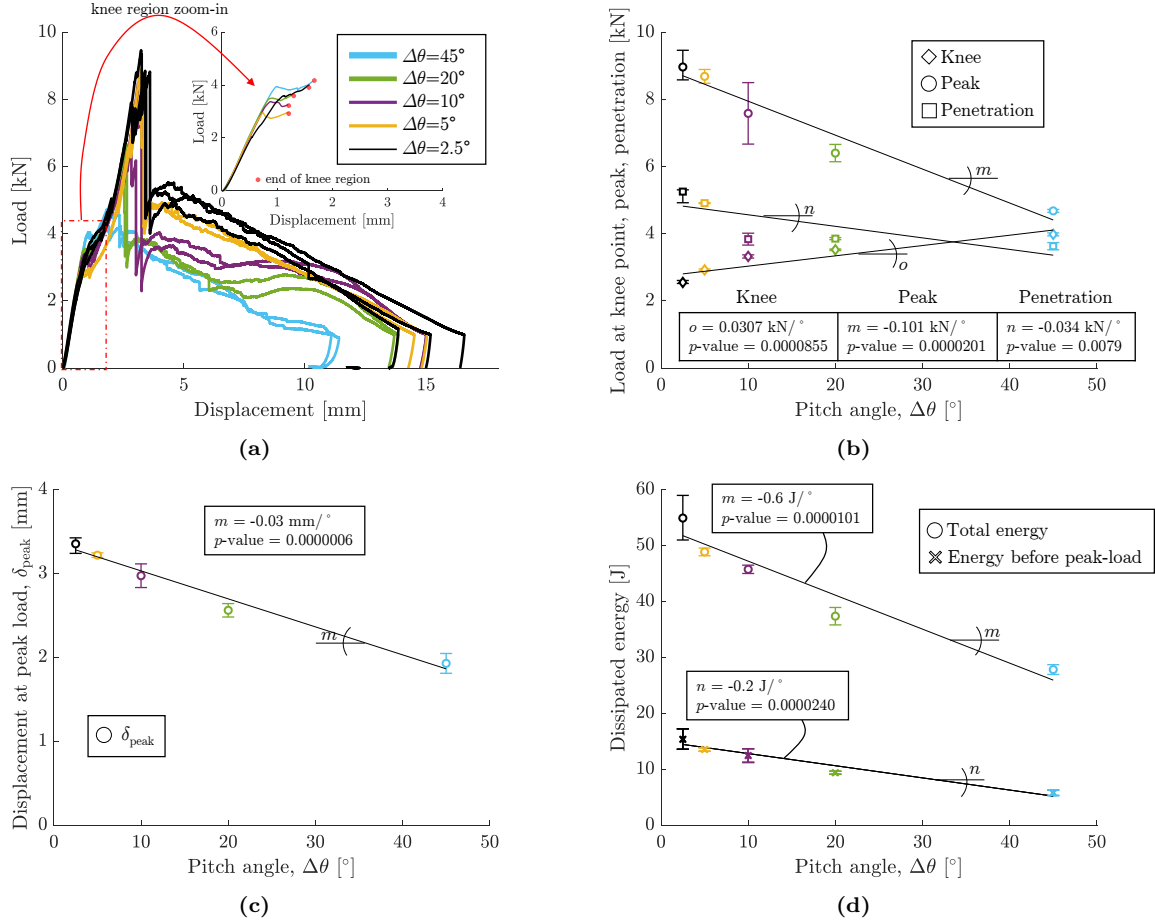


Figure 3.1: (a) Load vs displacement curves of the samples with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° tested under QSI. Effect of the pitch angle $\Delta\theta$ on (b) peak load, (c) displacement at peak load (critical fibre failure) and (d) dissipated energy.

to a 10% change in the slope of the load vs displacement curve with respect to the slope of the initial linear-elastic region. The end of the knee region corresponds to the load rising again at a constant rate. The penetration load corresponds to the maximum load reached after catastrophic failure has occurred (large load-drop in the load vs displacement curves in Figure 3.1a).

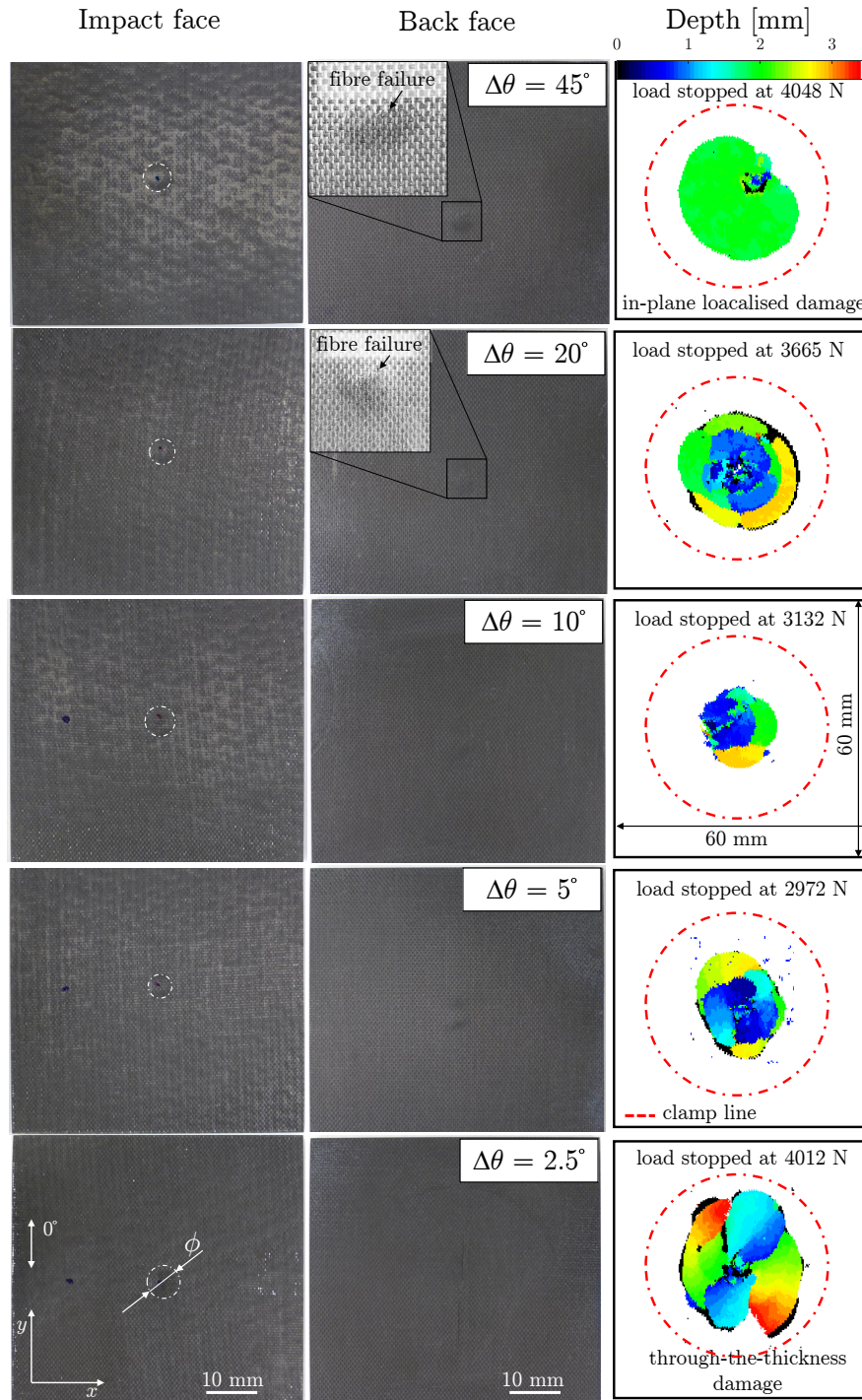
We used a least-square linear regression model to correlate the pitch angle $\Delta\theta$ to the load at the knee point, peak load, penetration load, displacement at peak load and dissipated energies (before-peak-load and total). In order to assess the statistical significance of the slope of each trendline, we calculated the associated p -value. We set the threshold $p \leq 0.05$ (as commonly used in the literature) to decide whether the correlations between the pitch angle $\Delta\theta$ and the measured quantity (i.e. slope of the trendline) were statistically significantly different from zero.

Figure 3.1 shows that reducing the pitch angle $\Delta\theta$ led to a statistically-significant:

- decrease in the load-threshold for the onset of sub-critical damage (load at knee point) — 36% smaller for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$;
- increase in the peak load — 92% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$;
- increase in penetration load — 45% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$;
- increase in the ‘warning region’, i.e. increased separation between knee and peak loads;
- increase in displacement at the load-drop (delay in catastrophic fibre failure) — 74% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$;
- increase in the sub-critical dissipated energy (energy dissipated before the peak load) — 165% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$; and
- increase in total dissipated energy — 97% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$.

Figure 3.2 shows photographs of the impact surface, back-face surface and delamination damage obtained via ultrasonic C-scans of representative samples for each pitch angle $\Delta\theta$. The images were taken at (Figure 3.2a) the end of the knee region and (Figure 3.2b) immediately after catastrophic failure had occurred (large load-drop in Figure 3.1a). The coloured areas in the C-scan images correspond to delaminations at a certain depth (see colour bar located on the top of each figure). The colour bar used is common to all the samples tested since the probing parameters were kept constant across samples. For any of the samples tested, no delamination damage was observed in the linear-elastic region (before the knee region). Additionally, while after the knee region (Figure 3.2a) delaminations have not reached the clamp line yet, after catastrophic failure (Figure 3.2b) delaminations reached the clamp which restrained them from a further in-plane extension.

Figure 3.3 shows photographs of the back face of the samples at the end of the test (full-penetration). The figure shows that while ply-fracture was localised on three main fracture planes for samples with pitch angles $\Delta\theta = 45^\circ$, 20° and 10° , leading to the formation of three large lips, damage was progressively more diffused in samples with $\Delta\theta = 5^\circ$ and 2.5° .



(a)

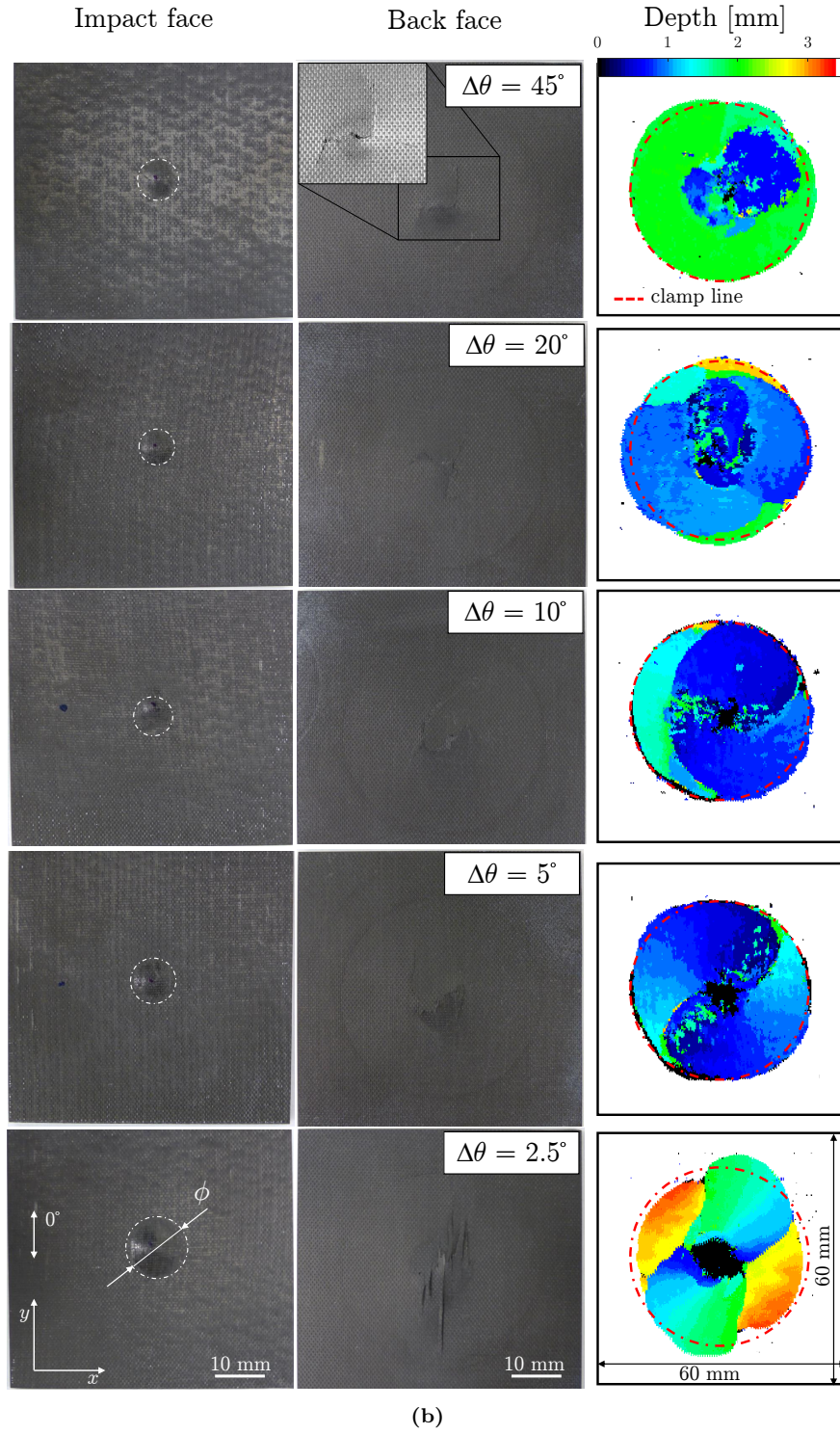


Figure 3.2: Photographs of the impact surface, back face surface and delamination damage obtained via ultrasonic C-scans of samples with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° taken (a) at the end of the knee region and (b) after catastrophic failure has occurred (load-drop). The red dotted line in the C-scan images indicates the presence of the clamp line.

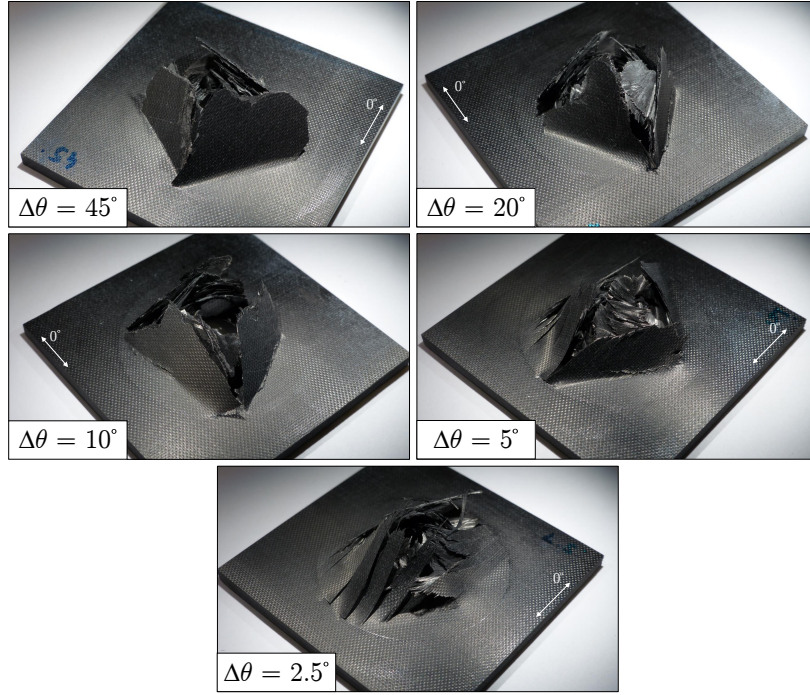


Figure 3.3: Photographs of the back face of samples representative of configurations with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° taken at the end of the QSI tests.

3.3.2 3PB test results

Figure 3.4a shows the load vs displacement curves for all the samples tested under 3PB. Specifically, the figure shows that, by reducing the pitch angle $\Delta\theta$, the maximum load reached during the test decreases (see Figure 3.4b). However, after the onset of damage, the load bearing capability increases as well as the displacement at final failure — 130% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$ (Figure 3.4b). Figure 3.4b shows the correlation between the energy dissipated during the test and the pitch angle $\Delta\theta$. Overall, the $\Delta\theta = 2.5^\circ$ samples dissipated 77% more energy on average than the $\Delta\theta = 45^\circ$ samples. The average values of peak load, displacement at final failure (ultimate displacement, δ) and total dissipated energy for each configuration with the associated maximum and minimum values are reported in Table 3.3.

Figure 3.5 shows the SEM images of the progressive failure of representative samples of configurations with pitch angle $\Delta\theta = 45^\circ, 20^\circ, 10^\circ, 5^\circ$ and 2.5° . For each figure, along with the SEM images, we report the load vs displacement curve of the sample tested with indications of the instants at which the test was interrupted for image capturing. The red arrows in the load vs displacement curves refer to load-drops due to relaxation following the interruption of

the test. The discontinuities which are not referred with red arrows correspond to unstable damage propagation during test.

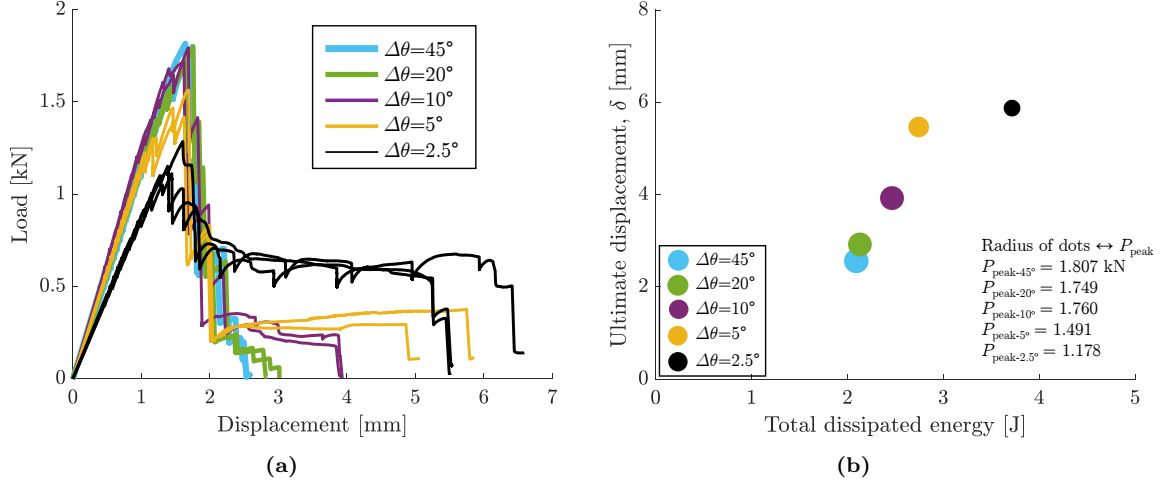
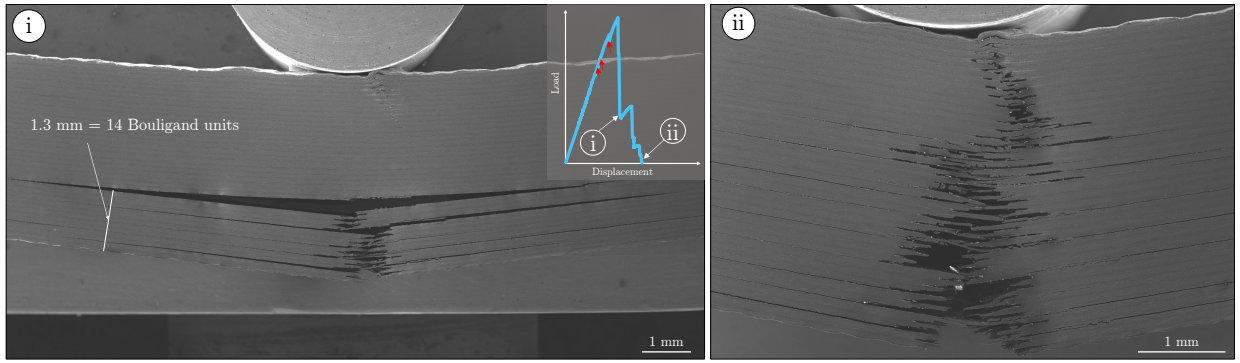


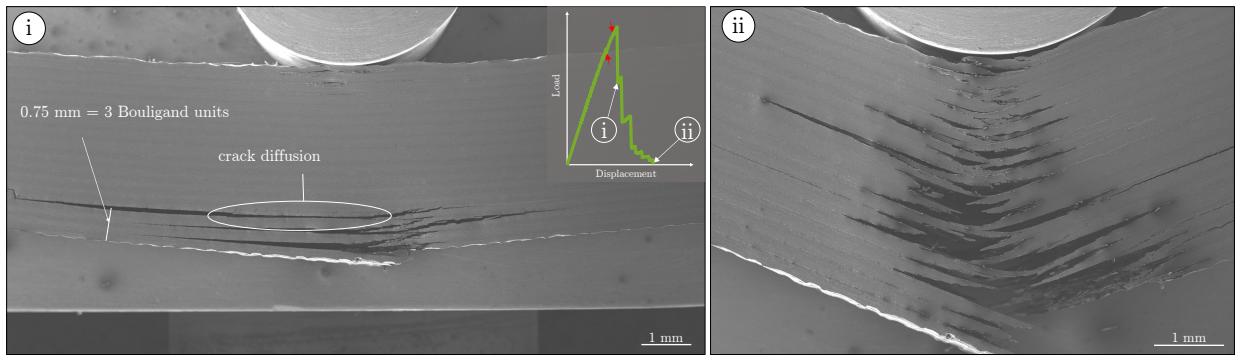
Figure 3.4: (a) Load vs displacement curves of the samples with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° tested under 3PB. (b) Average ultimate displacement (final failure) vs average total dissipated energy for each $\Delta\theta$ configuration; the radius of each dot in the graph represents the average maximum load reached during the 3PB test.

Table 3.3: Mean and max/min values of peak load, displacement at final failure and dissipated energy for the samples tested under 3PB.

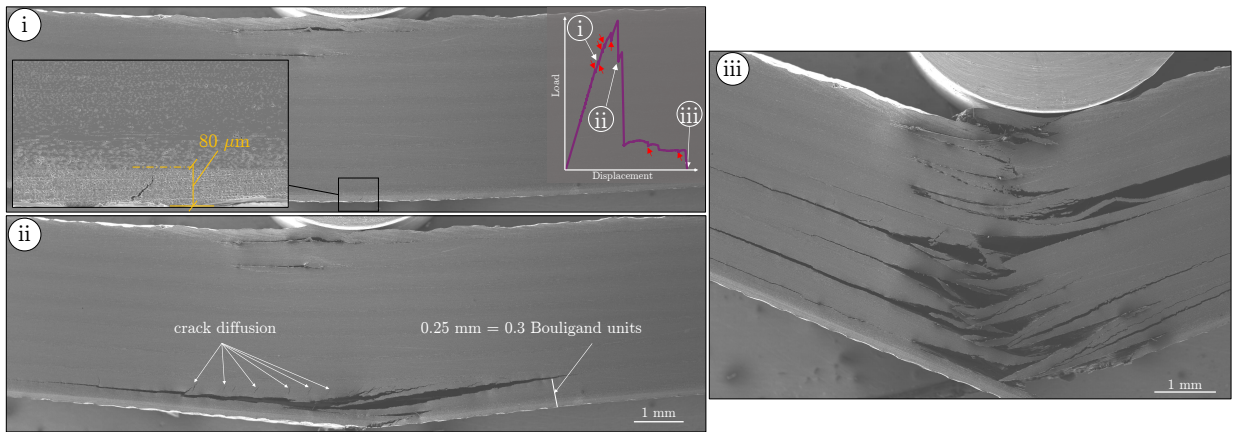
$\Delta\theta$	Peak load [kN]	Ultimate displacement, δ [mm]	Total dissipated energy [J]
45°	$1.807^{+0.009}_{-0.009}$	$2.64^{+0.016}_{-0.016}$	$2.09^{+0.024}_{-0.024}$
20°	$1.748^{+0.050}_{-0.050}$	$2.99^{+0.092}_{-0.092}$	$2.13^{+0.046}_{-0.046}$
10°	$1.760^{+0.034}_{-0.034}$	$3.98^{+0.007}_{-0.007}$	$2.46^{+0.032}_{-0.032}$
5°	$1.491^{+0.072}_{-0.072}$	$5.53^{+0.398}_{-0.398}$	$2.74^{+0.146}_{-0.146}$
2.5°	$1.178^{+0.107}_{-0.060}$	$5.92^{+0.669}_{-0.389}$	$3.72^{+0.471}_{-0.283}$



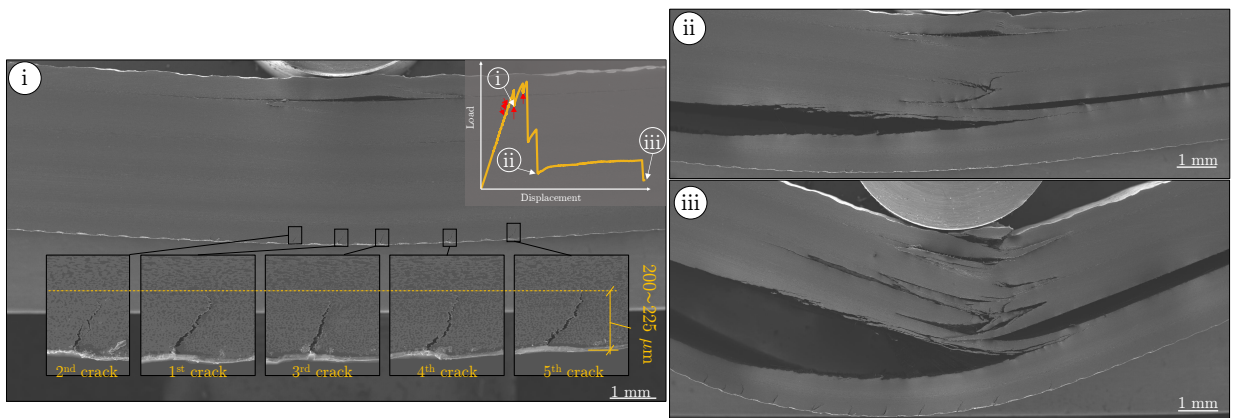
(a) $\Delta\theta = 45^\circ$



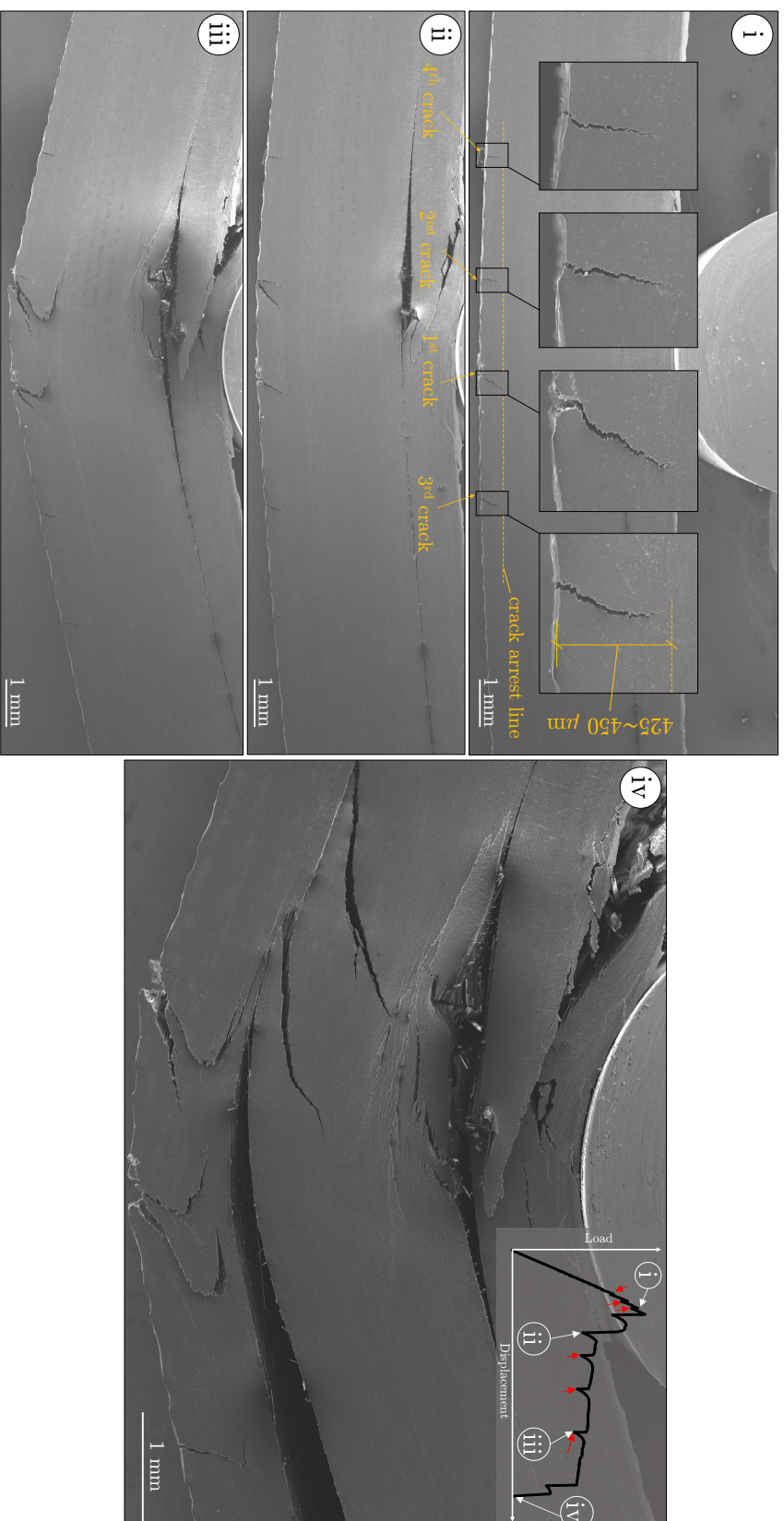
(b) $\Delta\theta = 20^\circ$



(c) $\Delta\theta = 10^\circ$



(d) $\Delta\theta = 5^\circ$



(e) $\Delta\theta = 2.5^\circ$

Figure 3.5: Reducing the pitch angle $\Delta\theta$ leads to a progressive transition from a brittle failure dominated by fibre failure to sub-critical diffused damage.

3.4 Discussion

3.4.1 Realising ultra-thin-ply CFRP structures highly resistant to out-of-plane loads

3.4.1.1 The sub-critical accumulation of damage in ultra-thin-ply Bouligand CFRP structures

As the pitch angle $\Delta\theta$ decreases, the onset of damage corresponding to the initial loss of linearity in the load vs displacement curves of Figure 3.1a (beginning of the knee region) occurs at lower applied loads. This allows configurations with decreasing pitch angles to develop damage at a sub-critical level in the form of: (i) a progressively better-defined helicoidal distribution of delaminations, diffused through-the-thickness of the laminate (Figure 3.2a); (ii) an increasing extent of matrix splits at the back face of the laminate (Figure 3.2a); and (iii) decreasing fibre breaks (Figure 3.2a).

Specifically, at the end of the knee region, we observed fibre breaks at the back face of the $\Delta\theta = 45^\circ$ and 20° samples, no damage in the $\Delta\theta = 10^\circ$ and 5° samples and one sub-critical matrix split (Bouligand matrix crack) in the $\Delta\theta = 2.5^\circ$ sample. The extensive accumulation of sub-critical damage in the $\Delta\theta = 2.5^\circ$ configuration resulted in a smooth knee region compared to the other configurations, characterised by sharp transitions in stiffness (Figure 3.1a). At this stage of the test, the delamination front has not reached yet the clamp line (Figure 3.2a). Therefore, we can conclude that the presence of the clamp did not significantly affect the delamination distribution in the samples.

Given the small span-to-thickness ratio ($\Gamma = 11.4$) used in the QSI tests, shear stresses developing in the laminate played a major role in the formation of sub-critical damage in the bulk of the laminate. In § 2.4.1 we showed via stress fields obtained from FE models of biomimetic thin-ply CFRP laminates, that the intra-laminar shear stress τ_{23} can be used to infer the distribution of shearing matrix cracks in laminates with different pitch angles $\Delta\theta$. Additionally, we analysed the conditions for the formation of delaminations initiating from the matrix cracks via the intra-laminar τ_{13} and τ_{23} shear stresses. The results of these analysis showed that, for laminates with small pitch angles, shearing matrix cracks are predicted to initiate in points in the laminate distributed helicoidally in the thickness direction.

The analysis of the intra-laminar shear stress τ_{13} revealed that in configurations with small pitch angles $\Delta\theta$, such as $\Delta\theta = 2.5^\circ$, the maximum τ_{13} values are distributed more uniformly through-the-thickness of the laminate (compared to large pitch angle laminates). This was expected to promote the failure of several interfaces, hence leading to diffused helicoidal delamination damage, as observed experimentally also in the QSI tests (see Figure 3.2). On the contrary, configurations with large pitch angles $\Delta\theta$, such as $\Delta\theta = 45^\circ$, showed a narrower through-the-thickness distribution of maximum τ_{13} values, with higher values reached at the mid-plane of the laminate. This was expected to promote the localisation of delamination damage at specific ply interfaces. This was also observed experimentally in the QSI tests (see Figure 3.2).

3.4.1.2 The catastrophic failure of ultra-thin-ply Bouligand CFRP structures

As the pitch angle $\Delta\theta$ decreases, the formation and propagation of diffused sub-critical damage progressively extending throughout the entire thickness of the laminates acted as a stress-relief mechanism for the tensile side. Therefore, the occurrence of catastrophic fibre failure was greatly delayed — displacement at peak load 74% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$; and the load-bearing capability of the composite was greatly increased — peak load 92% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$. The extensive sub-critical damage accumulation resulted in a large increase in energy dissipation at sub-critical levels, i.e. before the peak load — 165% larger for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$.

The inability of configurations with large pitch angles $\Delta\theta$ of diffusing sub-critical damage led to damage localisation, hence anticipating catastrophic failure which occurred at progressively smaller load increments after the knee region (see Figure 3.1b) as the pitch angle $\Delta\theta$ increases.

In all samples, the through-the-thickness distribution of delaminations did not significantly change between the knee region (Figure 3.2a) and the instant at which catastrophic failure occurred (Figure 3.2b). Therefore, the presence of the clamp did not significantly alter the through-the-thickness damage distribution, where the latter plays a fundamental role on the delay in catastrophic failure observed in configurations with decreasing pitch angles. Additionally, all the samples were subjected to the same boundary conditions. Therefore, a direct comparison between the samples tested is still suitable.

3.4.1.3 The full-penetration behaviour of ultra-thin-ply Bouligand CFRP structures

By reducing the pitch angle $\Delta\theta$, the load at which penetration occurred progressively increased (Figures 3.1a and 3.1b). As reported in Figure 3.2b, at the beginning of penetration the $\Delta\theta = 2.5^\circ$ sample preserved the helicoidal distribution of delamination damage, with relatively small propagation of the local delamination areas at a certain ply-interface. Additionally, fibre failure at the back face of the laminate was accompanied by the formation of several Bouligand and matrix cracks. Therefore, sub-critical damage could further diffuse in the laminate during penetration.

On the contrary, in configurations with large pitch angles, such as $\Delta\theta = 20^\circ$ and 45° , we could observe extensive propagation of the local delamination areas at few ply-interfaces (Figure 3.2b). Additionally, no Bouligand cracks formed at the back face of the laminates. Instead, damage localised on three main fracture planes, therefore reducing both the load-bearing capability and the energy dissipation capability during penetration.

The analysis of the back-face of the samples at the end of the penetration phase reported in Figure 3.3 reveals highly diffused damage in the $\Delta\theta = 2.5^\circ$ samples with the ply-fracture following a twisted helicoidal pattern. This phenomenon might be related to the helicoidal distribution of delaminations which, in combination with the small pitch angle ($\Delta\theta = 2.5^\circ$), allows fibres to locally re-orient, offering a mechanism of resistance to penetration. Therefore, by reducing the pitch angle, we obtained a progressively more damage-resistant highly-dissipative post-peak behaviour characterised by a large total dissipated energy — 97% larger in $\Delta\theta = 2.5^\circ$ than $\Delta\theta = 45^\circ$.

Overall, by decreasing the pitch angle $\Delta\theta$ to values mimicking natural Bouligand microstructures, it is possible to overcome the inherent sensitivity of thin-ply composite materials to out-of-plane loading. As shown in Figure 3.6, this was achieved via a simultaneous significant increase in (i) maximum load-bearing capability (92%), (ii) delay in catastrophic failure (74%) and (iii) energy dissipation (97%).

For small pitch angles configurations, the extensive sub-critical damage diffused during the entire failure process. This, along with the twisted ply-fracture pattern, allowed small pitch angle configurations to preserve a high structural integrity during the entire fracture process.

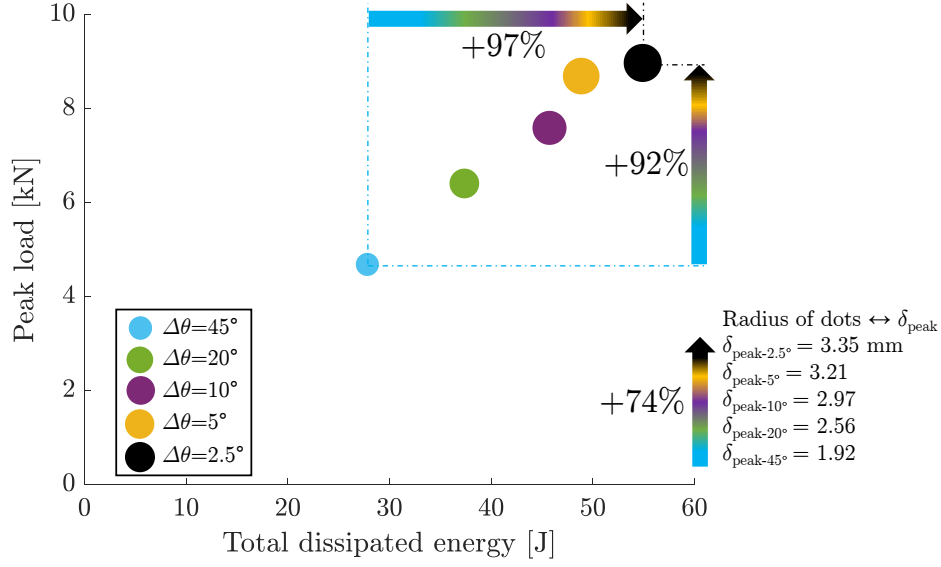


Figure 3.6: Reducing the pitch angle $\Delta\theta$ down to 2.5° leads to a simultaneous increase in maximum load-bearing capability (Peak load), dissipated energy and displacement at catastrophic failure (δ_{peak}).

The statistically-significant trendlines presented in this work suggest that, with ultra-thin-ply CFRP composites ($t_{\text{ply}} = 24 \mu\text{m}$), the smaller the pitch angle $\Delta\theta$ the larger the enhancement in damage resistance and structural integrity to out-of-plane loads.

3.4.2 A progressive failure analysis revealing the formation and propagation of Bouligand matrix cracks in ultra-thin-ply CFRP Bouligand structures

3.4.2.1 The role of the pitch angle $\Delta\theta$ on the propagation of sub-critical compression damage under 3PB

For each representative sample with pitch angle $\Delta\theta$, Figure 3.7 shows the formation of compressive failure during 3PB testing in the upper half of the sample. Similarly, for all samples, compression damage formed at a sub-critical level via small helicoidal delaminations neighbouring the 90° -plies.

Compression damage propagated further via the local micro-buckling of the delaminated area resulting into small drops in the load vs displacement curves (see Figure 3.7). Damage

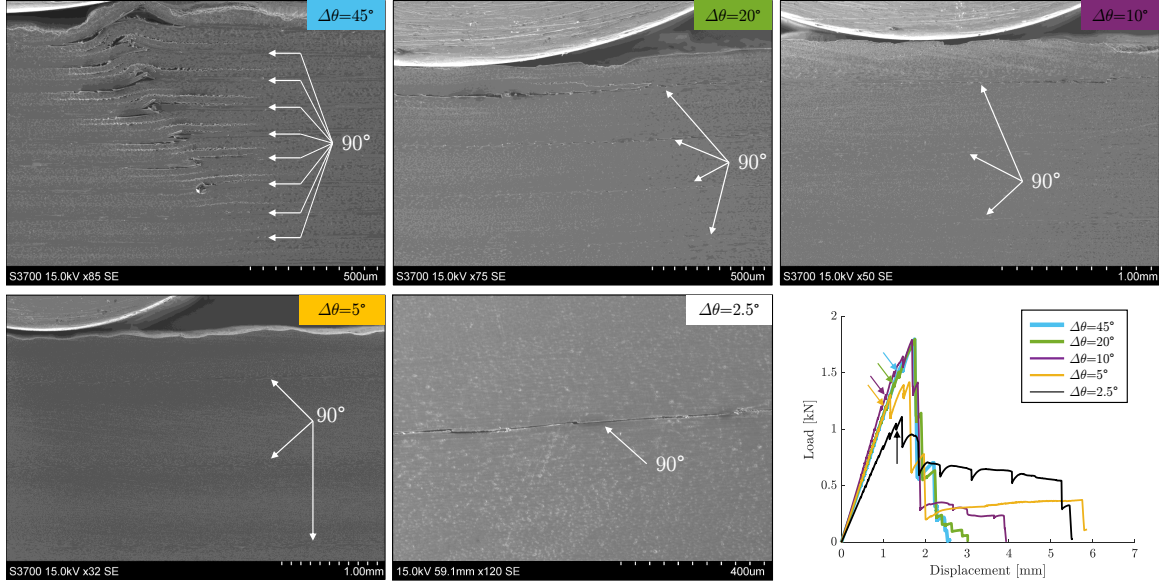


Figure 3.7: SEM images highlighting the sub-critical compression damage forming on the upper half of representative samples with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° .

in the micro-buckled areas did not propagate further, therefore not significantly affecting the damage evolution in the tensile side and the final failure of the samples.

3.4.2.2 Investigation of sub-critical Bouligand matrix cracks

In § 3.4.1, we observed that the formation of sub-critical damage in the form of helicoidal distributed delaminations and back-face Bouligand matrix cracks highly contributed to the enhancement in damage resistance of the configuration with $\Delta\theta = 2.5^\circ$.

The role of the pitch angle $\Delta\theta$ on the formation of helicoidal delamination damage in the bulk of ultra-thin-ply CFRP Bouligand laminates has been previously investigated experimentally and numerically in Chapter 2. In the next sections, by means of the 3PB tests results and detailed analytical modelling, we will present a combined experimental/analytical investigation of the role of the pitch angle $\Delta\theta$ on how damage forms and propagates from the back face of the laminate. We will explain the highly dissipative sub-critical mechanisms of failure responsible for the high damage resistance of small pitch angles ultra-thin-ply CFRP Bouligand structures.

3.4.2.3 $\Delta\theta = 45^\circ, 20^\circ$ and 10° : from dominant unstable fibre failure to delamination damage

For $\Delta\theta = 45^\circ, 20^\circ$ and 10° , Figures 3.5a, 3.5b and 3.5c show that, under 3PB, failure propagated catastrophically from the tensile side with no ($\Delta\theta = 45^\circ$ and 20°) or very little ($\Delta\theta = 10^\circ$) formation of sub-critical damage before the occurrence of unstable damage propagation. Damage on the compression side (for a detailed description refer to §3.4.2.1) formed and propagated in a stable manner and did not influence the global failure of any of the configurations tested in this work.

For $\Delta\theta = 45^\circ$, Figure 3.8 shows that fibre failure was the predominant failure mechanism. The $\Delta\theta = 45^\circ$ configuration was not able to diffuse damage at a sub-critical level, hence leading to the localisation of the fracture process. This is consistent with the analysis of the QSI test results (see §3.4.1).

For $\Delta\theta = 20^\circ$, Figures 3.5b and 3.9 show that although fibre failure was the predominant failure mechanism, crack twisting and delamination damage along with the formation of diffused small matrix cracks contributed to the failure process. The activation of these additional dissipative mechanisms led to an initial unstable crack propagation depth 42% smaller on average than the one observed in the $\Delta\theta = 45^\circ$ configuration.

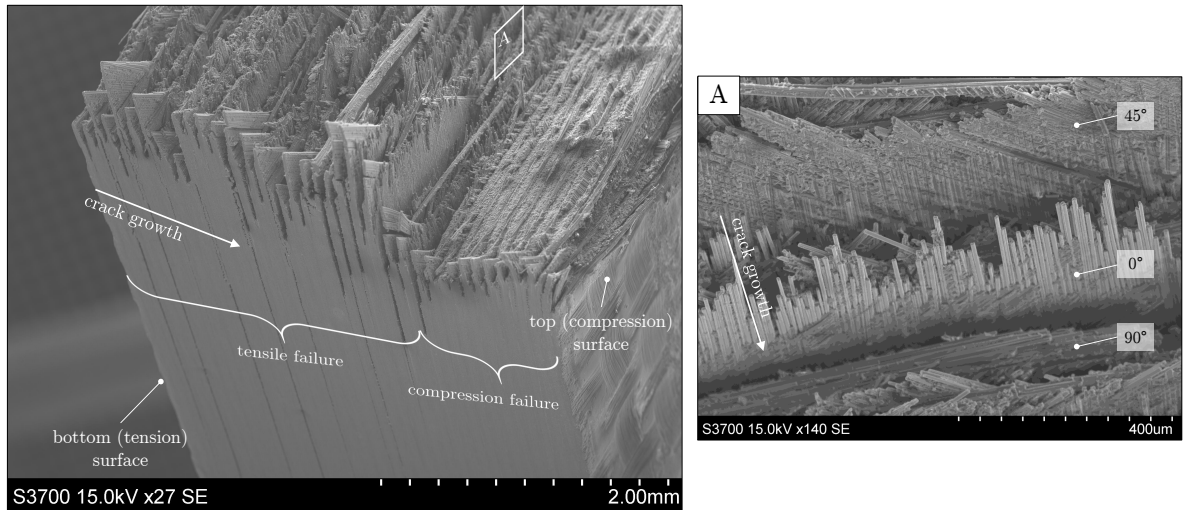


Figure 3.8: SEM image of the fracture surface of half a sample with pitch angle $\Delta\theta = 45^\circ$ after 3PB test. The image highlights that two regions of the sample which failed respectively in tension and compression were dominated by fibre failure with very little inter-laminar damage.

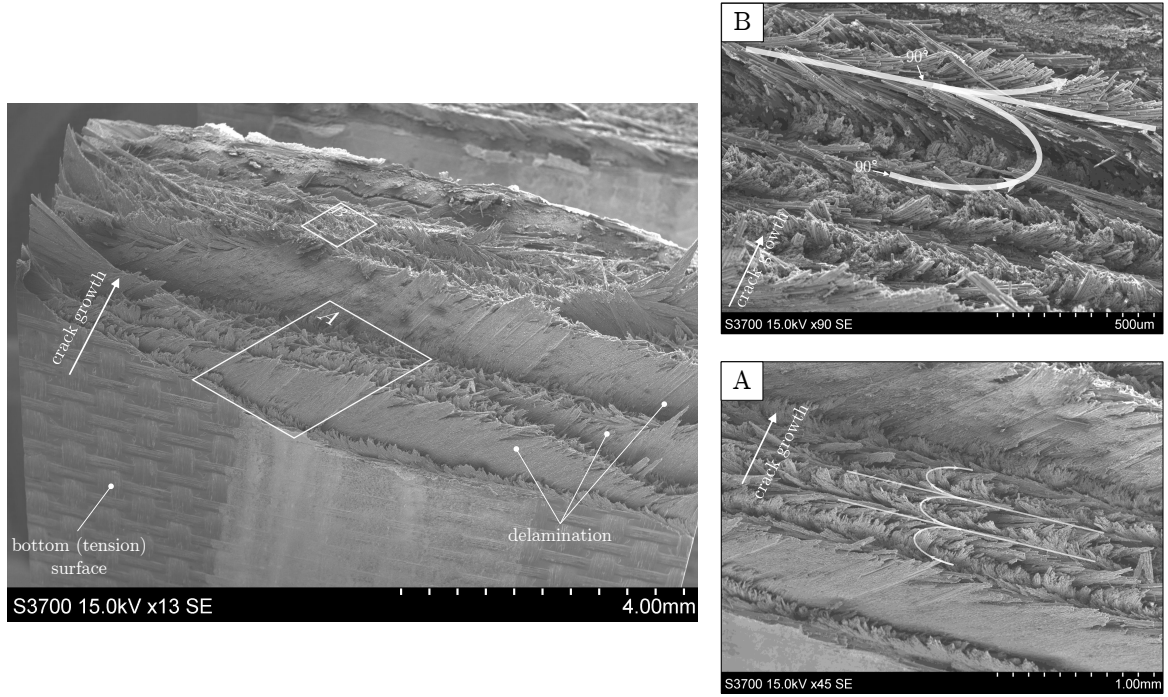


Figure 3.9: SEM image of the fracture surface of half a sample with pitch angle $\Delta\theta = 20^\circ$. Failure was dominated by fibre failure with the formation of a few delaminations.

By further reducing the pitch angle to $\Delta\theta = 10^\circ$, the presence of crack twisting (Figure 3.5c), delamination damage (Figure 3.10) and diffused small matrix cracks (Figure 3.5c) increased. Consequently, the initial crack-jump depth was further reduced — 80% smaller for $\Delta\theta = 10^\circ$ than for $\Delta\theta = 45^\circ$. Despite the large reduction in maximum load-bearing capability (85% on average with respect to the peak load) due to the initial unstable damage growth dominated by fibre failure (Figure 3.10), a short plateau in the load vs displacement curve (Figure 3.5c) before final failure indicates stable damage propagation in the $\Delta\theta = 10^\circ$ laminates.

Additionally, as shown in Figure 3.11 delaminations evolved following the helicoidal arrangement of the microstructure. This observation is consistent with the results obtained in the QSI tests (see Figure 3.2).

To summarise, reducing the pitch angle $\Delta\theta$ from 45° to 10° led to a progressively more stable failure process, less dominated by fibre failure, with an increasing predominance of helicoidal delamination, crack twisting and sub-critical damage.

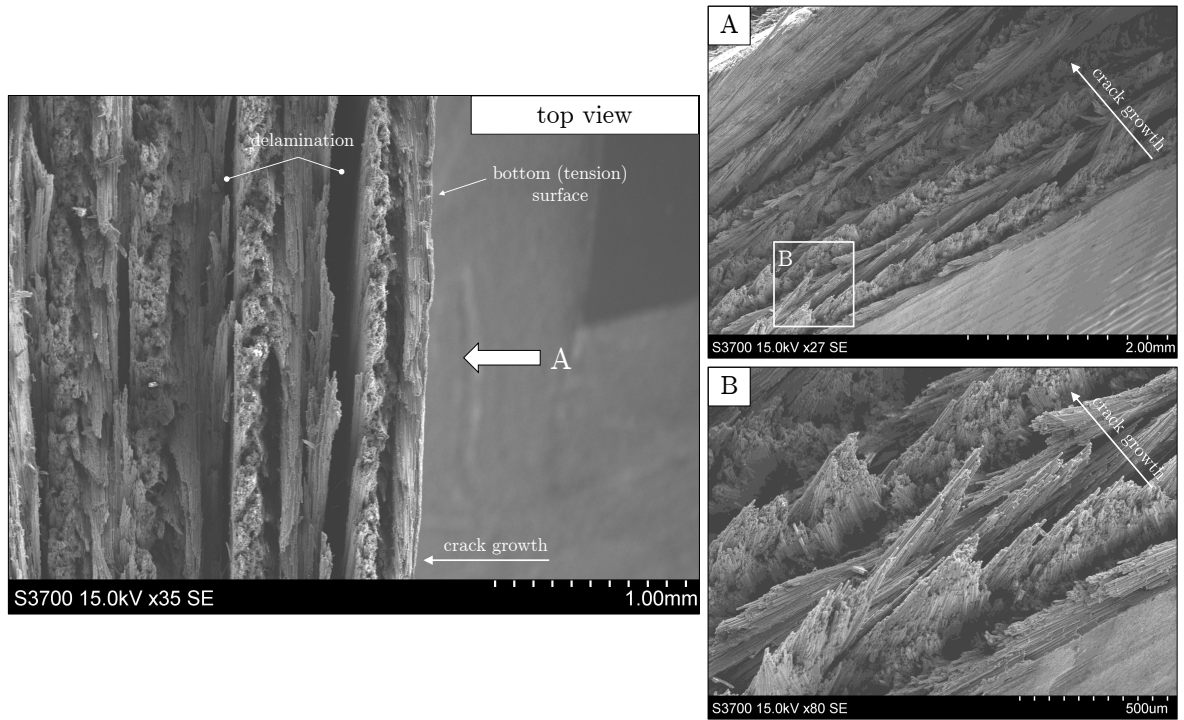


Figure 3.10: SEM image (top view) of the fracture surface of half a sample with pitch angle $\Delta\theta = 10^\circ$. Damage was characterised by fibre failure and delaminations.

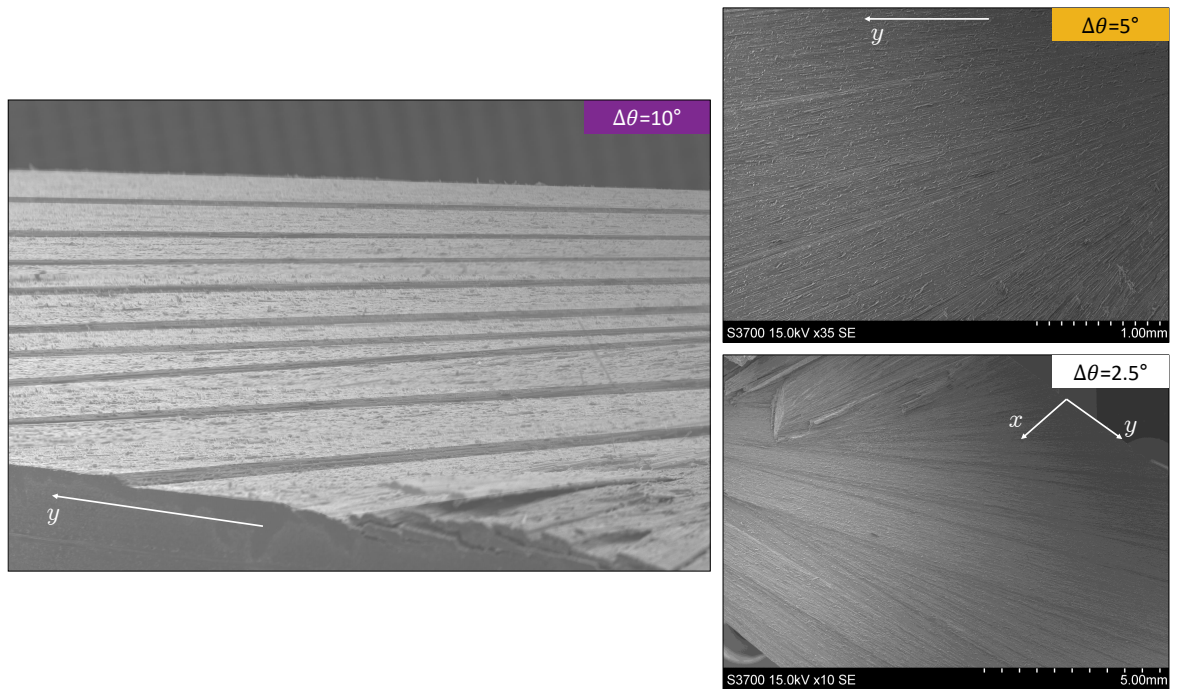


Figure 3.11: SEM images of the delaminated regions of samples with pitch angles $\Delta\theta = 10^\circ$, 5° and 2.5° . Delaminations were distributed helicoidally, following the fibre orientation dictated by the relative pitch angle $\Delta\theta$.

3.4.2.4 $\Delta\theta = 5^\circ$ and 2.5° : from delamination to extensive sub-critical propagation of damage

For $\Delta\theta = 5^\circ$, Figure 3.5d (instant (i)) shows that several sub-critical Bouligand matrix cracks formed on the tensile side of the laminate. For all the samples tested, the Bouligand matrix cracks formed sequentially; firstly, a crack under the pin (maximum tensile bending stress) formed, grew and arrested. The crack arrest mechanism allowed for the stress to redistribute, promoting crack nesting, thereby leading to damage dispersion on the tensile side. The formation of sub-critical Bouligand matrix cracks was followed by the unstable propagation of helicoidal delaminations (Figure 3.11) which formed at different ply interfaces (Figure 3.5d, instant (ii)). Even though the unstable growth of delamination damage in the $\Delta\theta = 5^\circ$ configuration led to a large reduction in load bearing capability (75% on average), the formation of delocalised damage and the limited occurrence of fibre breaks allowed the $\Delta\theta = 5^\circ$ samples to sustain load up to large applied displacements (extended load plateau in Figure 3.5d) before final failure.

Figure 3.5e (instant (i)) shows that reducing the pitch angle to $\Delta\theta = 2.5^\circ$ led to an initial failure similar to the one observed in the $\Delta\theta = 5^\circ$ samples, hence characterised by several sub-critical Bouligand matrix cracks progressively nesting on the tensile side of the $\Delta\theta = 2.5^\circ$ samples. These cracks arrested to at depth 104% larger on average than the one observed in the $\Delta\theta = 5^\circ$ configuration. Therefore, the $\Delta\theta = 2.5^\circ$ configuration was better able to grow and diffuse sub-critical Bouligand matrix cracks than any of the other configurations tested.

Contrarily to the $\Delta\theta = 5^\circ$ configuration, following the nesting of Bouligand matrix cracks, we did not observe any catastrophic failure in the $\Delta\theta = 2.5^\circ$ configuration. The sub-critical Bouligand matrix cracks progressively propagated via the activation of three main sub-critical dissipative failure mechanisms (instants (ii)–(iii) on Figure 3.12), namely

- crack twisting;
- crack branching; and
- secondary matrix cracks dispersion.

The activation of such failure mechanisms promoted the accumulation of diffused sub-critical damage on the tensile side of the $\Delta\theta = 2.5^\circ$ samples, leading to a stable failure process (smooth

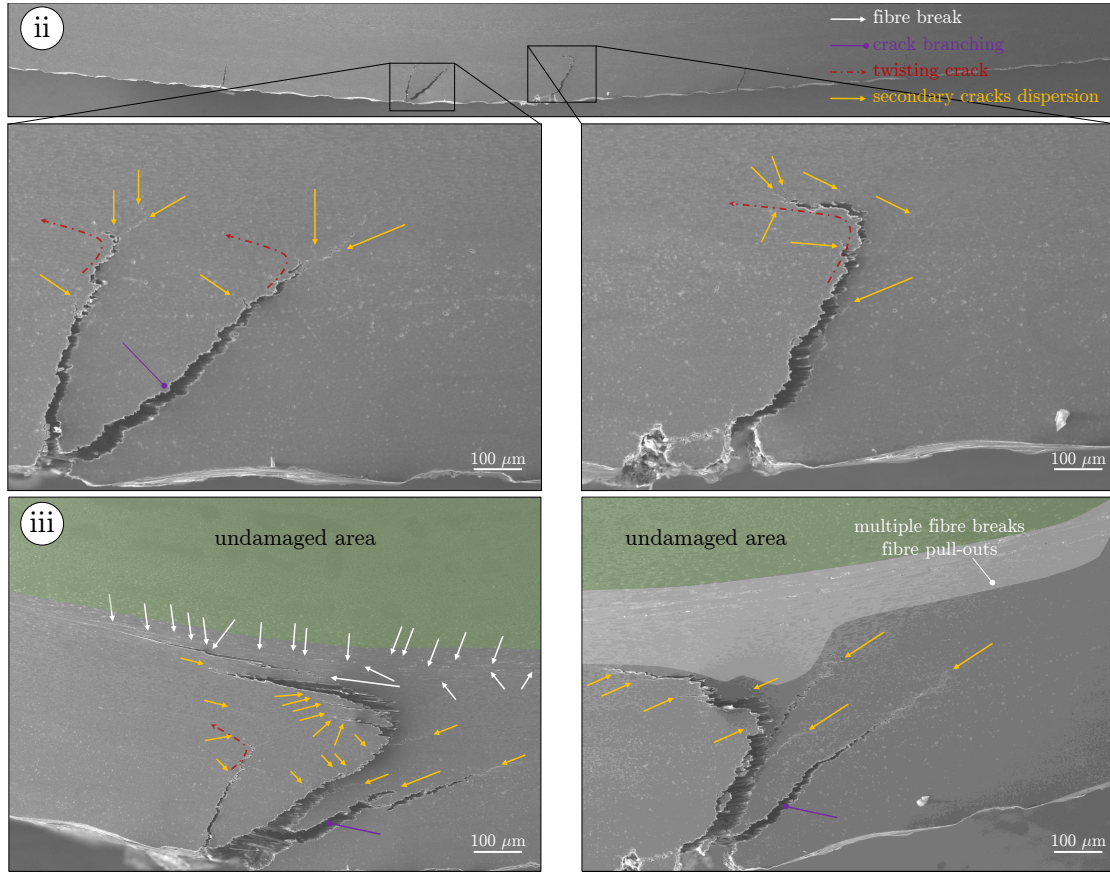


Figure 3.12: Progressive propagation of sub-critical Bouligand matrix cracks in the $\Delta\theta = 2.5^\circ$ configuration via the activation of several sub-critical dissipative failure mechanisms. These include crack twisting (red arrows), crack branching (purple arrows) and secondary matrix cracks dispersion (yellow arrows).

load vs displacement curve in Figure 3.5e) with the samples being able to dissipate energy while retaining about 50% on average of their maximum load bearing capability.

Figure 3.13 shows a detail of a region of the laminate close to the 90° interface at instant (iv) with the two Bouligand matrix cracks fully expanded. The figure highlights the presence of diffused fibre failure in the laminate. This was achieved via the propagation of sub-critical Bouligand cracks in the bulk of the laminate which led to a gradual stress relief in the tensile side. Therefore, stable and progressive ply fragmentation was achieved in the $\Delta\theta = 2.5^\circ$ configuration, further contributing to the stable energy dissipation process.

The final failure of the sample corresponded to the formation of a large helicoidal delamination on the tensile side. This was the first unstable damage propagation which occurred during the failure process of $\Delta\theta = 2.5^\circ$. Additionally, diffused Bouligand matrix cracks were observed both on the bulk of the laminate and on the compression side (instant (iv) on Figure 3.5e),

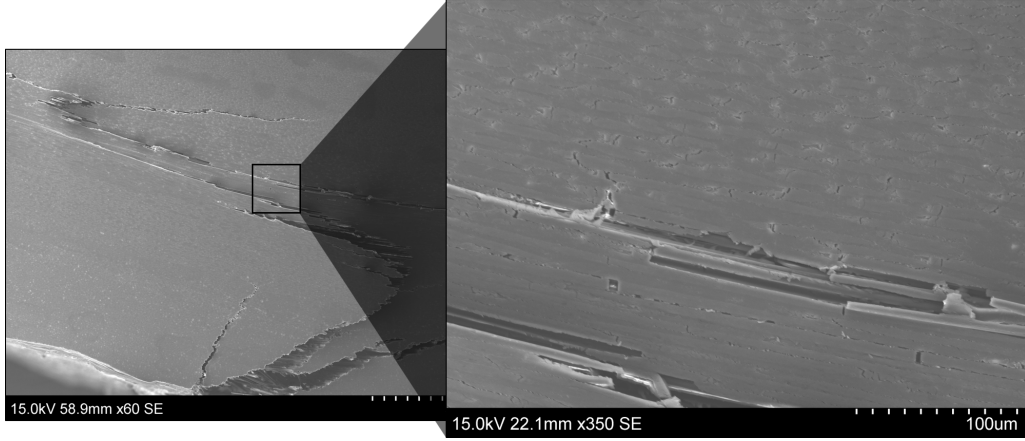


Figure 3.13: SEM image of a Bouligand matrix crack taken at instant for a sample with pitch angle $\Delta\theta = 2.5^\circ$. The zoom-in highlights the presence of diffused ply fragmentation.

further highlighting the capability of the $\Delta\theta = 2.5^\circ$ configuration in diffusing sub-critical Bouligand cracks.

Overall, by decreasing the pitch angle $\Delta\theta$ to values that mimic naturally-occurring Bouligand microstructures, the dominant damage mechanism in the failure process progressively shifted from fibre failure ($\Delta\theta = 45^\circ$ and 20°) to delamination ($\Delta\theta = 10^\circ$ and 5°) to stable sub-critical accumulation of damage ($\Delta\theta = 2.5^\circ$). By reducing the pitch angle $\Delta\theta$, we succeeded in significantly delaying final failure to a larger applied displacement — 130% larger on average for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$. For samples with $\Delta\theta = 2.5^\circ$, the activation of several sub-critical dissipative mechanisms of failure promoted sub-critical damage diffusion, it stabilised failure and largely increased the dissipated energy — 77% higher on average for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$, thus enhancing the resistance to through-the-thickness loads of the composite.

Interestingly, the analysis of the final failure of the samples tested revealed complete fracture in two halves of the samples with pitch angles $\Delta\theta = 45^\circ$, 20° and 10° , i.e the samples characterised by extensive fibre failure. On the contrary, even after reaching the maximum displacement allowed by the test-rig, it was not possible to physically split apart the samples with pitch angles $\Delta\theta = 5^\circ$ and 2.5° . Therefore, by reducing the pitch angle $\Delta\theta$, the structural integrity of the composite greatly increased.

3.4.3 Establishing an energy release rate threshold for the propagation of sub-critical Bouligand matrix cracks

As discussed in §3.4.2, sub-critical damage in the form of Bouligand matrix cracks was observed in configurations with pitch angles $\Delta\theta = 2.5^\circ, 5^\circ$ and 10° . Failure initiated at the back face (tensile side) of each sample. A single Bouligand matrix crack formed at the point with maximum curvature due to bending stresses. The crack twisted and propagated towards the bulk of the laminate (z -direction) up to a distance of about 425-450 μm , 200-225 μm and 80 μm for $\Delta\theta = 2.5^\circ, 5^\circ$ and 10° , respectively (see instant (i) in Figures 3.5e, 3.5d and 3.5c). For each configuration, the respective distance corresponds to a region with plies oriented at about 40° - 50° with respect to the fibre orientation at the back face of the sample (0°). We ensured that the cracks observed from the edge were extending throughout the entire width of the samples by interrupting the test and analysing the back-faces of the samples.

The use of the analytical model presented in §2.4.3 describing the growth of twisting Bouligand cracks in terms of the local (at the crack front) energy release rate (G) as function of the pitch angle ($\Delta\theta$), ply thickness (t_{ply}) and penetration depth (z) shows that, as a Bouligand matrix crack grows and twists starting from an initially flat crack (back face split in our case), the local energy release rate decreases (see Figure 3.14). In Figure 3.14, G_0 represents the energy

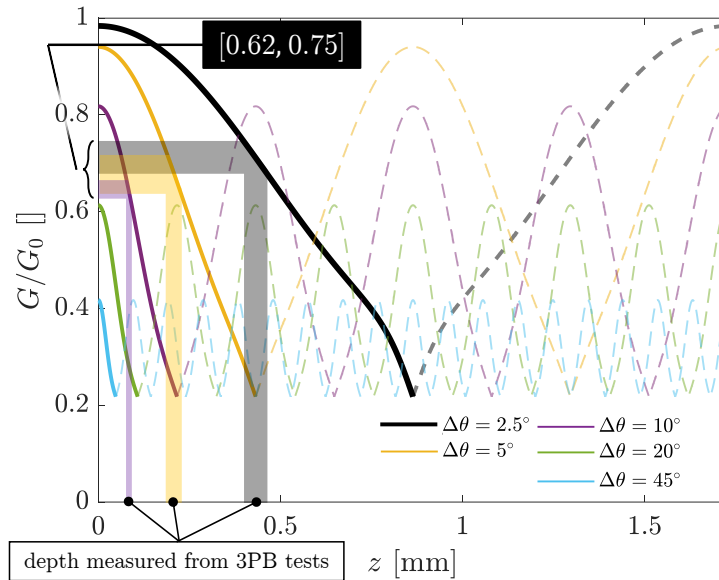


Figure 3.14: Energy release rate (G/G_0) vs. propagation depth z for pitch angles $\Delta\theta = 2.5^\circ, 5^\circ, 10^\circ, 20^\circ$ and 45° and $t_{\text{ply}} = 0.024 \text{ mm}$, calculated at the point of maximum along the crack front.

release rate if the crack were to remain flat. We therefore deduce that the gradual decrease in energy release rate below a certain threshold, $0.62 \leq G/G_0 \leq 0.75$ in this work, halted the crack propagation. This phenomenon, in addition to providing a mechanisms which is increasingly dissipative as damage propagates, promoted stress redistribution and, consequently, the nesting of additional matrix cracks at the back face of the laminate (instant (i) in Figures 3.5e, 3.5d). This is consistent with the observations made in the $\Delta\theta = 2.5^\circ$ samples tested under QSI (Figures 3.2b and 3.3).

As shown in Figure 3.14, the threshold of energy release rate for the arrest of sub-critical Bouligand matrix cracks was reached at larger penetration depths as the pitch angle $\Delta\theta$ decreases. This promoted the diffusion of sub-critical damage and delayed the formation of more catastrophic failure mechanisms such as delaminations and fibre failure. This finding correlates well with the experimental observation of a progressively delayed catastrophic failure as the pitch angle $\Delta\theta$ decreases obtained from the QSI tests (Figure 3.4).

Additionally, Figure 3.14 shows that, for any values of penetration depth (z), configurations with pitch angles $\Delta\theta = 45^\circ$ and 20° are characterised by values of G/G_0 smaller than the threshold for the arrest of sub-critical Bouligand matrix cracks. This finding correlates well with the absence of sub-critical damage on the back face of the $\Delta\theta = 45^\circ$ and 20° samples of both 3PB and QSI tests, where sudden fibre failure occurred instead.

3.4.4 Ultra-thin-ply vs. standard-ply-thickness Bouligand CFRP structures

In § 3.4.1, we have shown the unique simultaneous improvement in mechanical performances and damage resistance achieved with ultra-thin-ply Bouligand laminates characterised by small pitch angles $\Delta\theta$. This was obtained via the extensive formation of sub-critical damage in the form of (i) through-the-thickness distribution of helicoidal delaminations and (ii) Bouligand matrix cracks on the tensile side of the laminate.

The published literature on Bouligand-inspired CFRP laminates with standard-ply thickness (t_{ply}) counts several successful attempts to exploit Bouligand lay-ups in structural applications [12–19]. However, such a significant and simultaneous increase in three different

metrics for mechanical performance with respect to classical lamination sequences has not been reported yet.

Specifically, Shang et al. [13] performed QSI tests on CFRP Bouligand laminates ($t_{\text{ply}} = 80 \mu\text{m}$, $\Delta\theta = 10^\circ$ and 18°) and compared them against equivalent cross-ply (CP) laminates. Their results showed that the $\Delta\theta = 10^\circ$ laminates achieved a significant increase in peak load. However, the displacement at critical failure (load drop) and the dissipated energy were smaller. Pinto et al. [14] compared Bouligand CFRP ($t_{\text{ply}} = 125 \mu\text{m}$, $\Delta\theta = 15^\circ$) against QI laminates under LVI tests (30 J impact energy) achieving a 10% increase in peak load and a very similar dissipated energy. Ginzburg et al. [15] compared Bouligand CFRP ($t_{\text{ply}} = 125 \mu\text{m}$, $\Delta\theta = 12^\circ$) against QI laminates under LVI (40 J and 80 J impact energy). Their results showed that the QI laminates outperformed the Bouligand laminates both in terms of peak load and dissipated energy. More recently, Liu et al. [17] tested and compared thick (6 mm) Bouligand CFRP laminates under QSI ($t_{\text{ply}} = 80 \mu\text{m}$, $\Delta\theta = [2.5^\circ - 90^\circ]$) showing that the peak load increases when $\Delta\theta$ is reduced to 10° . However, for configurations with $\Delta\theta = 5^\circ$ and 2.5° the peak load as well as the displacement at critical failure was lower than for CP laminates.

Additionally, in standard-ply-thickness Bouligand CFRP laminates, reducing the pitch angle to small values usually leads to an in-plane spread of damage characterised by larger delamination areas and a lower degree of through-the-thickness damage compared to standard solutions [12, 15]. This damage distribution differs from the extensive diffusion of sub-critical through-the-thickness damage achieved in this work.

Therefore, the statistically-significant trendlines presented in this work suggest that, with ultra-thin-ply CFRP composites ($t_{\text{ply}} = 24 \mu\text{m}$), the smaller the pitch angle $\Delta\theta$ the larger the enhancement in damage resistance and structural integrity to out-of-plane loads; however, with standard-ply thickness, reducing the pitch angle $\Delta\theta$ to small values does not always improve the mechanical performances of standard lamination sequences.

As discussed in § 3.4.1, 3.4.2 and 3.4.3, the ability of the microstructure in diffusing damage at a sub-critical level via the formation of diffused helicoidal delaminations and Bouligand twisting matrix cracks is key for the improvement in performances to out-of-plane loads. For the same testing procedure, the activation and interaction of these failure mechanisms is dominated by the pitch angle $\Delta\theta$, the interface/matrix properties and the ply thickness. We therefore

conclude that the optimal choice of Bouligand structures should be driven by the optimal combination of pitch angle, matrix/interface properties and ply thickness.

3.5 Conclusions

In this work, we established how ultra-thin-ply CFRP laminates can overcome their inherent low resistance when loaded through the thickness by using original bio-inspired Bouligand layups with pitch angles $\Delta\theta$ down to 2.5° .

Our QSI tests showed that reducing the pitch angle $\Delta\theta$ leads to:

- progressively more significant through-the-thickness diffusion of sub-critical damage in the form of helicoidal delaminations and Bouligand matrix cracks. Such damage distribution acts as a stress relief mechanism for the tensile side of the laminate, largely delaying catastrophic fibre failure — 74% larger displacement at maximum load for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$;
- progressively larger:
 - energy dissipated at sub-critical levels — 165% larger for $\Delta\theta = 2.5^\circ$ than for 45° ;
 - total dissipated energy — 97% larger for $\Delta\theta = 2.5^\circ$ than for 45° ;
 - peak load — 92% larger for $\Delta\theta = 2.5^\circ$ than for 45° ; and
 - penetration load — 45% larger for $\Delta\theta = 2.5^\circ$ than for 45° .

These statistically-significant trends suggest that, with ultra-thin-ply CFRP materials, the smaller the pitch angle $\Delta\theta$ the larger the improvement in resistance to transverse loads;

- progressively more damage resistant post-peak behaviour. Laminates with small pitch angles offered higher resistance to penetration than samples with large pitch angles. In the former, this was achieved with an increasing diffusion of sub-critical damage and a twisted ply-fracture pattern. In the latter, sub-critical damage was not observed and ply-fracture mainly localised on three fracture planes.

The full-penetration QSI tests revealed the outstanding resistance to through-the-thickness loads of Bouligand laminates with small pitch angles. This was achieved by a large sub-critical accumulation of damage extending up to the distal face of the laminates.

We subsequently investigated experimentally and analytically how such damage, in the form of sub-critical Bouligand matrix cracks, initiates and diffuses in the bulk of the laminate depending on the pitch angle $\Delta\theta$ via 3PB tests in SEM environment. The combined experimental and analytical investigation showed that reducing the pitch angle $\Delta\theta$ leads to:

- progressive transition in dominant failure mode from fibre failure ($\Delta\theta = 45^\circ$) to co-present fibre failure and delamination ($\Delta\theta = 20^\circ$ and 10°) to delamination ($\Delta\theta = 5^\circ$) to sub-critical diffused damage in $\Delta\theta = 2.5^\circ$;
- nesting of Bouligand matrix cracks promoted by a crack arrest mechanism. The nesting and subsequent growth of Bouligand matrix cracks act as a stress relief mechanism on the tensile side of the laminates, thereby greatly delaying catastrophic failure — up to 130% larger ultimate displacement for $\Delta\theta = 2.5^\circ$ than $\Delta\theta = 45^\circ$;
- diffused sub-critical twisting helicoidal matrix cracks forming at the back face of the laminate in the $\Delta\theta = 2.5^\circ$ configuration characterised by the activation of several sub-critical dissipative mechanisms of failure such as crack branching, secondary cracks dispersion and stable ply fragmentation. The diffuse damage resulted in a large increase in total dissipated energy — 77% higher for $\Delta\theta = 2.5^\circ$ than for $\Delta\theta = 45^\circ$;
- larger propagation depth of sub-critical Bouligand matrix cracks. This finding can be explained using the analytical model we presented in § 2.4.3 in order to study the evolution of the energy release rate at the front of twisting Bouligand cracks; and
- increased residual structural integrity, as the 3PB specimens did not separate in two parts for the lower pitch angles.

To summarise, we demonstrated the extraordinary capability of ultra-thin-ply CFRP Bouligand structures in dissipating energy at sub-critical levels leading to a significant simultaneous increase in (i) maximum load-bearing capability (92%), (ii) delay in catastrophic failure (74%) and (iii) energy dissipation (97%). Very significantly, our findings establish firmly that the inherent low performance to transverse loading of thin-ply CFRP (with standard lamina-

tion sequences) can be overcome, therefore greatly expanding the potential of thin-ply CFRP materials for structural applications.

Chapter 4

Herringbone-Bouligand CFRP structures: a new tailorable damage-resistant solution

4.1 Introduction

In the past years, the high damage resistance of the mantis shrimp’s dactyl club has been largely attributed to the features of the so-called periodic region of the microstructure [4, 5]. However, a recent investigation of the mantis shrimp [5] has revealed that the microstructural traits of the club’s impact region and impact surface greatly contribute to the overall damage resistance of the club. These recently-identified features have never been explored with Carbon Fibre Reinforced Plastics (CFRPs) and might hold the potential to push forward the damage resistance performances of ‘classical’ Bouligand CFRP microstructures.

In addition to the periodic region, fully described in [Chapter 2](#), by observing the cross-section of the mantis shrimp’s dactyl club (Figure 4.2c), we can distinguish two additional areas: (i) the impact region and (ii) the impact surface [5] (Figure 4.2c). The impact region consists of Bouligand units (see Figure 4.1a), repeated through the thickness of the club and featuring a periodic out-of-plane (z -thickness direction) fibre component, resulting in several wavy plies

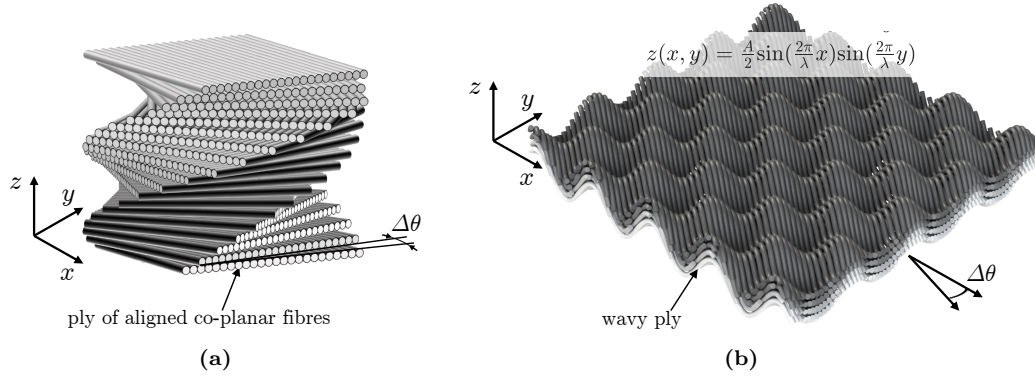


Figure 4.1: Schematics of (a) Bouligand and (b) Herringbone-Bouligand microstructures.

stacked in a helicoidal fashion (see Figure 4.1b and the ‘zig-zag’ (Herringbone) pattern in Figure 4.2c). The wavy architecture defines bi-sinusoidal interfaces between neighbouring plies that can be expressed as

$$z(x, y) = \frac{A}{2} \sin\left(\frac{2\pi}{\lambda}x\right) \sin\left(\frac{2\pi}{\lambda}y\right), \quad (4.1)$$

where A is the amplitude and λ the wavelength of the Herringbone pattern [5]. While the value of λ is uniform across the impact region ($\lambda \approx 45 \mu\text{m}$), the amplitude A progressively decreases from $100 \mu\text{m}$ at the impact region/impact surface interface to $70 \mu\text{m}$ at the impact region/periodic region interface, thus avoiding the presence of a weak interface between the two regions [5]. Finally, the impact surface consists of a highly-oriented crystalline apatite mineral phase layer [5].

For the impact region, Yaraghi et al. [5] have shown, using 3D-printed Herringbone-Bouligand structures compared against Bouligand architectures under uniaxial compression (a photo-polymer was used as fibre reinforcement while a rubber-like elastomer as matrix), that the Herringbone-Bouligand pattern provides a mechanism for stress redistribution, leading to a more uniform through-the-thickness stress state and a higher compressive stiffness. This was expected to promote damage diffusion under compression. Additionally, a combined experimental/numerical analysis of the impact surface of the club, showed that the nanoparticles within the impact surface are able to redistribute contact stresses via plastic deformation through particle motions, particle pile-up and crack redirection [5]. This was expected to prevent stresses from localising on the peaks of the underlying Herringbone-Bouligand pattern.

Recently, Han et al. [43] created a basalt fibre-reinforced sandwich composite attempting to mimic the Herringbone pattern with a 2D woven fabric and the periodic region with layers

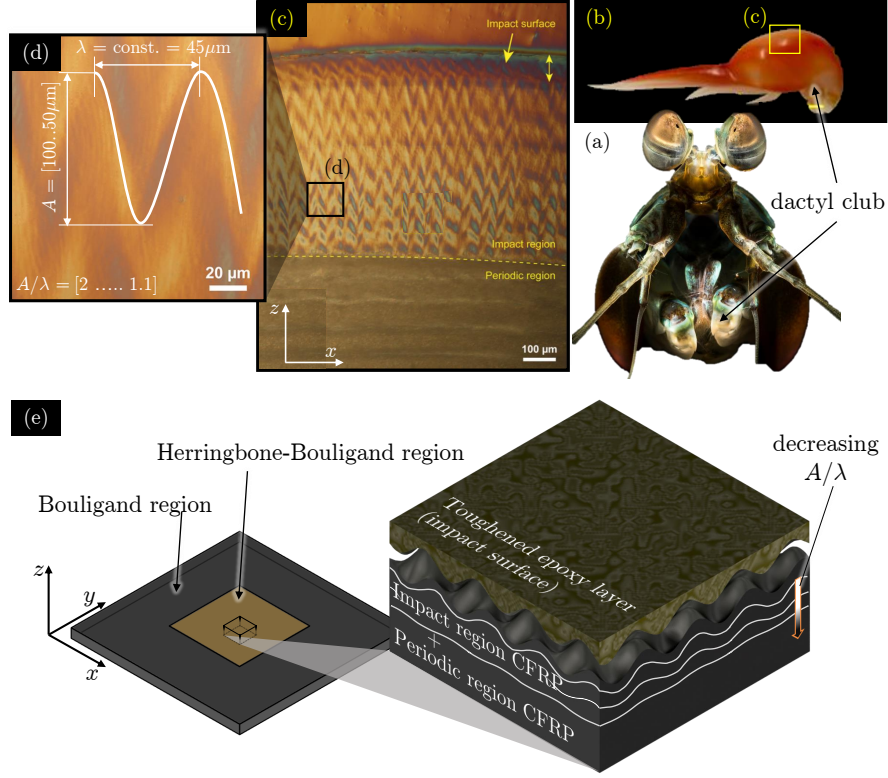


Figure 4.2: (a) Photograph of the mantis shrimp (*Odontodactylus scyllarus*) [22]. (b) Dactyl club. (c) Differential interference contrast image showing the impact surface, the impact region and the periodic region of the cross section of the mantis shrimp's dactyl club. (d) High resolution detail of the impact region highlighting the Herringbone motif (z -direction) of the impact region. (b-d) Adapted from [5]. (e) Schematic of the Herringbone-Bouligand sample designed in this work with zoom-in on the Herringbone-Bouligand region tailored at the centre of the sample.

of *discontinuous* basalt fibres. Their results showed that, compared to a traditional basalt *discontinuous* fibre composite, the club-inspired samples achieved higher tensile and compressive moduli, higher tensile strength and higher impact resistance.

While the highly-dissipative failure mechanisms typical of the periodic region have been successfully exploited with CFRPs (see [Chapter 2](#) and [Chapter 3](#)) the recently-uncovered features of the impact region and impact surface of the club, fundamental to the resistance of the mantis shrimp's dactyl club to through-the-thickness loads, have never been explored with CFRPs.

In view of this, we devised an original study to investigate, for the first time in the literature, high-performance Herringbone-Bouligand microstructures with CFRP materials, aiming at creating a point-by-point tailorable CFRP bio-inspired composite which comprises the

main features of the impact surface, impact region and periodic region of the mantis shrimp dactyl club (Figure 4.2e). To this end, we devised the first manufacturing technique, based on the micro-moulding of uncured prepreg CFRPs and used it to prototype the first CFRP Herringbone-Bouligand microstructure. We used Quasi-Static Indentation (QSI) tests to compare the damage resistance to through-the-thickness loads of the prototyped Herringbone-Bouligand microstructure against classical Bouligand laminates. We demonstrate that, by mimicking the main features of the microstructure of mantis shrimp's dactyl club, we can achieve similar toughening mechanisms to the ones observed in nature, hence greatly enhancing the damage resistance of CFRP structures.

This chapter contains the following original contributions:

- (i) we successfully design, prototype, test and analyse the first high-performance Herringbone-Bouligand microstructures (with CFRP);
- (ii) we devise the first prototyping technique, based on the moulding of uncured prepreg, suitable for designing point-by-point tailored Herringbone-Bouligand microstructural solutions;
- (iii) we show that, with a tailored Herringbone-Bouligand design, it is possible to achieve highly reduced in-plane spreading of damage, and contain this damage to the tailored Herringbone-Bouligand region;
- (iv) we demonstrate the capability of bi-sinusoidal CFRP ply interfaces in activating sub-critical dissipative mechanisms of failure such as discontinuous (i.e. non-linked) delamination areas, deflected delaminations, and diffused matrix cracks; and
- (v) we show that a tailored Herringbone-Bouligand microstructural design can greatly enhance the damage resistance of 'classical' Bouligand CFRP microstructures.

4.2 Design of Herringbone-Bouligand CFRP microstructures

We used four parameters to design the Herringbone-Bouligand microstructure, namely the pitch angle $\Delta\theta$, the number of Bouligand units (total number of plies), the amplitude (A) and the wavelength (λ) of the Herringbone pattern (see Equation 4.1).

Based on the studies presented in Chapter 2 and Chapter 3, we chose to build laminates with two Bouligand units and with pitch angle $\Delta\theta = 2.5^\circ$ for both Bouligand and Herringbone-Bouligand microstructures. The choice of A and λ was driven by two requirements (schematically reported in Figure 4.3a): ① the maximum bending strain of carbon fibres during the moulding process should not exceed the respective failure strain ($A < \frac{\varepsilon_f^* \lambda^2}{2\pi^2 r_f}$, where ε_f^* and r_f are respectively the failure strain ($\varepsilon_f^* = 1.9\%$, see Table 2.4) and the radius of the carbon fibres ($r_f = 3.4 \mu\text{m}$, see Table 2.4); and ② the mould manufacturability, i.e. tool size available to mill the mould, $A \leq \frac{\lambda^2}{2\pi^2 r}$, where r is the radius of the cylindrical tool used to mill the Herringbone mould.

The two driving requirements can be graphically represented in the design space reported in Figure 4.3b. In this work, the smallest tool available to manufacture the mould had a

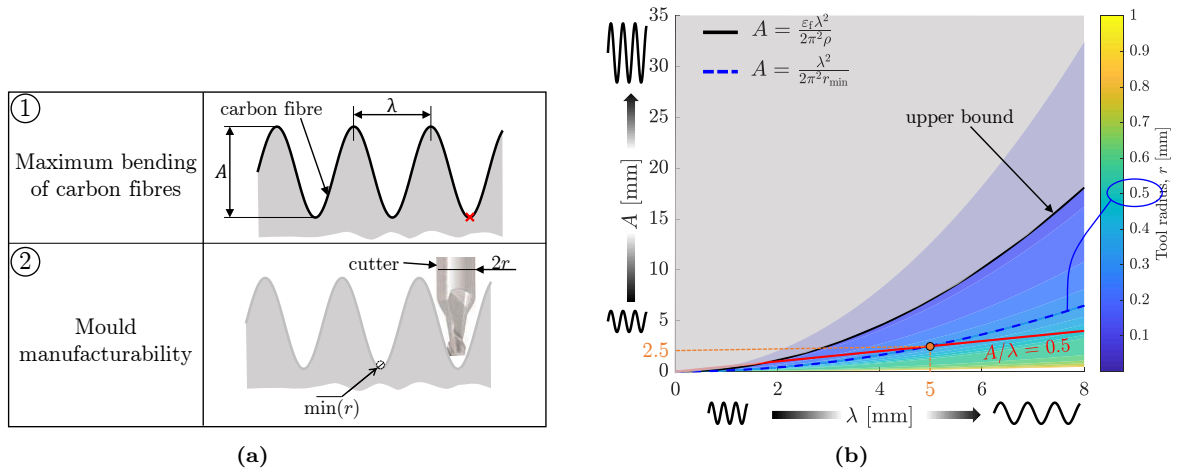


Figure 4.3: (a) Schematic and (b) graphical representations of the two design requirements used to drive the choice of the amplitude A and wavelength λ of the Herringbone pattern. Specifically, in (b) the black curve $A(\lambda)$ represents the upper bound of the design space, corresponding to the maximum bending strain of the carbon fibre equal to its failure strain. The colour map expresses a limitation on the mould manufacturability based on the tool sizes available to mill the Herringbone pattr. The dashed blue line refers to the minimum tool size available. The red line correspond to the adopted value of A/λ .

radius $r_{\min} = 0.5$ mm. We then decided to adopt an $A/\lambda = 0.5$ which resulted in values of $A = 2.5$ mm and $\lambda = 5$ mm compatible with the overall thickness of the Herringbone-Bouligand CFRP laminate.

As described in §4.1, in this work, we aimed at developing a point-by-point tailorable Herringbone-Bouligand microstructure. Therefore, we designed the mould (and the Herringbone-Bouligand CFRP sample) such that the Herringbone-Bouligand microstructure was included only in the central part of the sample (Herringbone-Bouligand region), extending for an area of 50 mm by 50 mm. The microstructure surrounding the Herringbone-Bouligand region was equivalent to a classical Bouligand layup with pitch angle $\Delta\theta = 2.5^\circ$. Figure 4.4 shows the high-quality finishing of the aluminium Herringbone mould used in the prototyping of the Herringbone-Bouligand CFRP samples.

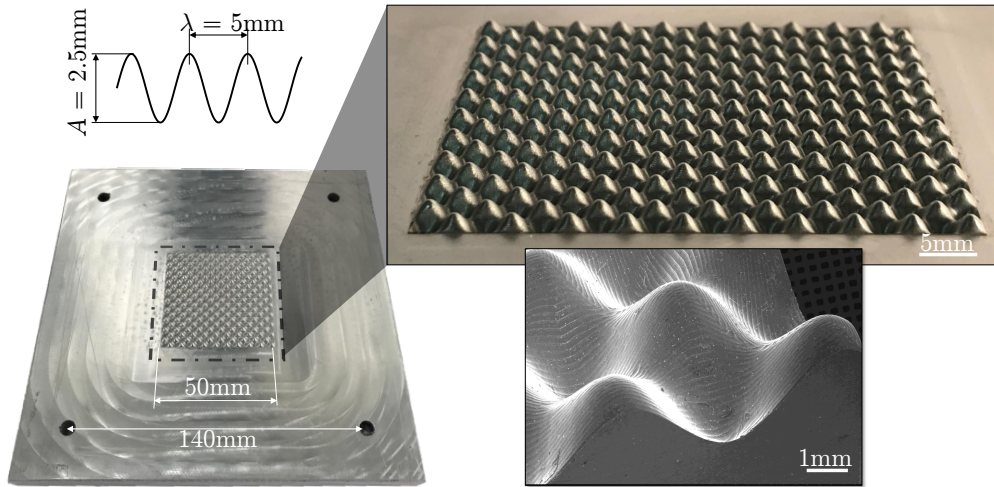


Figure 4.4: Photographs and SEM image of the mould used for prototyping tailorable Herringbone-Bouligand CFRP laminates.

4.3 Materials and methods

4.3.1 Materials

Bouligand and Herringbone-Bouligand samples were manufactured using Skyflex USN20A, a unidirectional prepreg tape with areal weight (20 gsm). The properties of the prepreg constituents, TR30S 3K carbon fibres made by Mitsubishi and K50 epoxy matrix from SK Chemicals, can be found in Table 2.4 along with the ply-level properties in Table 2.3. We used Scotch-Weld™ EC-9323, a high-impact-resistant epoxy-based adhesive, to create the impact surface of the Herringbone-Bouligand region (adhesive properties in [44]).

4.3.2 The first micro-moulding technique for prototyping Herringbone-Bouligand structures

Figure 4.5 shows the flow chart of the novel prototyping procedure we developed in order to manufacture CFRP Herringbone-Bouligand samples. The stacking sequence of the Herringbone-Bouligand samples was $[(0^\circ/2.5^\circ/\dots/177.5^\circ)/180^\circ/(177.5^\circ/175^\circ/\dots/0^\circ)]$.

For the pre-moulding procedure (passage A in Figure 4.5), the Herringbone mould was pre-heated at 50°C to soften the stacks of uncured prepreg. We covered the sublaminates using a breathing-cloth with a central hole to leave the Herringbone-Bouligand region uncovered. We placed the set-up under vacuum for 10 min and used an indenter to mechanically press down the sublaminates, hence improving the quality of the moulding. Rapid cooling was applied immediately after moulding to stabilise the moulded Herringbone pattern. The combination of moulding temperature (50°C), moulding time (10 min) and number of plies used in each sublaminate was the outcome of an experimental iterative study aiming at minimising fibre misalignment (due to fibre spreading) and maximising the mouldability of the sublaminates.

We performed an in-mould curing of the final stack s in the autoclave. While the mould was on the bottom side of the laminate during curing, the upper side was left free. This was expected to allow for the straightening of the outermost CFRP layers, thereby leading to a gradual transition in amplitude A of the Herringbone pattern from the maximum value (at the interface with the mould) to zero (at the back face of the laminate). We added an initial

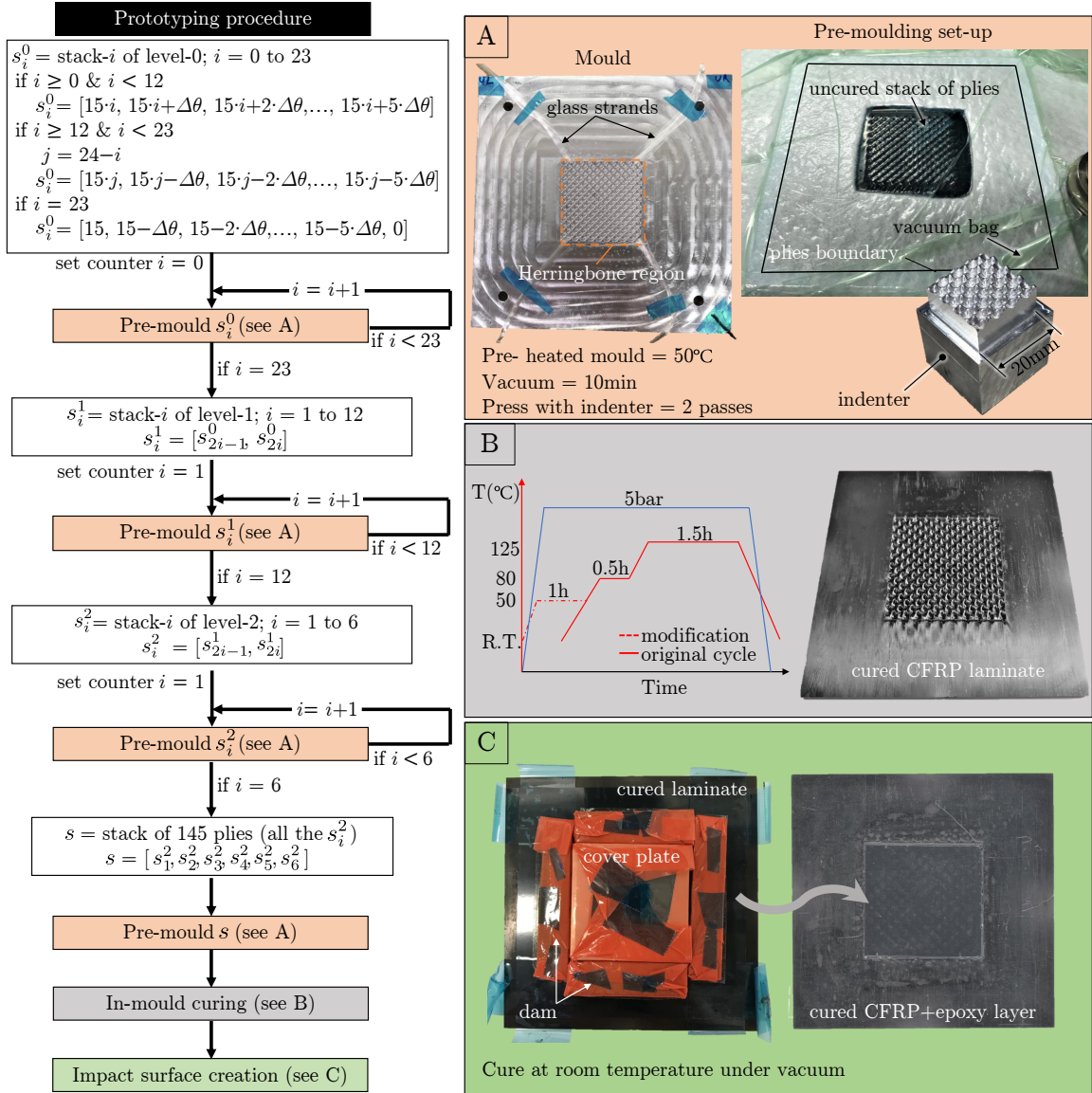


Figure 4.5: Flow chart of the prototyping procedure developed for manufacturing tailor-able Herringbone-Bouligand CFRP laminates with a graphical representation of (A) the pre-moulding procedure, (B) the curing cycle and a photograph of a cured Herringbone-Bouligand laminate and (C) the set-up used for the creation of the impact surface and a photograph of the final Herringbone-Bouligand laminate.

step to the curing cycle suggested by the manufacturer of Skyflex USN20A to maximise the moulding of the uncured laminate before curing.

Finally, we cut the cured CFRP Herringbone-Bouligand laminate with a water-jet and we created the impact surface by spreading a layer of toughened-epoxy adhesive on the Herringbone-Bouligand region. The adhesive was cured under vacuum, at room temperature, using the set-up shown in passage C in Figure 4.5. The average thickness of both Boulig-

and and Herringbone-Bouligand laminates is similar, and the most significant difference is the microstructure itself.

The stacking sequence of the Bouligand samples was the same as the one of the Herringbone-Bouligand samples, i.e. $[(0^\circ/2.5^\circ/\dots/177.5^\circ)/180^\circ/(177.5^\circ/175^\circ/\dots/0^\circ)]$. Similarly to the Herringbone-Bouligand samples, for the Bouligand samples we cut plies already at the specific angle and we used an alignment mould to perform the lay-up. The Bouligand samples were cured using the same autoclave cycle as the one of the Herringbone-Bouligand samples and the samples of both configurations were scanned for defects before testing using ultrasonic C-scans.

4.3.3 Analysis of the Herringbone-Bouligand CFRP microstructure

Figure 4.6 shows a photograph and several optical micrographs of a manufactured Herringbone-Bouligand sample following the prototyping procedure described in § 4.3.2.

Specifically, the micrograph of section A-A shows that we successfully created a layered CFRP Bouligand microstructure ($\Delta\theta = 2.5^\circ$) with bi-sinusoidal (Equation 4.1) ply-interfaces (Herringbone motif) characterised by a progressive variation of the amplitude to wavelength ratio (A/λ) decreasing from the top surface of the laminate to the bottom surface. The micrograph of section A-A shows that the toughened-epoxy used to create the impact surface of the Herringbone-Bouligand microstructure (as described in § 4.3.2 and in Figure 4.5) successfully filled the gaps between the peaks of the underlying CFRP Herringbone-Bouligand structure, creating a flat layer with a very good geometrical stability. Finally, section C-C highlights the good quality of the transition zone between the Bouligand region and the Herringbone-Bouligand region.

While the overall moulding procedure was successful, we could identify three types of manufacturing features in the microstructure: (i) a small amount of fibre misalignment confined to a small region close to the top surface of the laminate, where A/λ is maximum; (ii) the limited presence of voids (see section C-C in Figure 4.6) on the bottom half of the laminate; and (iii) the presence of not fully-moulded peaks resulting in a periodic flattening of the bi-sinusoidal outermost layers (see section B-B). The latter might have originated during the curing of the laminate due to the presence of trapped air between the top surface of the laminate and the mould as reported in the schematic. A top view of the cured Herringbone-Bouligand CFRP

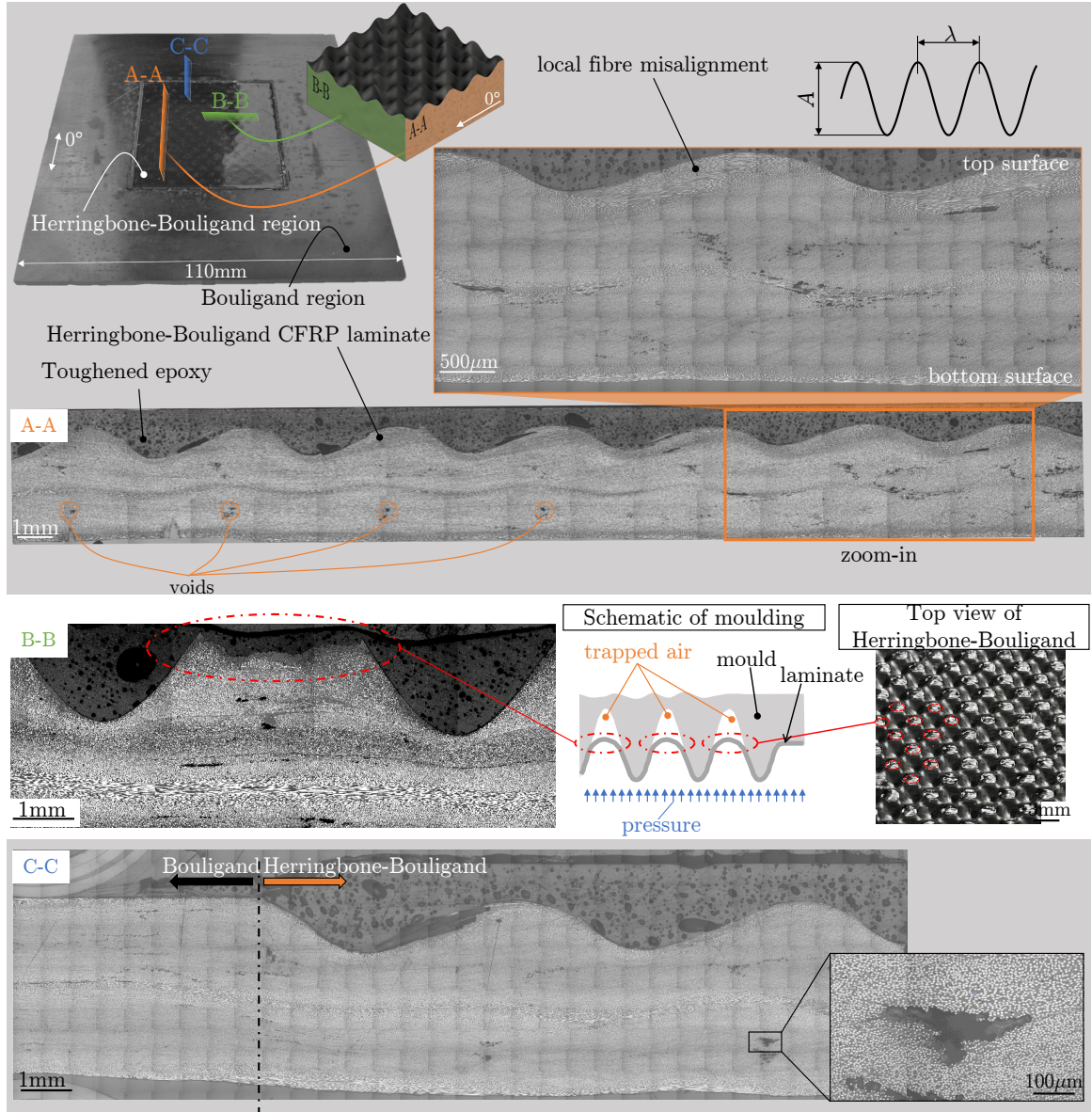


Figure 4.6: Photograph and micrographs of a prototyped Herringbone-Bouligand sample with indication of three cutting planes used for optical microscopy.

laminate highlighting the flattened peaks is reported in the figure. As it will be shown in § 4.4, the aforementioned defects did not significantly interact with the failure mechanisms and consequently they did not affect the mechanical performances of the Herringbone-Bouligand laminates under QSI.

Overall, the prototyping procedure led to a good quality of the final laminate and a good geometrical control over the parameters defining the Herringbone-Bouligand microstructure.

Therefore, we were successful in mimicking the features of the impact surface, impact region and periodic region of the mantis shrimp's dactyl club.

4.3.4 QSI test method

We performed full-penetration QSI tests using a custom-made test rig designed for the samples developed in this work. Each square sample (110 mm wide and ≈ 3.5 mm thick) was clamped between two aluminium plates (each 25 mm thick) with a central square opening 80 mm wide. The geometry of the opening was defined based on the geometry of the tailored Herringbone-Bouligand region of the Herringbone-Bouligand samples. The resulting span-to-thickness ratio of the QSI samples was ≈ 22.8 , in the range of values suggested in the ASTM D6264 standard [45].

We used a 50 kN Instron machine to load the samples at 1 mm/min displacement rate, using a hemispherical indenter made of steel 20 mm in diameter. We acquired the data with an interval of 100 ms. Additionally, we used an Imetrum camera to record the displacement at the indenter tip and correct for the machine compliance. For both Herringbone-Bouligand and Bouligand microstructures, we tested three samples until full-penetration (test stopped below a measured load of 1 kN) and one sample until an applied load of 4 kN to perform optical microscopy. All the samples tested until full-penetration were tested using step-loading cycles. We interrupted the test at an applied load of 2 kN, 4 kN and immediately after catastrophic failure (load-drop). For each laminate, between each loading step, we analysed the surface damage via visual inspection of the impact and back surfaces, and delamination damage via ultrasonic C-scans performed in an immersion tank.

Table 4.1: Mean $\pm$ STD of peak load, displacement at peak load, penetration load and total dissipated energy for the Herringbone-Bouligand and Bouligand samples tested under QSI.

Microstructure	Peak load [kN]	Displacement at peak load [mm]	Penetration load [kN]	Total dissipated energy [J]
Herringbone-Bouligand	8.19 ± 0.68	4.78 ± 0.10	5.47 ± 0.24	58.78 ± 4.62
Bouligand	7.45 ± 0.11	5.30 ± 0.05	4.85 ± 0.85	52.02 ± 5.69

4.4 Results

For the Herringbone-Bouligand and Bouligand CFRP samples tested under QSI, Table 4.1 shows the average values and standard deviations of peak load, displacement at peak load, penetration load and total dissipated energy. Figures 4.7a shows the load vs displacement curves. Figure 4.7b shows the peak load and total dissipated energy. Figures 4.7c-4.7d respectively show the evolution of the dent diameter (ϕ) and the total projected delamination area at different stages of the test (applied load of 2 kN, 4 kN, critical failure (load-drop) and full penetration (end of test)).

Figure 4.7 shows that, compared to the classical Bouligand microstructure, the Herringbone-Bouligand architecture led to: (i) a stiffer mechanical response during the initial stages of

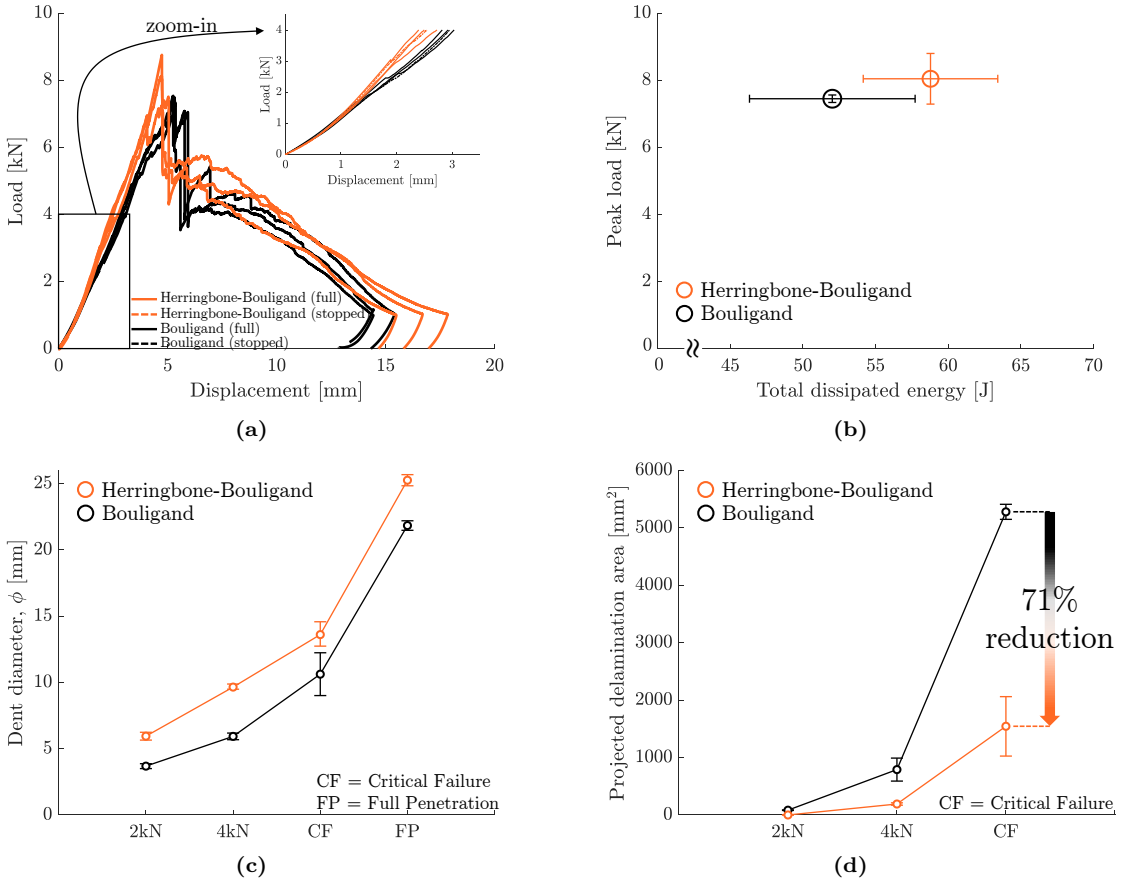


Figure 4.7: Results of the QSI tests for Herringbone-Bouligand and Bouligand samples. (a) Load vs displacement curves. (b) Peak load vs total dissipated energy. (c) Dent diameter and (d) total projected delamination area calculated at an applied load of 2 kN, 4 kN and at critical failure (load-drop) for the total projected delamination area and up to full-penetration for the indenter.

the loading; (ii) a 10% increase in maximum load bearing capability; (iii) a 11% decrease in displacement at critical failure; (iv) a 13% increase in penetration load; (v) a 13% increase in total dissipated energy; (vi) a larger dent diameter (on average 62% larger before critical failure and 22% larger after critical failure); (vii) a greatly reduced (71%) total projected delamination area.

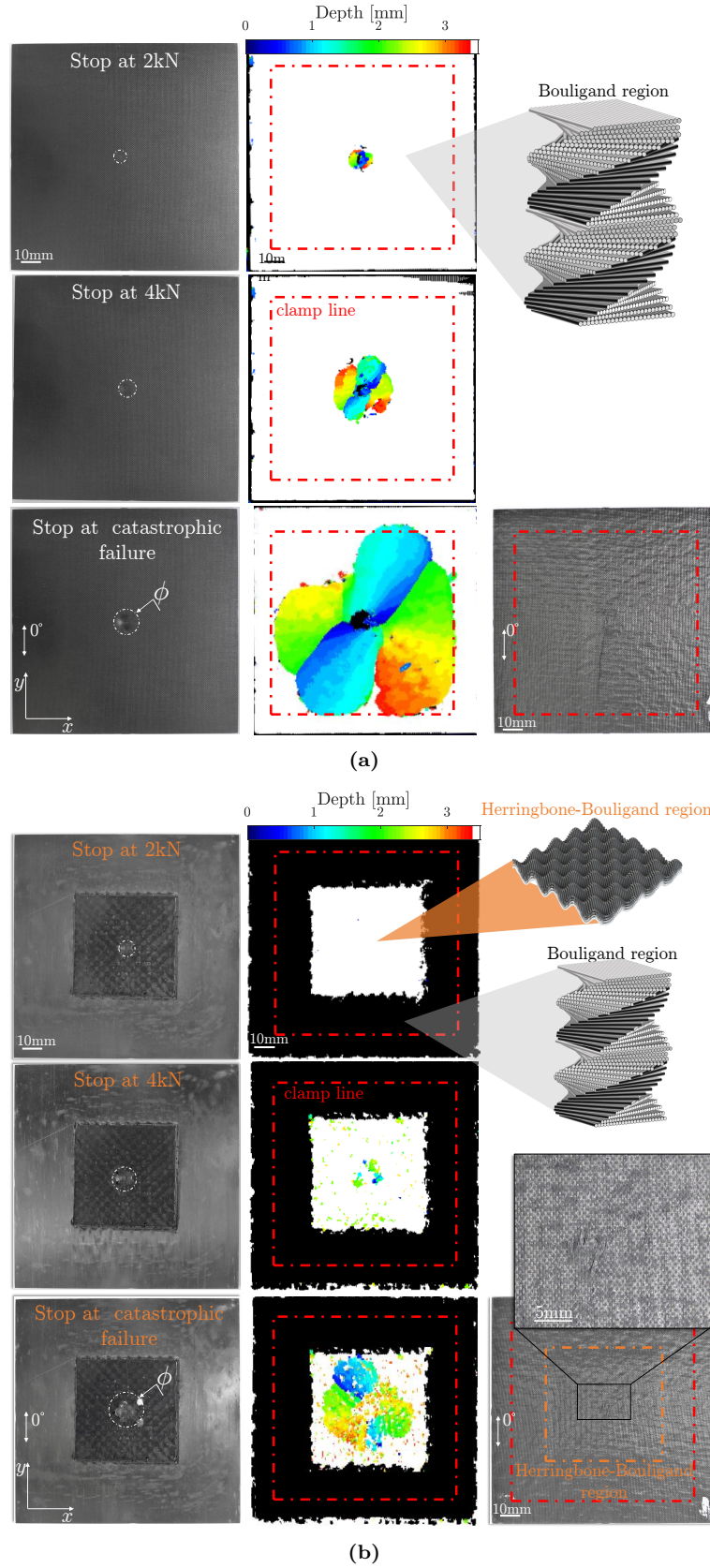


Figure 4.8: Photographs of the indentation face, back face and delamination damage obtained via ultrasonic C-scans of representative (a) Bouligand and (b) Herringbone-Bouligand samples taken by interrupting the test at an applied load of 2 kN, 4 kN and at critical failure (load-drop).

For two representative Bouligand and Herringbone-Bouligand samples, Figure 4.8a and Figure 4.8b respectively show photographs of the impact face, the back face and the delamination damage detected via ultrasonic C-scans. Schematics of the type of microstructure characterising a certain region in the laminate are reported in the figures. The coloured areas in the C-scans refer to delamination at a certain ply-interface (see colour bar on top of each figure). The photographs and the C-scans images were taken by interrupting the test at an applied load of 2 kN, 4 kN and right after catastrophic failure (load-drop).

Figure 4.9 shows photographs of two representative Herringbone-Bouligand and Bouligand samples tested up to a load of 4 kN and then cut for optical microscopy (sections A-A and B-B for the Herringbone-Bouligand and section C-C for the Bouligand sample). Note that the microstructural features described in § 4.3.3 did not significantly interact with the damage evolution in the Herringbone-Bouligand laminates.

Figure 4.10 shows photographs of the back face of two representative Herringbone-Bouligand and Bouligand samples after full-penetration, i.e. end of QSI test; the location of the clamp line as well as the boundary of the Herringbone-Bouligand region is reported.

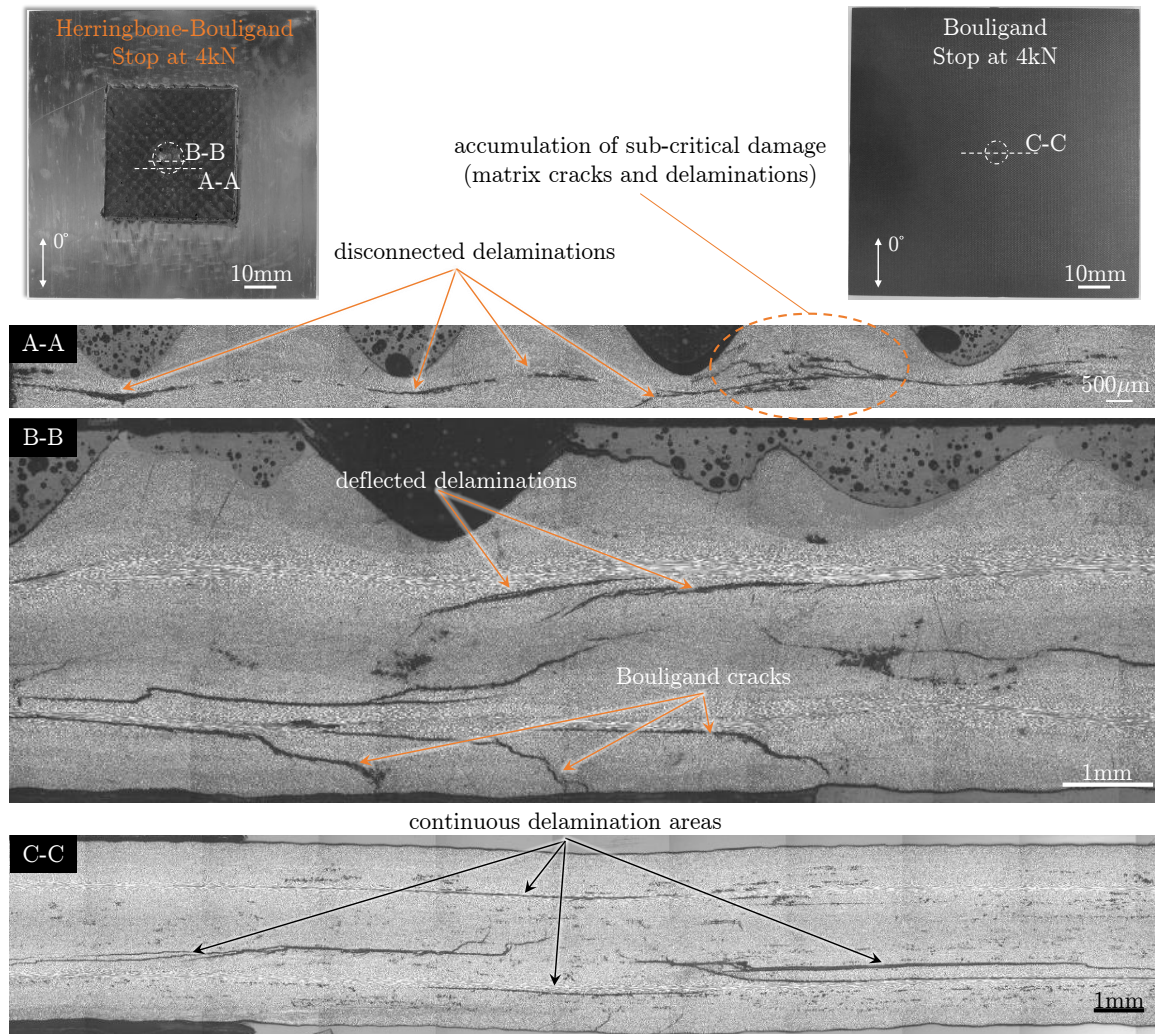


Figure 4.9: Photographs of Herringbone-Bouligand (top-left) and Bouligand (top-right) samples tested up to an applied load of 4kN and cut for microscopy. Specifically, for the Herringbone-Bouligand, sections A-A and B-B highlight the presence of disconnected delamination areas, deflected delaminations and diffused sub-critical matrix cracks. For the Bouligand sample, section C-C highlights the lower degree of sub-critical damage, including continuous delamination areas.

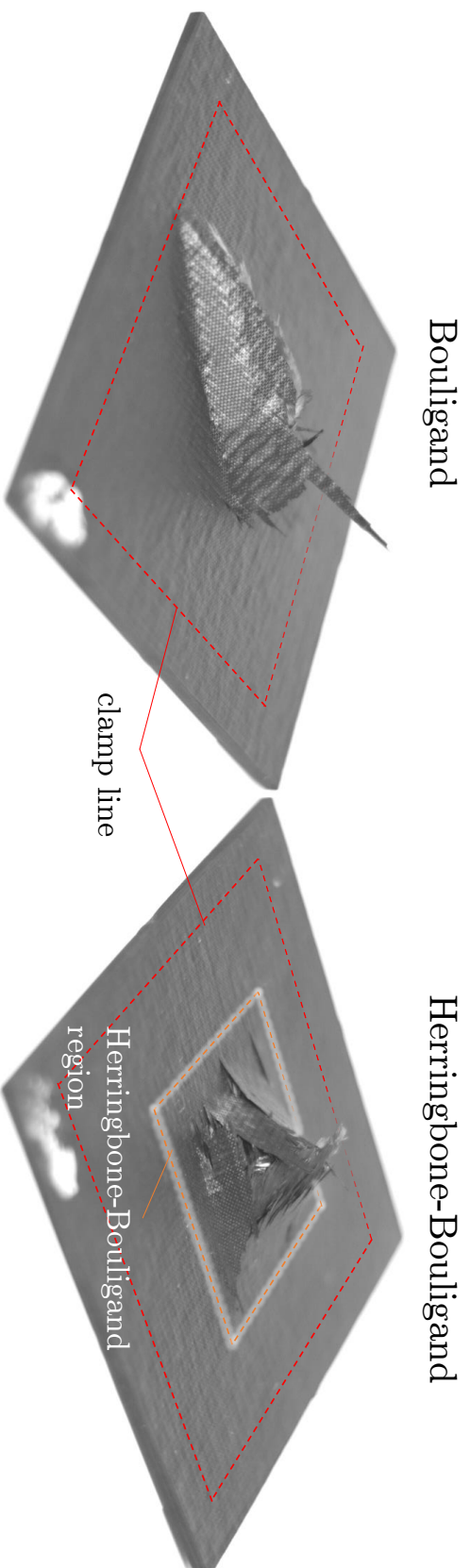


Figure 4.10: Photographs of the back face of representative samples of Herringbone-Bouligand and Bouligand configurations at the end of the QSI test (full penetration) highlighting how the Herringbone-Bouligand microstructure managed to confine damage to the Herringbone-Bouligand region.

4.5 Discussion

4.5.1 Enhancing the damage resistance of classical Bouligand structures with a Herringbone-Bouligand design

4.5.1.1 The sub-critical high-damage-resistant accumulation of damage in Herringbone-Bouligand structures

Figure 4.8a shows that, at an applied load of 2 kN, delamination damage was already present in the Bouligand microstructure. On the contrary, no damage could be detected with the ultrasonic probe in the Herringbone-Bouligand microstructure (Figure 4.8b). Therefore, the Herringbone pattern successfully delayed the onset of delamination damage. This finding is in line with previous studies which show that by increasing the amplitude to wavelength ratio (A/λ) of sinusoidal and triangular interfaces, crack propagation is delayed and the toughness increases for both predominant Mode I [5, 46–49] and Mode II [50] opening.

As the load increases, the smaller extent of delamination damage in the Herringbone-Bouligand laminates contributed to a stiffer mechanical response (Figure 4.7a). On the contrary, the earlier formation of helicoidal delamination damage (Figure 4.8a) in the Bouligand samples led to a gradual softening (knee region), corresponding to a non-linear load response in Figure 4.7a.

At an applied load of 4 kN, section C-C in Figure 4.8a and Figure 4.9 show that the helicoidal distribution of delaminations in Bouligand laminates contain various continuous (in-plane ($x - y$)) delamination areas at the interface between two generic plies. On the contrary, for the Herringbone-Bouligand samples, disconnected delamination areas appeared at the same interface between two generic plies (Figure 4.9 and sections A-A and B-B in Figure 4.8b). As reported in § 4.2-4.3, the Herringbone pattern designed in this work defines bi-sinusoidal interfaces between consecutive plies, with ratios A/λ ranging nominally from 0.5 (on the top surface of the laminate) to 0 (on the bottom surface). Due to the nature of the loading in QSI tests, these interfaces are subjected to dominant Mode II. Consequently, any bi-sinusoidal interface with $A/\lambda > 0$ forms a mechanical interlock. This results in an important damage-resistant mechanism which: (i) together with the lower amount of delamination damage contributes to a stiffer mechanical response; (ii) results in a higher resistance to form and grow delaminations

(‘adhesive’ type of failure [50]) leading to the formation of discontinuous delamination areas; and (iii) promotes the activation of sub-critical ‘cohesive’ mechanisms of failure [50] (more prominent in the compression side of the laminate where the values of A/λ are larger) such as delamination deflection and accumulation of matrix cracks (as observed in sections A-A and B-B of Figure 4.9).

Figure 4.7c shows that the dent diameter ϕ before and after critical failure (load-drop) was larger in the Herringbone-Bouligand than in the Bouligand samples. As mentioned in § 4.3.1, we created the impact surface in the Herringbone-Bouligand samples by spreading a layer of a high-toughness high-impact-resistant epoxy-based adhesive characterised by a higher toughness compared to the CFRP composite impact surface of the Bouligand samples. This resulted in higher energy dissipation during the contact between the indenter and the Herringbone-Bouligand laminates, hence leading to a larger dent diameter ϕ . Additionally, the epoxy-based adhesive used to create the Herringbone-Bouligand impact surface is characterised by a higher capability of plastic deformation and lower Young’s modulus compared to the brittle and stiff CFRP impact surface of the Bouligand samples. This has promoted contact stresses redistribution [51] which might have prevented stress concentrations to localise at the peaks of the underlying Herringbone-Bouligand CFRP structure. This is in line with the absence of premature damage due to stress concentrations observed in the Herringbone-Bouligand laminates (see section A-A and B-B in Figure 4.9). We were therefore successful in mimicking the high-damage resistance of the impact surface of the mantis shrimp’s dactyl club [5] (described in § 4.1).

Overall, the effect of a tough impact surface, together with the disconnected delamination areas (sections A-A and B-B in Figure 4.9 and Figure 4.8b) and other sub-critical dissipative mechanisms such as:

- delamination deflection,
- accumulation of subcritical matrix cracks in the compression side, and
- the formation of several fully-developed sub-critical Bouligand matrix cracks in the tension side (analysed in depth in Chapter 3),

have contributed to increasing the sub-critical damage diffusion capability of Herringbone-Bouligand microstructures with respect to classical Bouligand layups, thereby enhancing the damage resistance to through-the-thickness loads.

4.5.1.2 The catastrophic failure and the reduction of in-plane spreading of damage in Herringbone-Bouligand structures

As discussed in §4.5.1.1, the Herringbone-Bouligand laminates showed a high through-the-thickness sub-critical damage diffusion characterised by the accumulation of disconnected delaminations and matrix cracks (Figure 4.9 and section A-A and B-B in Figure 4.8b). The lack of continuous delamination areas together with the interlocking mechanisms offered by the bi-sinusoidal interfaces of the Herringbone pattern led to a stiffer response (Figure 4.7a) and to a 10% increase in maximum load bearing capability with respect to Bouligand laminates (Figure 4.7b). However, the larger in-plane ($x - y$) spreading of delamination damage of the Bouligand samples was more effective in relieving the back-face tensile stresses, hence leading to a delay in the occurrence of fibre failure (displacement at load-drop 11% smaller in the Herringbone-Bouligand laminates).

Interestingly, for the Bouligand microstructure, Figure 4.8a shows that immediately after critical failure the delamination damage reached the clamping line, occupying most of the available testing area. This indicates that delaminations could have potentially extended further. On the contrary, the ability of the Herringbone-Bouligand laminates in resisting delamination formation and growth resulted in a delamination damage typically contained inside the Herringbone-Bouligand region. Overall, Herringbone-Bouligand laminates achieved a large reduction in total projected delamination area — 71% smaller than in the Bouligand laminates (Figure 4.7d). Additionally, Figure 4.8 shows that while fibre failure extended up to the clamp line in the Bouligand microstructure, this was entirely contained inside the Herringbone-Bouligand region in the Herringbone-Bouligand samples, further highlighting the high damage resistance of the Herringbone-Bouligand microstructure.

4.5.2 The full-penetration behaviour of Herringbone-Bouligand microstructures: a damage containment solution

Table 4.1 and Figure 4.7a show that, after critical failure has occurred, the lower extent of in-plane spreading of damage allowed Herringbone-Bouligand laminates to delay penetration at higher loads (11% increase in penetration load). As reported in Figure 4.7c, for the Herringbone-Bouligand laminates, the dent at this stage of the test was still larger than the one in the Bouligand laminates. This indicates that the presence of a tough impact surface, along with the ability of the Herringbone-Bouligand microstructure in accumulating damage in the compression side, avoided the localisation of failure due to contact throughout the entire duration of the test.

Figure 4.7b shows that the Herringbone-Bouligand microstructure dissipated 13% more energy during the entire fracture process than the Bouligand microstructure. Therefore, although in the Herringbone-Bouligand samples the in-plane extent of damage was greatly reduced, the highly-dissipative sub-critical failure mechanisms activating in Herringbone-Bouligand laminates (discussed in § 4.5.1) successfully increased the energy dissipation capability compared to ‘classical’ Bouligand structures.

Additionally, the analysis of the back face of a representative Herringbone-Bouligand laminate at the end of the penetration phase reported in Figure 4.10 reveals that the overall damage, including fibre failure, delaminations and matrix cracks, was successfully contained inside the Herringbone-Bouligand region. On the contrary, for the Bouligand laminates, damage extended up to the clamp line during penetration (Figure 4.10).

4.6 Conclusions

In this chapter, we have designed, prototyped, tested and analysed the first CFRP Herringbone-Bouligand microstructure, mimicking the full microstructure of the formidable damage-resistant biological hammer of the mantis shrimp. In order to manufacture the bespoke microstructure, we developed a novel prototyping procedure based on micro-moulding uncured prepreg composite materials. We compared the performances, under through-the-

thickness loads, of the Herringbone-Bouligand microstructure against a classical Bouligand layup with pitch angle $\Delta\theta = 2.5^\circ$ using quasi-static indentation tests.

Our test results show that the Herringbone-Bouligand microstructure achieved enhanced damage resistance as demonstrated by the following:

- highly reduced in-plane spreading of damage – 71% decrease in total projected delamination area;
- successful containment of the damage (including fibre failure, delaminations and matrix cracks) inside the Herringbone-Bouligand region;
- increase in peak load (10%), in penetration load (13%) and in total dissipated energy (13%) with a more significant through-the-thickness diffusion of damage;
- higher resistance to form delaminations, resulting in a delay in the onset of delamination damage (see Figure 4.8); and
- higher degree of through-the-thickness sub-critical damage diffusion achieved through:
 - the tough impact surface dissipating energy on the contact area between the indenter and the Herringbone-Bouligand laminate;
 - the tough bi-sinusoidal interfaces between consecutive plies defining the Herringbone pattern, which resulted in higher resistance to grow delamination damage and in the formation of mechanical interlocks between neighbouring plies. These important damage-resistant mechanisms promoted the formation of disconnected delamination areas, deflected delaminations and the accumulation of sub-critical matrix cracks.

In summary, we demonstrated the capability of Herringbone-Bouligand microstructures for diffusing damage, resisting delamination and containing failure within the Herringbone-Bouligand region. Therefore, this new CFRP microstructure constitutes an excellent tailorable damage-resistant solution with great potential for applications where resistance to through-the-thickness loads is paramount.

Additionally, we have demonstrated that the novel manufacturing procedure developed in this work can be used to locally tailor the microstructure of prepreg-based composite materials. Therefore, this novel technique can potentially be used to widen the design space for point-by-point tailorable solutions in the composite industry.

Chapter 5

An original multi-tailored bone-inspired design strategy: enhancing the damage resistance of SRPP/CFPP hybrid structures

5.1 Introduction

Self-Reinforced Polypropylene (SRPP) is well known for its good recyclability and processability, outstanding impact resistance [52, 53], high strain to failure [53] and high fracture toughness [54]. However, the low stiffness and yielding point of SRPP have hindered its use in various structural applications. Hybridising carbon fibres with SRPP provides a good combination of reasonable stiffness and high strain to failure [55, 56]. However, the use of carbon fibres, in addition to providing a challenge with recycling, increases the brittleness of the composite [56–59], hence compromising the excellent damage resistance of the SRPP.

The majority of the literature on Self-Reinforced Polypropylene/Carbon Fibre Polypropylene (SRPP/CFPP) has focused on the tensile [56–60] and impact behaviour [58,60] of various SRPP/CFPP hybrid composites. In comparison, no research has yet focused on investigating original strategies to improve the damage resistance of SRPP/CFPPs.

Studies on the tensile behaviour of continuous carbon fibre/SRPP hybrid composites have shown that the presence of carbon fibres, even in small volume fraction (3%), tends to localise damage at the carbon fibre layers, with the localisation being more severe as the carbon volume fraction increases [56,57]. The abrupt failure of the carbon fibres leads to a high energy release rate that generates delamination and unstable damage growth. Tensile tests on discontinuous carbon fibre/SRPP hybrid composites showed that a transition from a ductile to a brittle behaviour occurs at very low carbon fibre volume fraction ($<6.9\%$) [58]. Penetration impact tests conducted on the same hybrid composites showed that increasing the volume fraction of carbon fibre ($>5.5\%$) decreases the dissipated energy and leads to a brittle mechanical response [58]. In addition, the penetration occurs at lower indentation depths compared to SRPP laminates [58], hence reducing the damage resistance of the composite. Therefore, hybridising SRPP even with small volume fractions of carbon fibres in an inter-layer configuration leads to a localisation of damage near where carbon fibres fail, hence decreasing the damage resistance of the structure.

An enhancement in damage resistance can be achieved via locally engineering the microstructure using a bio-inspired approach. The concept of biomimetics can be defined as ‘borrowing ideas from nature for shaping and creating our surroundings’ [61]. Milwich et al. [61] highlights a fundamental aspect of biomimetics: biomimetics should be used to address engineering problems with the help of Nature’s wisdom without it being reduced to a blind mimic of the observed structures. Specifically to the bone microstructure [8], the hierarchical arrangement of staggered patterns of stiff mineral inclusions embedded in a soft matrix (also typical of nacre [7]) allows for the activation of several toughening mechanisms. Amongst them, the sliding of the stiff inclusions along one another during deformation results in a highly stable pull-out mechanism that stabilises failure and allow for large inelastic macroscopic strains [8]. Additionally, the staggered patterns of stiff inclusions lead to highly dissipative ‘staircase’-type of crack paths with high energy dissipation capability [7]. In this context, the presence of staggered arrangements of discontinuous stiff inclusions aligned along the loading direction has

been a successful source of inspiration for the design of CFRPs microstructure with enhanced damage resistance [23, 62–65].

Bullegas et al. [62] showed that the introduction of discontinuities across the load-carrying fibres via tailored laser-cut patterns in compact tension tests of cross-ply thin-ply CFRPs can lead to an improvement of the work of fracture of about 460% and to an increase of 43% in energy dissipation of quasi-isotropic thin-ply CFRPs under quasi static indentation. The occurrence of extensive pull-outs was found to be one of the major causes of such a large increase in energy dissipation (up to 64% of the total work of fracture). Narducci et al. [63–65] manufactured a nacre-like CFRP microstructure showing that the use of carefully designed discontinuities forming a tiled microstructure can lead to crack deflection, stable failure and damage diffusion under three-point bending of CFRPs and GFRP/CFRP tiled composites.

Based on the above [23, 62–65], as well as on other studies [66, 67], Tang et al. [59] showed that introducing discontinuities in the form of laser-cuts in the carbon fibre plies of a SRPP/CFPP tensile sample can successfully lead to a smooth and stable tensile response of the hybrid composite. Therefore, the use of discontinuities to locally engineer the microstructure of typically brittle composite materials can be a versatile tool to improve their mechanical and damage resistance performance.

We therefore decided to design two patterns of discontinuities, via laser-cuts across the load carrying fibres of CFPP plies, to improve two aspects of the damage resistance of a SRPP/CFPP continuous fibre Cross-Ply (CP) hybrid laminate: (i) increasing damage diffusion and hence energy dissipation and (ii) enhancing the impact damage resistance. Regarding the former, we assessed the toughness of the hybrid SRPP/CFPP composites and related energy dissipation capability using the Essential Work of Fracture (EWF) method [68–71] and Double Edge Notched Tensile (DEN-T) tests. Regarding the latter, we conducted low-velocity penetration impact tests to evaluate the performances of the hybrid composites under impact.

This chapter contains the following original contributions:

- (i) we devise the first multi-tailored material design concept by proving that patterns of discontinuities can be tailored to meet different damage resistance requirements within the same structure, while using the same technique to engineer the microstructure and the same material system;

- (ii) we provide a measure of fracture toughness and energy dissipation capability of SRPP/CFPP hybrid composites;
- (iii) we demonstrate that the use of carefully designed patterns of discontinuities in the CFPP plies of a SRPP/CFPP hybrid composite significantly improves the damage diffusion and hence the energy dissipation capability of the hybrid structure;
- (iv) we demonstrate that the use of carefully designed patterns enhances the impact damage resistance of SRPP/CFPP cross-ply structures; and
- (v) we provide an understanding of the role of pull-outs in the energy dissipation performances of engineered hybrid SRPP/CFPP structures in DEN-T tests.

5.2 Materials and methods

5.2.1 Materials

The hybrid SRPP/CFPP laminates comprise three basic constituents:

- (i) a Polypropylene (PP) tape fabric (an overfed twill 2/2 weave with an areal density of 130 g/m^2 supplied by Propex Fabrics GmbH) [72]. The compacted SRPP has a thickness of about $141 \text{ }\mu\text{m}$;
- (ii) a PP film of the same PP grade as the tapes in the fabric. This is $20 \text{ }\mu\text{m}$ thick and 0.92 g/cm^3 in density. More details on the properties of these tapes and films, how their melting temperatures are different, and how they change after hot compaction can be found in the following two references [73, 74]; and
- (iii) carbon fibres (T700SC-50C) supplied in a Unidirectional (UD) spread tow tape from Chomarat. The fibres are held together by fibre sizing [59]. The tape has an areal density of 71.5 g/m^2 .

5.2.2 Design of DEN-T and impact samples

We manufactured two pairs of configurations respectively for DEN-T and impact tests. Each pair comprises a baseline (without laser-cuts and identical for both DEN-T and impact

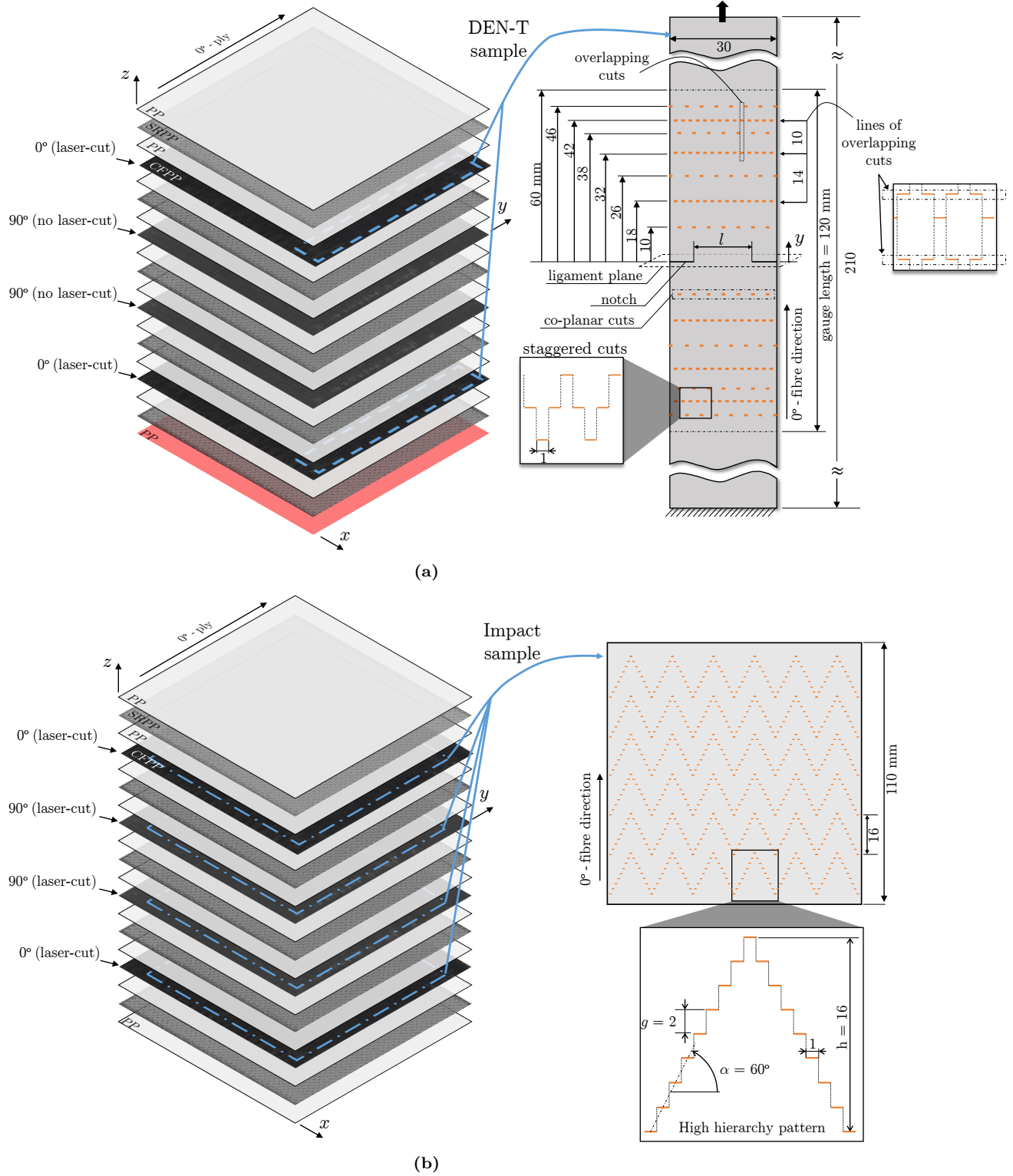


Figure 5.1: Lay-up and laser-cuts pattern for (a) DEN-T and (b) impact samples.

tests) and an engineered configuration (with two laser-cut patterns tailored for DEN-T and impact respectively). The stacking sequence and geometry for the DEN-T and impact samples

are schematically reported in Figure 5.1a and Figure 5.1b respectively. We chose a cross-ply lay-up with the following stacking sequence: $[F/S/F/C_{0^\circ}/F/S/F/C_{90^\circ}/F/S]_{so}$ ¹ where F stands for PP film, S for SRPP and C for a layer of UD CFPP prepreg. We preferred a dispersed lay-up over other inter-layer architectures as it improves the impact and tensile performance of the composite [58]. We interleaved a PP film in between each CFPP ply and SRPP ply to guarantee excellent hot compaction quality [59, 75, 76].

In the DEN-T samples, the load is aligned with the fibres of the C_{0° plies. Furthermore, the C_{90° plies do very little work during the failure of the laminate compared to the C_{0° plies [77, 78]. Therefore, in the DEN-T samples, only the C_{0° plies of the engineered microstructure contains laser-cuts. In the engineered configuration of the impact samples, the C_{90° plies have the same function as the C_{0° plies; therefore, all the carbon fibre plies contain laser-cuts.

In the DEN-T test, we designed the samples such that the C_{0° layers (Figure 5.1a), i.e. the ones containing laser cuts, were the most external carbon fibre layers in the lay-up. This was intended to facilitate observation of the evolution of damage, taking advantage of the semi-transparency of the SRPP. In addition, to monitor strain evolution via DIC on the DEN-T samples, we substituted one of the two most external PP films with an equivalent (same chemical and mechanical properties) red one (Figure 5.1a). The reduced transparency of the red PP film allowed for an enhanced contrast with the black speckle pattern, thereby increasing the quality of the strain measurements. For the impact samples, this was not needed because DIC was not performed during these dynamic tests. Additionally, a translucent PP film enabled a more clear observation of the post-impact damage.

5.2.3 Design of the laser-cut patterns

5.2.3.1 DEN-T samples

Figure 5.1a schematically shows the pattern of laser-cuts designed for the DEN-T samples with engineered microstructure. The pattern can be broken down into three lines of dense laser-cuts, each one neighboured by two coarse lines. The distance between each line of dense cuts and two neighbouring coarse lines decreases as y increases. The resulting pattern is then mirrored about the ligament plane, resulting in a symmetric sample.

¹SO=symmetric odd, i.e. without doubling the mid-plane layer

The choice of cut length, the spacing between co-planar cuts and the relative spacing between overlapped lines of cuts in Figure 5.1a follow the outcomes of a previous parametric studies conducted by Tang et al. [59, 79, 80] on un-notched tensile SRPP/CFPP samples comprising one layer of UD CFPP layer packed between six layers of SRPP.

Specifically, we chose the cut length based on the following observations [59, 79]: (i) a larger cut length leads to a larger cut opening during manufacturing, hence decreasing the control achieved over the microstructure; (ii) a smaller cut length results in a smaller distance between co-planar cuts. A smaller distance between co-planar cuts promotes interaction between adjacent discontinuities, leading in turn to increased stress concentrations at the bundle root and hence promoting bundle fracture over bundle pull-out. In this work, we designed the pattern to promote bundle pull-out over bundle fracture, aiming at achieving a stable highly-dissipative failure process. Additionally, limiting the cut opening during manufacturing was desirable to achieve a good control over the microstructure. Based on the above, including quantitative details in [59, 79] we chose a 1 mm cut length.

For each half of the sample (above and below the ligament plane) we decided to use three units of staggered cuts with two levels of hierarchy each. This allowed to alternate one line of co-planar cuts with higher cut-density (i.e. number of laser-cuts per sample width [59, 79]) to a line with lower cut-density. This was shown to achieve a good balance between strain de-localisation capability and load-bearing capability [80]. We designed the pattern such that there were no continuous fibre bundles in between adjacent cuts (along the transverse direction), aiming at preventing fracture of continuous CF bundles which, in turn, would have led to localised failure rather than bundle pull-out. Additionally, Tang et al. [59, 80] showed that when the distance between two overlapped cuts increases (larger than 16 mm), the longitudinal tensile stress fully recovers within the discontinuous bundles formed by the two overlapped cuts. Therefore, the two lines of overlapped cuts act independently. Since, in the DEN-T samples, we want to promote interaction between the laser-cuts to maximise damage diffusion, in our design we progressively reduced the distance between two lines of overlapping cuts as the distance (y) from the ligament plane increases. This was expected to promote damage initiation in areas of the sample far from the notches and then to grow towards the ligament plane promoting extensive damage diffusion.

5.2.3.2 Impact samples

Figure 5.1b schematically shows the pattern of laser-cuts designed for the impact samples with engineered microstructure. We designed the patterns with a 1 mm cut length (see § 5.2.3.1). The pattern can be broken down into a triangular unit of staggered cuts. The unit is repeated along the transverse direction to form a ‘shark-teeth’ pattern (inspired by the work of Bullegas et al. [81]) and then along the longitudinal direction with a step of 16 mm (see Figure 5.1b). We decided to use a relatively high hierarchy level, i.e. five in this work, in order to (i) create highly deflected crack paths aiming at spreading the damage caused by the indenter; and (ii) to create large pull-out interfaces aiming at preserving the load-bearing capability of the composite.

We designed the pattern such that there were no continuous fibre bundles in between adjacent cuts (along the transverse direction). This was expected to promote energy dissipation at sub-critical level (i.e. before the occurrence of the load-drop which corresponds to fibre failure). We chose the value of g and α in Figure 5.1b based on the following considerations: (i) the height of a single unit should be smaller than the striker diameter ($\phi = 20$ mm) and (ii) at least 4 units should be inside the area delimited by the inner diameter of the clamps ($\phi = 40$ mm). In the engineered impact samples, a full recovery of stress along the discontinuous bundles during loading was desirable to preserve the load-bearing capability of the engineered hybrid samples. Therefore, we chose a spacing of 16 mm between the staggered units (see § 5.2.3.1).

5.2.4 Manufacturing

We manufactured the hybrid SRPP/CFPP plates in three steps: (i) prepregging of the carbon fibre layer, (ii) laser-cutting of the prepregs and (iii) hot pressing of the hybrid laminate.

In step (i), we hot-pressed a 250 mm x 250 mm preform of UD carbon fibres sandwiched between two PP films. The same procedure was applied to all the carbon fibre plies. The hot pressing of the prepreg was carried out using a Fontijne Grotnes LabPro 400, preheated at 188 °C. We applied a surface pressure of 5 bar for 5 min and then cooled the prepreg down to 40 °C in 5 min.

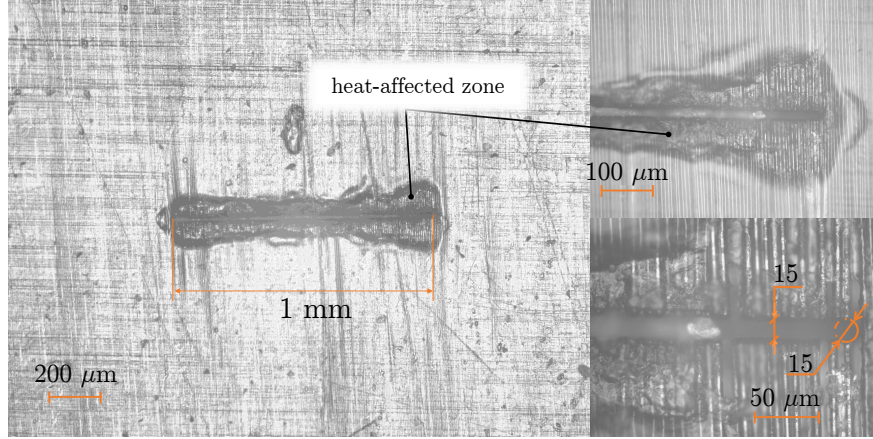


Figure 5.2: Detail of a laser-cut on a CFPP prepreg.

In step (ii), we performed the laser-cuts using an Oxford Lasers Diode Pumped Solid State (DPSS) micro-machining system. The high precision laser-milling ensured excellent quality of the cuts and high reproducibility. As shown by the micro-graphs of a representative cut in Figure 5.2, we managed to produce neat and precise cuts with a constant thickness and a round neat tip of the cut-end with a diameter of 15 μm . The heat generated during cutting, created only a very small heat-affected zone around the cut (Figure 5.2).

In step (iii), we laid-up the hybrid laminate with the stacking sequence of § 5.2.2 and then hot-compacted the 250 mm x 250 mm plate at 188 $^{\circ}\text{C}$ for 5 min with an applied surface pressure of 39 bar. We did not use any frame during the hot compaction of the laminates. The higher pressure was necessary to prevent the shrinkage of PP tapes [59]. The composite plate was then cooled down to 40 $^{\circ}\text{C}$ in 5 min. The final thickness of all the plates ranged between 1.14 mm and 1.18 mm with an estimated carbon volume fraction of 14%. We then cut each plate to the final sample dimensions (210 mm x 30 mm and 110 mm x 110 mm for DEN-T and impact tests respectively) using a bandsaw. Finally, we pre-notched the DEN-T samples using a wire hand-saw and used a razor blade to create a sharp tip with a radius of 125 μm . For each configuration, we manufactured 15 DEN-T samples with nominally evenly-spaced ligament lengths ranging between 5 mm and 19 mm. We further manufactured three nominally-equivalent impact samples for the baseline and engineered configuration, respectively.

5.2.5 Essential Work of Fracture and DEN-T tests

We performed quasi-static DEN-T tests on an Instron 5567 universal test machine with a 30 kN load cell and a displacement rate of 2 mm/min using a gauge length of 120 mm. The surface of the samples with the red external PP layer was covered with ink-based black speckles (for DIC). We preferred ink to standard paint-based speckles to avoid premature fracture of the paint due to the high failure strain of SRPP. We used two digital cameras to record both the front and back surfaces of the sample during test. The optimal DIC correlation parameters included a subset size of 19 pixels and a step size of 6 pixels.

We used the EWF method [68–71] on the load vs. displacement diagrams of the DEN-T samples to assess the fracture toughness and energy dissipation capability of the baseline and engineered microstructures. The EWF method, developed for highly ductile polymers such as polypropylene, was preferred to J-Integral and crack tip opening displacement because it provides a clear distinction between the energy dissipated at the ligament plane (essential part) and the work dissipated in the volume of the sample (non-essential part) [82]. The first corresponds to a measure of fracture toughness while the second correlates with the damage diffusion and energy dissipation performances of the structure.

The EWF method can be applied if the following requirements are met [71]:

- self-similarity of the load vs. displacement diagrams;
- full ligament yielding prior to crack growth; and
- plane stress condition and outer ‘plastic’ volume proportional to the square of the ligament length (l^2).

where the last two conditions are met if the maximum net section stress is independent of the ligament length (Hill’s criterion [83]).

The work of fracture (W_{fracture}) for a DEN-T sample with a certain ligament length is the integral of the load vs. displacement diagram. This can be written as:

$$W_{\text{fracture}} = W_{\text{essential}} + W_{\text{plastic}} \quad (5.1)$$

with $W_{\text{essential}}$ being the energy dissipated at the fracture plane (ligament plane) and W_{plastic} the energy dissipated in the volume surrounding the ligament plane ('plastic' region). Assuming that the volume of the 'plastic' region scales with l^2 , Eq. 5.1 can be rewritten as follows:

$$W_{\text{fracture}} = w_{\text{essential}} \cdot lt + \beta w_{\text{plastic}} \cdot l^2 t \quad (5.2)$$

$$w_{\text{fracture}} = \frac{W_{\text{fracture}}}{lt} = w_{\text{essential}} + \beta w_{\text{plastic}} \cdot l \quad (5.3)$$

where t is the sample thickness and β is the shape factor of the 'plastic' region. $w_{\text{essential}}$ and w_{plastic} , independent of l , are respectively an energy per unit surface and an energy per unit volume. The intercept of the trend-line of Eq. 5.3 for zero ligament length, $w_{\text{essential}}$, is a measure of fracture toughness. The slope of the trend-line βw_{plastic} is a measure of the damage diffusion and energy dissipation performances of the sample. Since βw_{plastic} is associated to the shape of the 'plastic' region, it depends on both the material and the geometry of the sample.

5.2.6 Penetration impact test

We performed penetration impact tests on square samples of 110 mm x 110 mm clamped with a circular clamp of outer and inner diameter of 60 mm and 40 mm respectively, and a clamping pressure of 1780 kPa on the sample surface. We impacted the samples with an hemispherical ($\phi = 20$ mm) striker with a weight of 26.17 kg. The striker was dropped from a height of 1 m, thereby impacting the sample with an energy of 256.72 J. The impactor reached the sample surface with a speed of 4.42 m/s.

We quantified the impact performance of the hybrid composites by computing the total dissipated energy and the energy dissipated before the peak load. For each sample, we estimated the two dissipated energies by computing the area under the load vs. displacement diagram, where the displacement represents the displacement of the impactor.

5.3 Results

5.3.1 DEN-T tests results

For the DEN-T tests of the baseline samples (i.e. samples without laser-cuts) with ligament lengths $5 < l < 19$ mm, Figure 5.3a shows self-similar load vs. displacement diagrams. Figure 5.3b shows that the maximum load scales linearly with the ligament length—Hill's criterion applies. Therefore, the requirements for the application of the EWF were met. In Figure 5.3c, the work of fracture W_{fracture} is plotted against the ligament length l . This is well fitted by a quadratic polynomial as expected from Eq. 5.2. Figure 5.3d shows the specific work of fracture w_{fracture} plotted against l . The data are distributed linearly as expected from

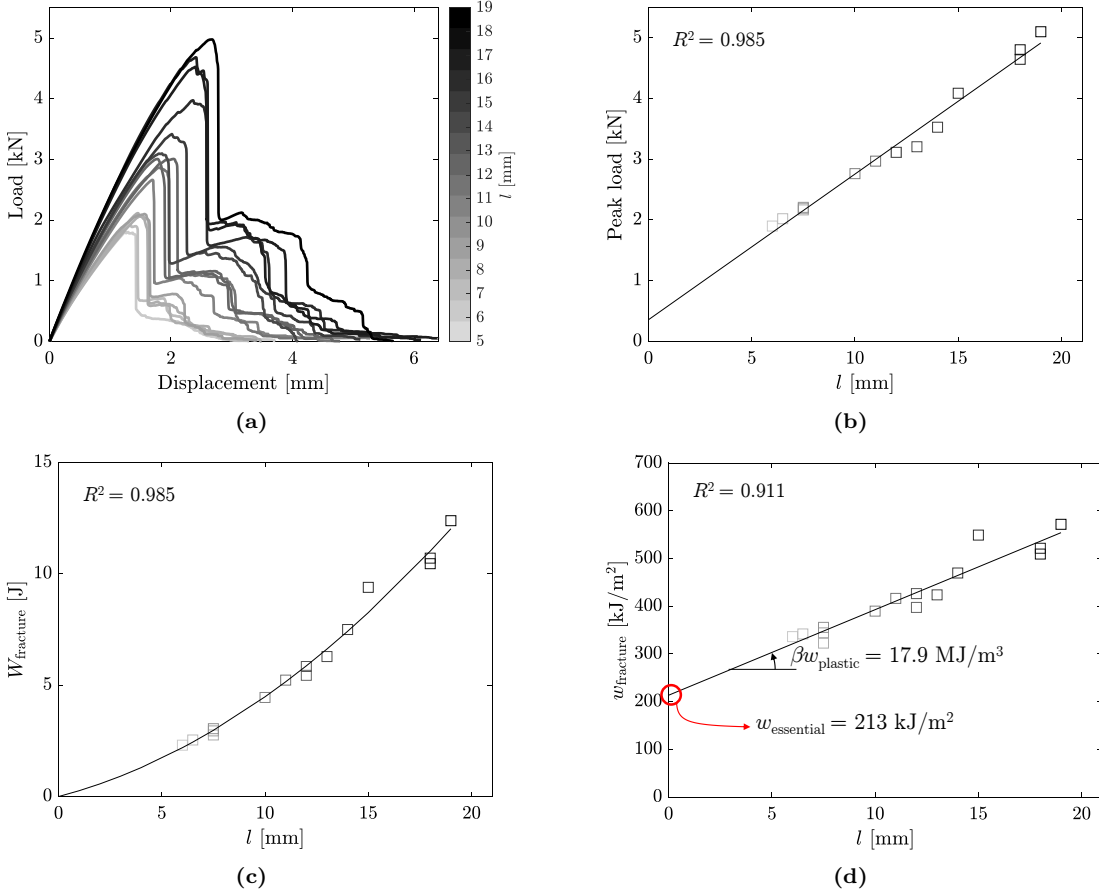


Figure 5.3: DEN-T test results for the baseline configuration, i.e. without laser-cuts (a) load vs. displacement diagrams, (b) linear fitting of the peak load with respect to the ligament length and associated R^2 , (c) quadratic fitting of the work of fracture and associated R^2 and, (d) linear fitting of the specific work of fracture and associated R^2 .

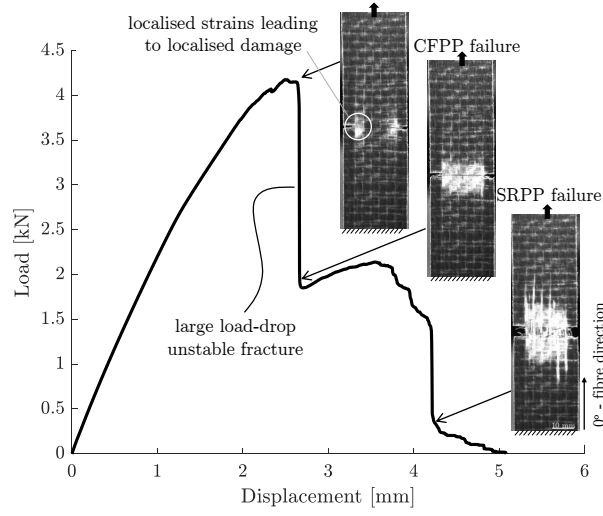


Figure 5.4: Load vs. displacement diagram highlighting the fracture process of a DEN-T sample of the baseline configuration with $l = 17$ mm.

Eq.5.3. The trend-line obtained from the linear regression intercepts the zero-ligament length axis at 213 kJ/m^2 with a slope of 17.9 MJ/m^3 .

The load vs. displacement diagram and photographs of a baseline sample with $l = 17$ mm in Figure 5.4 show that damage (whitening in Figure 5.4) was highly localised at the notch tips right before the unstable damage propagation (load-drop). The unstable damage propagation is mainly associated with the sudden failure of the CFPP plies. The load-drop is followed by a short load rising phase until the final failure of the SRPP layers.

For the DEN-T tests of the engineered configuration, Figure 5.5a shows the load vs. displacement diagrams of the engineered samples (together with those of the baseline). The engineered microstructure, despite a consistent reduction in the maximum load achieved during the test, showed a gradual load vs. displacement response. In addition, the engineered microstructure reached higher loads than the baseline during damage growth (after the load drop occurred in the baseline) and higher ultimate displacements, as shown in Figure 5.5b. Specifically, for $l > 16$ mm, the increase in failure displacement was 190% higher than the displacement at the onset of unstable damage growth in the baseline configuration (CFPP failure) and 50% higher than the displacement calculated at the final failure of baseline samples with similar ligament lengths.

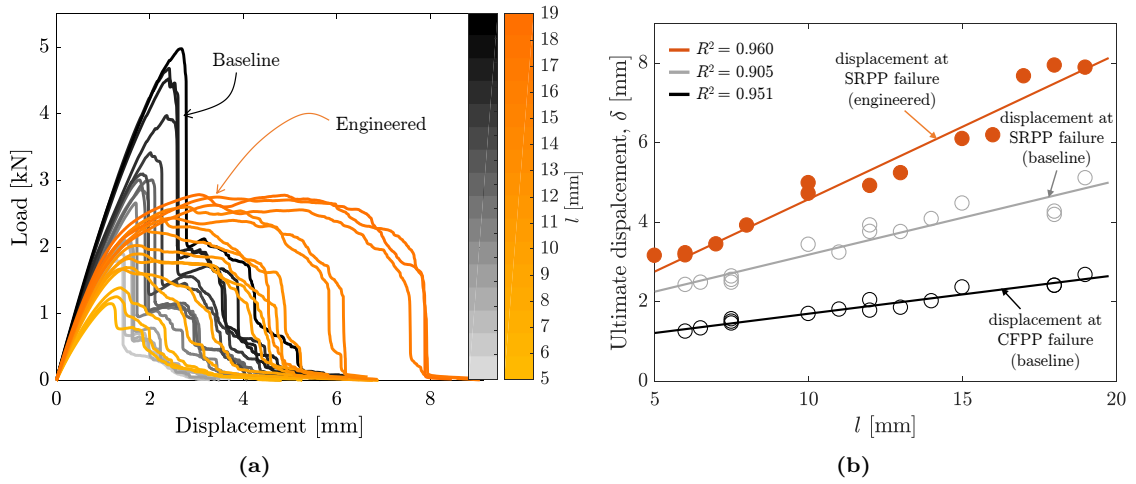


Figure 5.5: Comparison between (a) the load vs. displacement diagrams of the baseline and the engineered structure and (b) failure displacement for different ligament lengths of the baseline and engineered structures. The black line refers to the ultimate displacement at the CFPP failure of the baseline (load-drop) while the gray line refers to the ultimate displacement at the SRPP failure (displacement at 90% drop of the peak load) of the baseline.

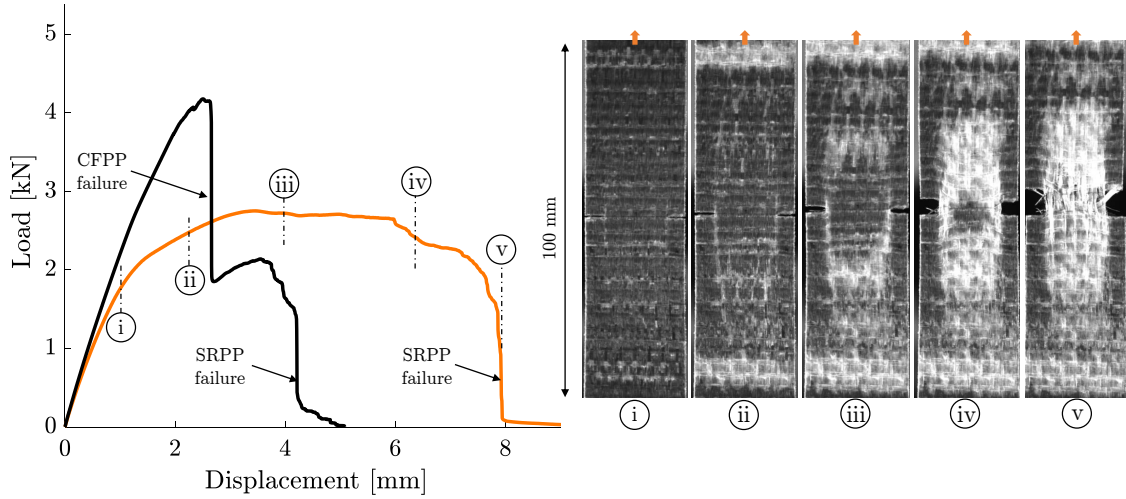


Figure 5.6: Load vs. displacement diagram highlighting the fracture process of a DEN-T sample of the engineered configuration with $l = 17$ mm.

Figure 5.6 shows the load vs. displacement diagram and photographs of an engineered DEN-T sample with $l = 17$ mm highlighting the highly diffused damage occurring in the engineered microstructure.

Figures 5.7a and 5.7b respectively show a comparison between the work of fracture and the specific work of fracture of the baseline and engineered configurations. Specifically, Figure 5.7b highlights the 90% increase in the slope of the specific work of fracture of the engineered configuration compared to the baseline.

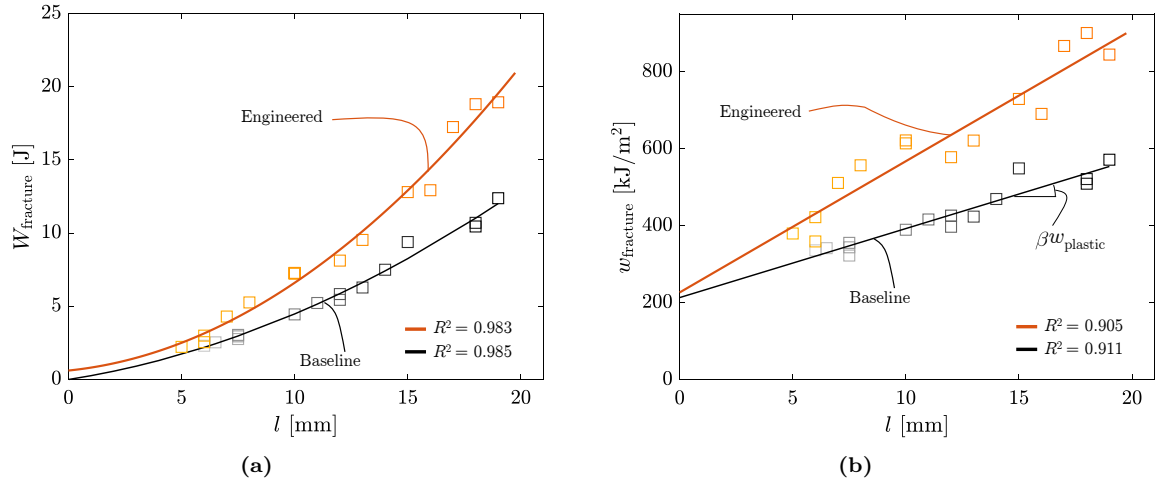


Figure 5.7: Comparison between (a) the work of fracture and (b) specific work of fracture of baseline and the engineered microstructure.

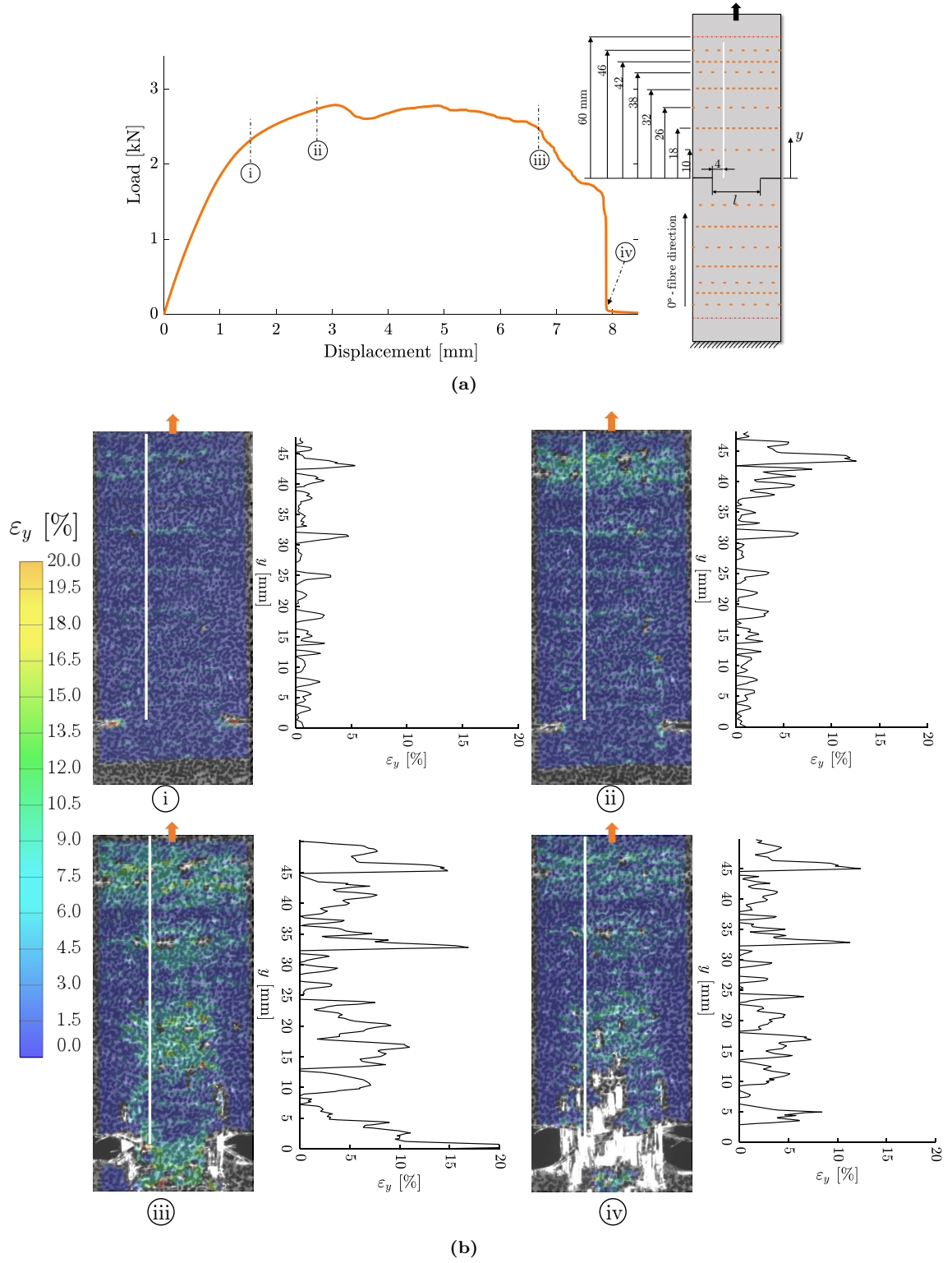


Figure 5.8: (a) load vs. displacement of a DEN-T sample with engineered microstructure and $l = 18 \text{ mm}$ and schematic of the sample. (b) ϵ_y obtained from DIC of the DEN-T sample shown in (a) taken along a longitudinal path at different instances during the test. For clarity, we report only half of the sample in each DIC map.

Figure 5.8 shows the DIC strain maps of an engineered sample with $l = 18$ mm. The graphs in Figure 5.8b represent the tensile strain ε_y evaluated along the white line in Figure 5.8a. The large peaks, which correspond to high strain regions, match very well with the physical position of the laser-cuts along the sample.

5.3.2 Low-velocity penetration impact tests results

Figure 5.9 shows the load vs. displacement diagrams of the penetration impact tests for the baseline and engineered microstructures. The load-drop of the engineered samples was delayed to larger applied displacements. The increase was found to be 26% on average with respect to the baseline in Figure 5.10a, and to be statistically significant (p -value = 0.0305, calculated with the One-sample t -test and a standard 5% significance level). In addition, Figure 5.9 shows that the engineered microstructure preserved the load-bearing capability of the baseline. The energy dissipated prior to the load drop in Figure 5.10b was found to be on average 42.7% higher for the engineered configuration than for the baseline configuration, and to be statistically significant (p -value = 0.0005). The total dissipated energy was very similar for the two configurations with a statistically non-significant average difference of 3.2% (p -value = 0.178).

In Figures 5.11 and 5.12, photographs taken from the surface of impacted baseline and engineered samples, respectively, reveal that damage area (whitening in the figures) was increased by 43% in the engineered configuration with respect to the baseline. The threshold used in

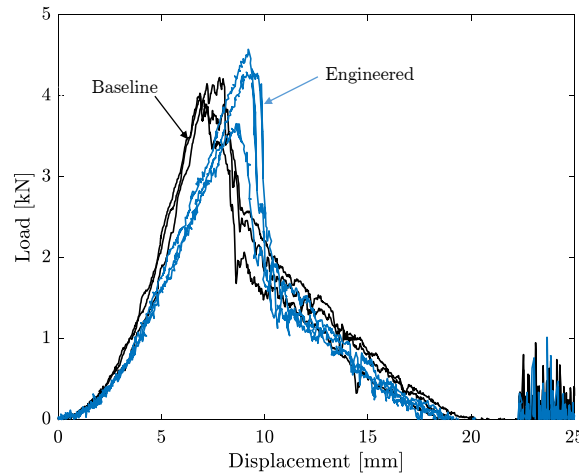


Figure 5.9: Load vs. displacement of the baseline and the engineered SRPP/CFPP hybrid composite.

the binarization process of the impact surface (in Figures 5.11c and 5.12c) was the same for engineered and baseline samples.

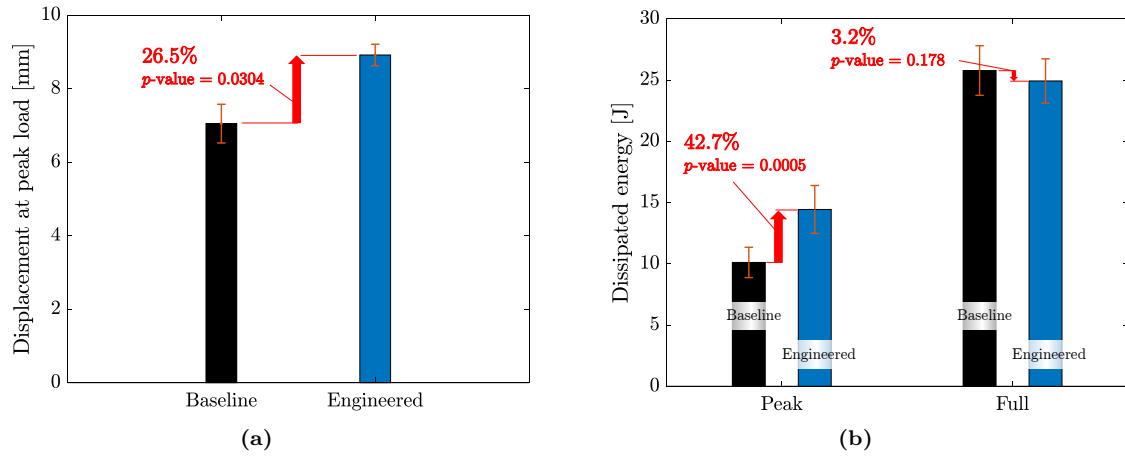


Figure 5.10: Average values and associated standard deviations of (a) displacement at fibre failure, corresponding to the initiation of the penetration of the sample and, (b) dissipated energy calculated before peak-load (sub-critical phase) and total dissipated energy of the baseline and the engineered SRPP/CFPP hybrid composite.

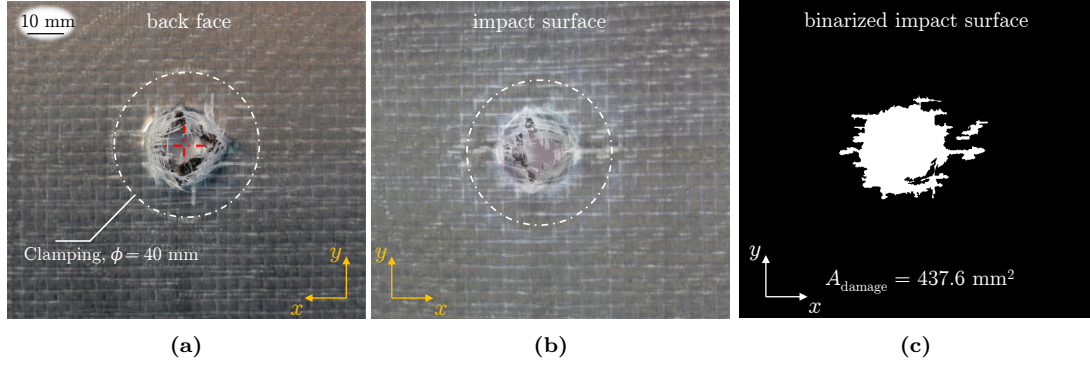


Figure 5.11: Photographs of (a) the back face and (b) the impact face of a baseline impact sample. (c) Binarized image of the impact surface of a baseline impact sample. The white area corresponds to damage.

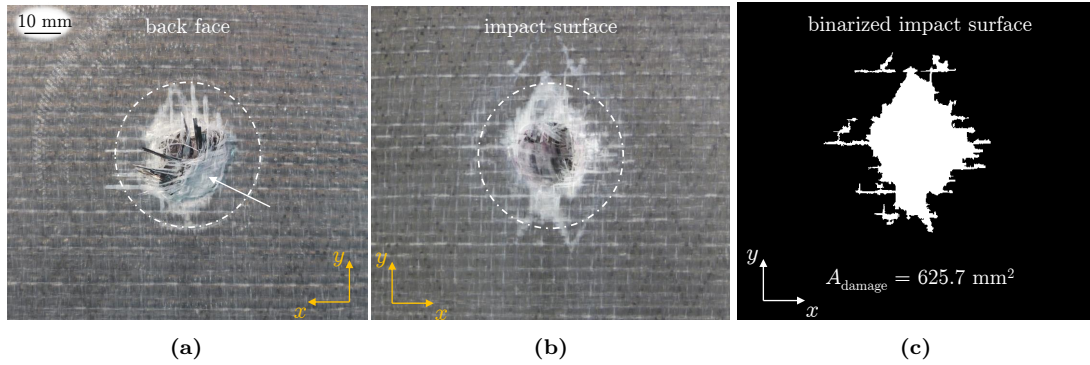


Figure 5.12: Photographs of (a) the back face and (b) the impact face of an engineered impact sample. (c) Binarized image of the impact surface of an engineered impact sample. The white area corresponds to damage.

5.4 Discussion

5.4.1 The essential work of fracture of hybrid SRPP/CFPP structures

From the application of the EWF to the baseline SRPP/CFPP cross-ply DEN-T samples (Figure 5.3), we obtained an estimate of fracture toughness ($w_{\text{essential}} = 213 \text{ kJ/m}^2$) and a measure of the energy dissipation capability of the sample, $\beta w_{\text{plastic}} = 17.9 \text{ MJ/m}^3$. The latter is associated with the whitening in Figure 5.4 and includes delamination of the CFPP layers, PP tape plastic deformation, debonding and fibrillation. βw_{plastic} depends on the specimen geometry [82], yet it provides an important measure of the capability of the structure to dissipate energy in a sub-critical way, shielding the ligament plane where critical crack propagation occurs. Therefore, within a design space where damage resistance is a relevant requirement, high values of both βw_{plastic} and $w_{\text{essential}}$ are desirable.

We compared the baseline configuration with the results of several materials gathered from the literature [54, 78, 82, 84] in Figure 5.13. In this figure, the x - and y -axis correspond to $w_{\text{essential}}$ and βw_{plastic} respectively. Figure 5.13 shows that hybridising SRPP with continuous carbon fibres has resulted in a tough material with a good capability of energy dissipation relatively to other materials in the literature.

Further analysis of the damage evolution in a representative baseline sample with $l = 17 \text{ mm}$ in Figure 5.4 shows that, prior to the load-drop, strains are highly localised around the notch tip (bright whitening). This has led to the localisation of damage close to the ligament plane and hence, to the abrupt failure of the CFPP plies (load-drop in Figure 5.4). Therefore, we infer that the high strain localisation of the baseline microstructure has limited the capability of diffusing damage away from the ligament plane.

5.4.2 Enhancing the damage diffusion of hybrid SRPP/CFPP structures

In this section we will discuss how engineering the microstructure with tailored laser-cut patterns promotes extensive damage diffusion and increases volumetric energy dissipation.

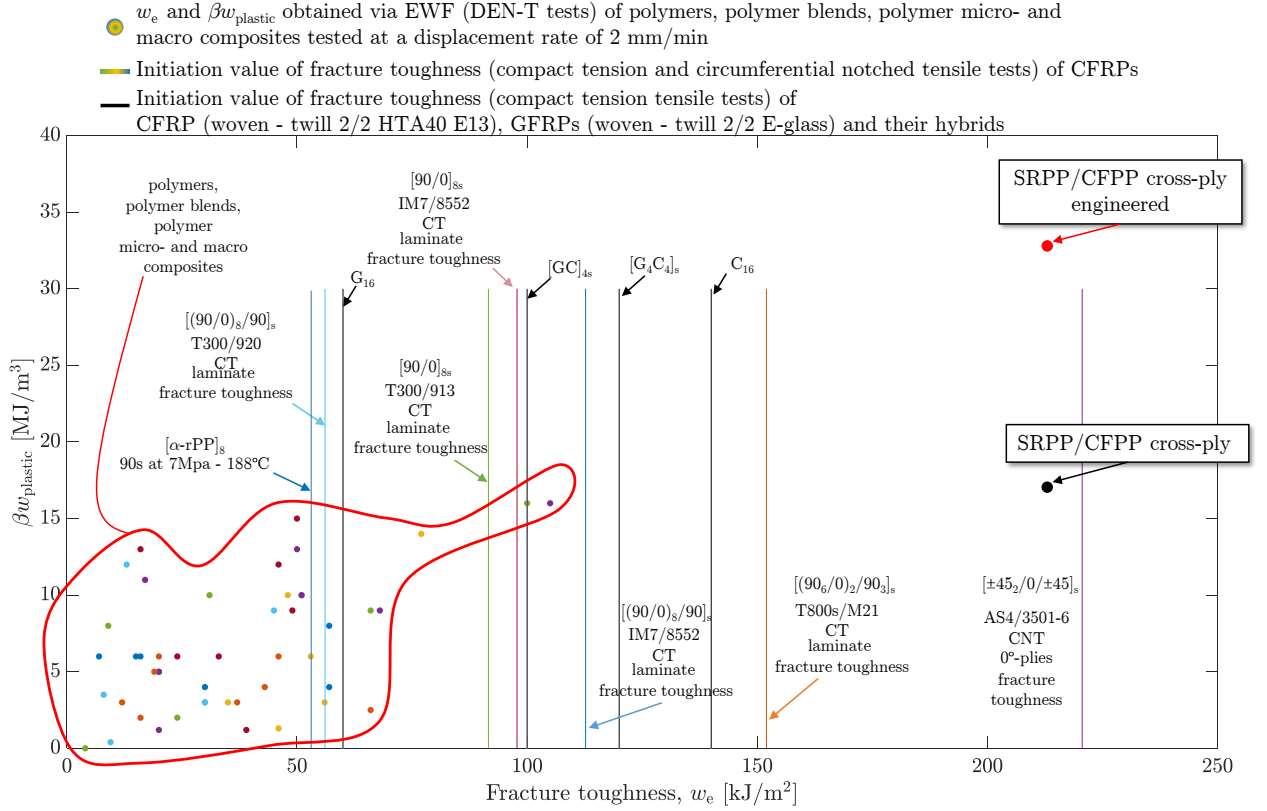


Figure 5.13: βw_{plastic} vs. $w_{\text{essential}}$ bench-marking of polymers, polymer blends, polymer composites, Carbon fibre/Epoxy, GF/Epoxy and Carbon fibre/GF-Epoxy composites. The full coloured dots refer to a large variety of polymers (DEN-T tests at 2 mm/min displacement rate) [54,82]. The vertical lines refer to the initiation value of fracture toughness for Carbon fibre/Epoxy composites [78] and Carbon fibre/Epoxy, GF/Epoxy, Carbon fibre/GF-Epoxy woven composites [84] (Compact Tension (CT) and Centre Notched Tension (CNT) tests). Given the nature of the test, it is not possible to give a measure of βw_{plastic} , which for this data-set has not been reported. The two black and red large dots on the right side of the plot refer to the composites tested in this study.

Figure 5.5a shows that the engineered microstructure successfully achieved a stable failure process characterised by a gradual stiffness degradation and a load plateau of increasing length with the ligament length. As shown in Figure 5.8b(i-ii), damage firstly developed in regions far from the ligament plane (high cut density regions); therefore, strain concentrations around the ligament region were successfully delocalised. Then, damage gradually migrated towards the ligament plane as shown in Figures 5.6 and 5.8. The resulting extensive damage diffusion protected the ligament plane, thereby delaying final failure. In addition, the DIC maps of Figure 5.8b reveal that ε_y on the SRPP outer layers never exceeded 17.5%, i.e. about 85% of the failure strain of SRPP [59]. Therefore, we can infer that damage diffusion, in the form of plastic deformation of the PP tape, PP debonding and pull-outs, occurred at a sub-critical level.

The extensive diffused damage has resulted in an increase of about 90% of the slope of the specific work of fracture (w_{fracture}) with respect to the baseline configuration (Figure 5.7b). Although the slope of w_{fracture} is a geometry-dependent property, we can still use it to compare baseline and engineered samples since the specimens have the same geometry. Therefore, the increase in slope can be interpreted as an inherent increase in the diffused energy dissipation capability of the structure.

The engineered microstructure increased the diffused energy dissipation (Figure 5.7a). However, in the samples with engineered microstructure, we do not change the material in the ligament, and hence we expect that the essential work of fracture should not be altered; this is consistent with the experimental observation in Figure 5.7b.

Using the comparison map of Figure 5.13, the engineered microstructures shows both high fracture toughness ($w_{\text{essential}}$) and high damage diffusion capability. Therefore, tailoring a pattern of discontinuities in the microstructure has greatly enhanced the damage resistance of the hybrid SRPP/CFPP composite.

5.4.3 The role of pull-out of carbon fibre bundles in the enhanced energy dissipation of the engineered hybrid SRPP/CFPP structure

In § 5.4.2, we discussed the enhanced damage diffusion of the engineered microstructure and the related increase in energy dissipation (90% increase of the slope of w_{fracture} with respect to the baseline in Figure 5.7b). In this section, we will discuss the role of pull-out of carbon fibre bundles as the main dissipative mechanism that caused this increase.

As mentioned in § 5.2.5, the EWF method assumes that the area of the ‘plastic’ region (A_{ultimate}) scales with l^2 ; therefore, W_{plastic} is proportional to l^2 ($W_{\text{plastic}} \propto l^2$). For the engineered configuration, the volumetric work of fracture (W_{plastic}) results from the contribution of: (i) debonding, plastic deformation and fibrillation of the PP tapes ($W_{\text{plastic,PP}}$) and (ii) pull-outs of carbon fibre bundles (W_{pullout}).

In this work, we can estimate the area of the ‘plastic’ region A_{damage} contributing to $W_{\text{plastic,PP}}$ at each moment during the test using the whitened area (due to local debonding, fibrillation and plastic deformation of the PP tapes) observable from the surface of the sample.

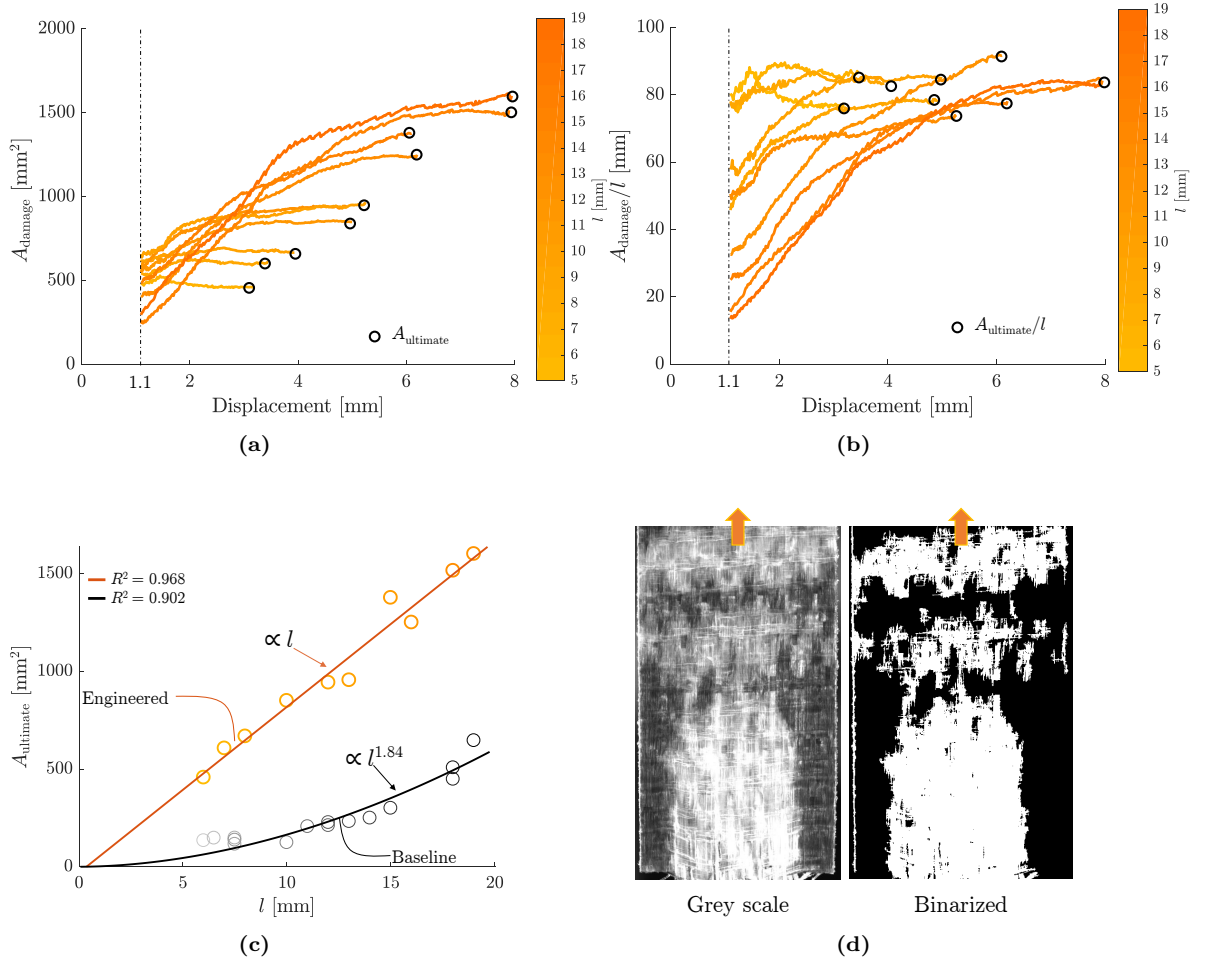


Figure 5.14: Image analysis of the ‘plastic’ region of DEN-T samples with engineered microstructure. (a) Area of damage vs. displacement diagrams, (b) Area of damage normalised by the ligament length, (c) ultimate area of damage (calculated at specimen failure) for the engineered and baseline microstructure and (d) example of grey scale image recorded during test and corresponding binarized image. The white area in the binarized image indicates damage.

We conducted several image analyses of the surface of the samples (Figure 5.14d) to quantify A_{damage} as a function of the applied displacement (see Figure 5.14a). As it is shown in Figure 5.14a, for each sample we report the surface damage from an applied displacement of 1.1 mm until the final failure. Damage at an applied displacement smaller than 1.1 mm is not reported because it is affected by the noise in the image due to the poor contrast between the little damage formed at this early stage and the undamaged region of the sample. In Figure 5.14b we divided A_{damage} by the respective ligament length. Interestingly, it appears that the curves tend to similar values of A_{ultimate}/l , independently of the ligament length. This implies that, for the samples with engineered microstructure, $A_{\text{ultimate}} \propto l$ (Figure 5.14c); therefore since $W_{\text{plastic}} \propto A_{\text{ultimate}}$, then $W_{\text{plastic,PP}} \propto l$. This finding can be explained as

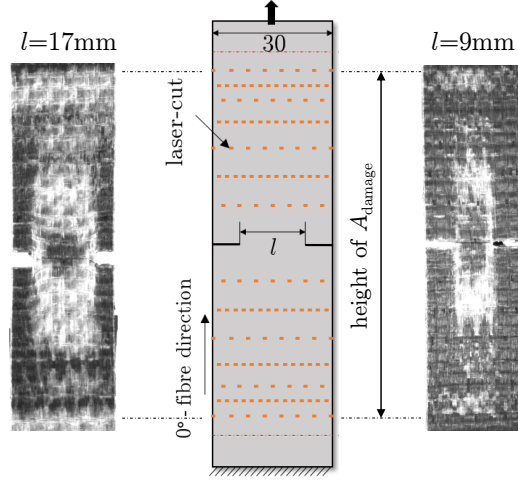


Figure 5.15: Two photographs of DEN-T samples with engineered configuration with ligament length $l = 17$ mm and $l = 9$ mm showing that the height of A_{damage} is bounded to the location of the laser-cuts.

follows: the presence of laser-cuts (fixed position along the sample length) delocalises damage which is therefore forced to develop around them. Consequently, being the height of the ‘plastic’ region bounded by the laser-cuts (see Figure 5.15) and therefore independent of the ligament, A_{ultimate} scales linearly with l .

The work done by pull-outs (W_{pullout}) is proportional to the number of laser-cuts involved in the failure process (N_{cuts}) and to the ultimate displacement δ calculated at the sample final failure (under the assumption, supported by experimental evidence in Figure 5.6, of a nearly simultaneous activation of the pull-out mechanism in all the laser cuts involved in the failure process). Therefore, $W_{\text{pullout}} \propto N_{\text{cuts}} \cdot \delta$. The number of cuts, from which bundles of carbon fibre can be pulled-out, is proportional to l . In addition, given the self-similarity of the load vs. displacement diagrams, $\delta \propto l$ (Figure 5.5b). Therefore, even without deriving a full expression for W_{pullout} , we know that $W_{\text{pullout}} \propto l^2$.

Combining the analysis conducted in this section ($W_{\text{plastic,PP}} \propto l$ and $W_{\text{pullout}} \propto l^2$) with the results shown in Figure 5.7b, it becomes clear that the major contribution to the large slope of w_{fracture} vs. l of the engineered microstructure is due to the extensive pull-out of carbon fibre bundles.

5.4.4 Enhancing the impact damage resistance of hybrid SRPP/CFPP structures

The load vs. displacement diagrams of baseline and engineered microstructures under penetration impact in Figure 5.9 show that the engineered microstructure succeeded in delaying critical failure (the penetration, which occurs at the peak-load [58]) to larger applied displacement (average increase of 26.6% with respect to the baseline in Figure 5.10a).

The designed pattern of laser-cuts created a large number of discontinuous carbon fibre bundles, each one 16 mm long, thereby locally tailoring the stiffness of the laminate. In addition, the laser-cuts created controlled hot-spots of strain concentrations where sub-critical damage developed in a pre-determined and stable way prior to penetration. This sub-critical damage allowed the laminate to dissipate energy yet preserving its load-bearing capability. Therefore, we infer that the enhancement in impact damage resistance of the engineered structure is the result of (i) the locally-tailored stiffness of the composite and (ii) the activation of extensive sub-critical damage.

The examination of the impacted samples in Figures 5.11 and 5.12 reveals a larger extent of whitening on the engineered sample — 40% increase on average with respect to the baseline. The larger amount of damage observable from the sample surface correlates well with the average increase of 42.7% in the energy dissipated at sub-critical levels prior to the peak load (Figure 5.10b).

5.5 Conclusions

We tailored laser-cut patterns to engineer the microstructure of a hybrid SRPP/CFPP cross-ply laminate to improve two aspects of its damage resistance: (i) sub-critical damage diffusion (i.e. energy dissipation capability) and (ii) impact damage resistance. With this purpose, we conducted DEN-T and penetration impact tests on a baseline (non-engineered) material and on specimens with engineered microstructures. The main results of this study are summarized below.

Hybridising SRPP with continuous carbon fibre layers resulted in an extremely tough material with laminate fracture toughness values obtained via EWF data reduction of about 213 kJ/m².

Introducing artificial discontinuities in SRPP/CFPP hybrid structures under DEN-T tests has led to:

- delocalising strain concentrations from the notch tip to the laser-cuts. This has promoted extensive sub-critical damage diffusion developing away from the notches resulting in a stable failure process;
- a significant reduction in the maximum load carried by the engineered microstructure with respect to the baseline before the onset of damage. However, the load bearing capability of the engineered microstructure after the onset of damage was higher than the one of the baseline microstructure;
- delaying the ultimate failure (occurring at the ligament plane) of the engineered microstructure. For a ligament length $l > 16$ mm, the increase in ultimate failure displacement was: (i) 190% larger than the baseline failure displacement calculated at the unstable crack-growth, occurring at the CFPP failure; and (ii) 50% larger than the baseline ultimate failure displacement calculated at SRPP failure; and
- increasing the energy dissipation capability of the material. The formation of wide-spread pull-outs of carbon fibre bundles dissipating energy away from the ligament plane has resulted in an increase of the ‘plastic’ work (slope of the specific work of fracture) of about 90% with respect to the baseline.

Introducing artificial discontinuities in SRPP/CFPP hybrid structures under penetration impact tests has led to:

- enhancing the impact damage resistance via a more ductile impact response. The use of tailored laser-cuts led to an increase of 42.5% in energy dissipation at sub-critical levels via formation of damage (PP debonding, fibrillation, plastic deformation and pull-out of carbon fibre bundles) and locally-tailored stiffness; and
- delaying the occurrence of critical failure (SRPP fibre failure) to larger indentation depths (increase of 26.6% in deflection at fibre failure).

Taking the DEN-T and penetration impact results together, we can further conclude that laser-cut patterns can be tailored point-by-point to meet different damage resistance requirements at different locations within the same structure. This has been achieved using the same technique to engineer the microstructure and the same material system. Therefore, the technique of introducing discontinuities via laser-cuts provides engineers with a significantly expanded design space and therefore holds the potential to achieve significant industrial impact.

Chapter 6

Conclusions

6.1 Introduction

The individual conclusions related to each individual work have been reported in the respective chapters. This chapter serves to bring together those conclusions, and also as a statement of originality of this PhD thesis, highlighting the main contributions to the development of novel microstructural solutions for high-performance Fibre Reinforced Plastics (FRPs) with enhanced damage resistance. Overall conclusions are reported, aiming at delivering the key message of the PhD thesis.

6.2 Novelty and impact

This PhD thesis contains the following original contributions:

- In [Chapter 2](#), we demonstrated the enhanced impact damage resistance of ultra-thin-ply Bouligand Carbon Fibre Reinforced Plastic (CFRP) microstructures with small pitch angles – 2.5° . This enhancement was achieved via a highly diffused through-the-thickness helicoidal distribution of sub-critical damage, thus, for the *first* time, successfully mimicking with CFRP the toughening mechanisms of naturally-occurring Bouligand structures.

- In [Chapter 3](#), we revealed the highly-dissipative sub-critical failure mechanisms activating during the propagation of twisting Bouligand matrix cracks, including crack twisting, crack branching, secondary cracks dispersion and stable ply-fragmentation. We revealed that, with thin-ply CFRP, Bouligand microstructures with decreasing pitch angles achieved a progressively more stable failure, characterised by a predominance of sub-critical damage over fibre failure and localised delaminations. We demonstrated that with a tailored Bouligand bio-inspired design it is possible to achieve the *largest* damage resistance improvement to quasi-static through-the-thickness loads *ever reported in the up-to-date literature* with thin-ply CFRP composites. We therefore revealed the full potential of Bouligand microstructures and used it to establish firmly that the inherent low performance to transverse loading of thin-ply CFRP (with standard lamination sequences) can be overcome, therefore greatly expanding the potential of thin-ply CFRP materials for structural applications.
- In [Chapter 4](#), we reported the design, prototyping, testing and analysis of *the first* high-performance Herringbone-Bouligand microstructure with CFRP. We showed that Herringbone-Bouligand microstructures can be tailored, point-by-point, in the structure leading to a high damage resistance solution capable of a largely reduced in-plane spreading of damage, with the latter mostly contained within the tailored Herringbone-Bouligand region. Therefore, this new CFRP microstructure constitutes an excellent tailorable damage-resistant solution with great potential for applications where resistance to through-the-thickness loads is paramount.
- In [Chapter 4](#), we presented the development of *the first* prototyping procedure for point-by-point tailored Herringbone-Bouligand microstructures based on the micro-moulding of uncured prepreg. We showed that this novel technique can potentially be used to widen the design space for point-by-point tailorable solutions in the composite industry.
- In [Chapter 5](#), we presented *the first* multi-tailored material design concept based on tailored patterns of staggered discontinuities inspired to the bone- and nacre-like microstructures. We showed that by tailoring the patterns of discontinuities, it is possible to improve different aspects of the damage resistance of a Self-Reinforced Polypropylene/Carbon Fibre Polypropylene (SRPP/CFPP) hybrid composite. We effectively engineered different microstructures at different locations within our specimens, but crucially always using the same material system and within a single coherent manufacturing

set-up. We showed that this versatile technique provides engineers with a significantly expanded design space and therefore holds the potential to achieve significant industrial impact.

6.3 Overall conclusions

In this PhD thesis, based on the works reported in [Chapter 2](#), [Chapter 3](#), [Chapter 4](#) and [Chapter 5](#) and the respective conclusions reported in §2.5, 3.5, 4.6, 5.5 and 6.2, we have demonstrated that the bio-inspired microstructural concepts of Bouligand, Herringbone-Bouligand and multi-tailored bone-inspired can be successfully exploited to diffuse sub-critical damage in synthetic composite materials. Specifically, compared against classically-designed composite structures:

- *Bouligand* microstructures demonstrated enhanced through-the-thickness sub-critical damage diffusion characterised by helicoidal delamination damage, twisting Bouligand matrix cracks, crack branching, stable ply fragmentation and reduced in-plane delamination areas;
- *Herringbone-Bouligand* microstructures were demonstrated to further enhance the damage diffusion capability of ‘classical’ Bouligand structures with the formation of disconnected delamination areas, highly reduced in-plane spreading of damage, delamination deflection and containment of damage within the Herringbone-Bouligand region; and
- *multi-tailored bone-inspired* microstructures demonstrated the ability to control failure via diffusing sub-critical damage along the designed patterns of staggered discontinuities, hence resulting in a stable highly-dissipative failure process.

The ability of these microstructural concepts to diffuse sub-critical damage, harnessing the failure process and preventing more critical failure mechanisms to occur — such as localised delaminations and fibre failure — resulted in a large enhancement in damage resistance, residual structural integrity and mechanical performances of composite structures commonly used in engineering.

We have showed that the developed bio-inspired concepts can be used to tailor point-by-point the properties of the structure, using manufacturing techniques suitable to be integrated

in an automated manufacturing process and hence suitable for the Composite Industry 4.0. Therefore these novel microstructural concepts and design strategies hold the potential for achieving large industrial impact.

In addition to the common ability of the Bouligand, Herringbone-Bouligand and multi-tailored bone-inspired microstructural concepts in diffusing damage at sub-critical levels, the distinctive traits and toughening mechanisms of the bespoke microstructures make them suitable to be tailored for different structural applications and loading conditions. Specifically, while the multi-tailored bone-inspired material concept demonstrated enhanced damage resistance via diffused in-plane spreading of damage, the Bouligand and Herringbone-Bouligand concepts showed enhanced damage resistance via through-the-thickness diffusion of sub-critical damage. Therefore, for a given application, the most suitable microstructural concept should be selected depending on whether in-plane or through-the-thickness spreading of sub-critical damage is desirable.

Additionally, for the multi-tailored bone-inspired design, we demonstrated that the enhanced energy dissipation capability is mainly governed by pull-outs. Therefore, this design is suitable for applications which undergo loading conditions that promote the formation of pull-outs, i.e. where tensile stresses are predominant (pure tensile loading or bending of thin structures). Similarly, for the Bouligand and Herringbone-Bouligand microstructures, we demonstrated their ability of diffusing sub-critical damage through-the-thickness of the laminates via the formation of (i) shear-driven failure mechanisms such as delaminations and shearing matrix cracks and (ii) bending-driven failure mechanisms such as Bouligand matrix cracks. Therefore, these microstructures are particularly suitable for applications where transverse shear stresses as well as bending stresses play a major role.

6.4 A philosophical note

In the first decade of the 21st century, when thin-ply composite technologies were firstly introduced in the market, Professor Stephen W. Tsai stated [85]:

“...thin plies have much to offer.

Laminates and structures can be made strong and damage tolerant...

The fear of premature failure by micro-cracking and delamination may be a thing of the past. Designers now have more options to select the best combinations of materials and processes.”

In this PhD thesis, we have demonstrated that the ability of the bio-inspired Bouligand and Herringbone-Bouligand microstructures to diffuse sub-critical damage in the form of highly dissipative matrix cracking paths and sub-critical delaminations has been greatly beneficial to the damage resistance and overall mechanical performances of thin-ply composites. Additionally, the exploitation of controlled sub-critical damage to enhance various damage resistance requirements of composite materials has been also successfully exploited to design multi-tailored bone-inspired microstructures with SRPP/CFPP hybrid composites. Therefore, taking together all the works reported in this thesis, we deliver the key message of this PhD thesis, by proposing a modified version (in *italic*) of Professor Tsai’s quote, which can be extended beyond the use of thin-ply composites and generalised to a wide variety of composite materials:

“...*composite materials* have much to offer.

Laminates and structures can be made strong, damage tolerant *and damage resistant*...

The fear of *critical and uncontrolled* premature failure by micro-cracking and delamination may be a thing of the past.

The exploitation of controlled sub-critical damage may be a thing of the future.

Designers now have more options to select the best combinations of materials, processes and *tailored microstructural solutions*.”

6.5 Recommendations for future work

The contributions of this work create pathways for further exploitation and investigation of the presented bio-inspired microstructural concepts. These are listed below.

- The ability of Bouligand-inspired FRPs to diffuse damage under through-the-thickness loads via the accumulation of matrix cracks and delaminations, preventing fibres from failing, can be used, in combinations with a thermoplastic matrix, to develop Bouligand Fibre Reinforced Thermoplastic (FRTP) composites with healing capabilities. This could potentially lead to high-performance multiple-impact resistant composite structures.

- In this thesis, we have explored Bouligand and Herringbone-Bouligand architectures using a single material system (Skyflex USN20A). However, we have developed analytical and numerical tools which can be used to explore Bouligand architectures depending on the material system. It would be interesting to explore Bouligand architectures with different material systems aiming at expanding further the use of Bouligand architectures for industrial applications.
- In nature, Bouligand and Herringbone-Bouligand microstructures are usually embedded in structures with single or double geometrical curvatures (such as shells or clubs). Therefore, while we have investigated these microstructures with flat samples, the investigation of the effect of the structure geometry on the performances of these microstructures might be relevant. Specifically, the novel prototyping technique developed in this work for the making of the first Herringbone-Bouligand CFRP microstructures could be used to study, for the first time with high-performance FRPs, telson-inspired [86] Bouligand architectures.
- The ability of Bouligand and Herringbone-Bouligand architectures to promote twisting matrix cracks, crack deflection mechanisms and discontinuous (non-linked) damage, could be exploited to enhance the damage resistance of FRPs under different loading conditions from the one explored in this work (through-the-thickness loads). Specifically, the in-plane properties of conventionally-designed FRPs, in which failure is usually characterised by a brittle and localised fracture, could benefit from such bio-inspired designs.
- The multi-tailored bone-inspired concept developed in this work was used to achieve a specific increase in energy dissipation capability and impact damage resistance. Based on this, a design framework could be developed which provides engineers with the possibility of choosing/designing a specific pattern of discontinuities, tailored to achieve a specific increment in damage resistance, taking into account other structural requirements/constraints such as strength and stiffness.
- In this thesis, the damage resistance capabilities of Bouligand, Herringbone-Bouligand and multi-tailored bone-inspired microstructures have been demonstrated only under quasi-static or low strain-rate conditions. However, these microstructures hold the potential to be used in applications subjected to high strain-rate loading conditions. Therefore, investigating such loading scenarios might be relevant to further explore the potential of these microstructures for industrial applications.

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