

Bristol Composites Institute (ACCIS)



Imperial College London

The Composites Centre

for research, modelling, testing and training in advanced composites

High Performance Ductile Composite Technologies (HiPerDuCT) EPSRC Programme Grant

Final Report [EP/I014322/1]

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HiPerDuCT Programme Grant Final Report [EP/I014322/1]

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This report summarises the research carried out under the EPSRC Programme Grant on High Performance Ductile Composites Technology between 2011 and 2018. A large collaboration between Bristol University and Imperial College investigated different ways of introducing ductility and pseudo-ductility into high performance composites. The programme initiated a new field of research and demonstrated that it is possible to create composites that fail more gradually.

1. Introduction

1.1 Background

Composites have become established as the materials of choice due to their high mechanical performance and continue to expand rapidly into new markets and applications. Conventional polymer matrix composites offer high strength and stiffness, low weight, and low susceptibility to fatigue and corrosion. Properties can be tailored to particular applications and additional functionality can be incorporated, e.g. for sensing, self-healing, morphing or energy storage. Composites are increasingly being used in aerospace and other applications, such as wind turbine blades, sporting goods and civil engineering. Despite this progress, a fundamental limitation of current composites is their inherent brittleness. Failure is usually sudden and catastrophic, with little warning or residual load carrying capacity. Structures that satisfy a visual inspection can fail suddenly at loads much lower than expected, so complex maintenance protocols are required, and significantly greater safety margins than for other materials.

1.2 Creativity in Composites Engineering

A call for programme grant proposals on 'Creativity in Composites Engineering' was issued by EPSRC in 2010. The aim of this call was to address longer-term challenges to help fully realise the potential of composite materials. Both Bristol University and Imperial College independently identified a key limitation of conventional composites as their inherent lack of ductility. A natural partnership emerged and it was agreed that solving this problem, whilst being a highly ambitious undertaking, would have the most significant impact on the usability of composites materials in a range of applications.

1.3 Aim

The aim of this programme was to realise a new generation of high performance composites that overcome the key limitation of conventional composites: their inherent lack of ductility. We sought to design, manufacture and evaluate a range of composite systems with a ductile or pseudo-ductile response, while maintaining strength and stiffness. The ability to yield and recover, be notch insensitive, exhibit high work of fracture, and fail in a benign manner, would offer a step change in damage tolerance, increasing the scope of applications and enabling new processing techniques.

2. Programme

2.1 Introduction

The programme on High Performance Ductile Composites Technology (HiPerDuCT) ran from July 2011 until June 2018. Prof. Michael Wisnom was the Principal Investigator and chair of the Management Team, which consisted of all the co-Investigators: Prof. Alexander Bismarck, Prof. Paul Robinson and Prof. Milo Shaffer from Imperial, and Prof. Kevin Potter, Prof. Ian Bond and Prof. Paul Weaver at Bristol. Sadly Dr Joachim Steinke from Imperial died in January 2013. Soraia Pimenta, a researcher on the programme obtained an academic staff position at Imperial in September 2013 and joined the management team. Prof. Ian Hamerton also joined following his appointment at Bristol in 2016.

2.2 Structure of Programme

Fig. 1 shows the overall structure of the programme, which was split into two domains: (1) *Architecture* and (2) *Constituents*. These were sub-divided into inter-related themes addressing three fundamental mechanisms to create ductility: by reorientation of fibres to take advantage of excess length (*Theme A*); by slip and fracture between aligned fibres (*Theme B*); and by means of ductile constituents (*Theme C*). This approach allowed the challenge to be tackled at length scales from molecular to structural.



Fig. 1. Structure of HiPerDuCT Programme

2.3 Management

The Management Team met monthly with the purpose of reviewing milestones and deliverables; planned publications; Intellectual Property; programme finances and allocation of funding. In addition, each work package was periodically reviewed to decide whether it should carry on or not.

An advisory board met annually to review the programme and provide feedback and comments on future direction. This was chaired by Prof. Dame Julia Higgins, FRS, FREng (Imperial College, and former Chair of EPSRC) and comprised: Prof. Anthony Bunsell (Ecole des Mines); Prof. Tsu-Wei Chou (University of Delaware); Prof. Ignace Verpoest (KU Leuven); Prof. Karl Schulte (TU Hamburg-Harburg);

Dr David Attwood, Brett Hemingway, Dr Amir Rezai (BAE Systems); Dr Rob Backhouse, Adam Bishop (Rolls-Royce); Prof. Paul Curtis (Dstl); Dr Alex Baidak, Dr David Tilbrook (Hexcel); and Dr Dan Kells, Dr Dan Thompson (NCC). Additional industry review meetings were held in between advisory board meetings.

2.4 Collaborators

The industrial partners in HiPerDuCT were: BAE Systems, Hexcel Composites, and Rolls-Royce. Collaborations were developed within the wider composites communities at both Bristol and Imperial, with a number of activities involving academics who were not investigators on the grant. Additional external partnerships were established, for example with Manchester University on textile composites, Exeter University on cellulose fibre precursors, Strathclyde University on fatigue sensors, Dundee University on laser modification of fibres, Cambridge University on carbon nanotube fibres, KU Leuven on hybrid composites, IMDEA in Madrid on high resolution micro strain measurements, BAM in Berlin on interfacial characterisation of modified fibres, and BME in Budapest on environmental characterisation of hybrids.

3. Highlights of the Programme

The programme investigated a number of different potential mechanisms for creating more gradual failure and successfully demonstrated pseudo-ductility in all of them. A key measure used to assess progress was pseudo-ductile strain, defined as the difference between the final failure strain, and the elastic strain at the same stress.



Fig. 2 Pseudo-ductile strain ε_d in glass/carbon thin ply hybrid

- Thin-ply carbon angle-ply composites have been shown to allow fibre rotation under load producing 1.2% pseudo-ductile strain without delaminating and a maximum stress of 950 MPa. Deformation was shown to be reversible, indicating that this is true ductility (WP A1).
- Wavy ply sandwich composites have been created with up to 9% strain at the structural level and high energy absorption (WP A7, patent submitted).
- Co-mingling of different fibre types has been achieved, giving a more gradual failure with 14% increase in maximum strain (WP B1).

- A new mechanism of ply fragmentation has been demonstrated in thin ply hybrids, producing a non-linear stress-strain response with a plateau and pseudo-ductile strains of up to 2.7%. Quasiisotropic laminates have been produced demonstrating notch insensitivity. Fatigue performance is good. Pseudo-ductility has also been demonstrated under compression, bending, and bearing loads (WP B3).
- Ply fragmentation has been combined with angle plies to create all-carbon laminates with a strength of 700 MPa and pseudo-ductile strain of 2.2%. A tubular tension member has successfully demonstrated pseudo-ductility whilst carrying 89kN.
- Glass/carbon hybrids produce a striped pattern that can be used as a simple static or fatigue overload sensor (WP B3).
- Hybrid specimens can avoid grip failures, enabling improved unidirectional tests and giving new understanding of the hybrid effect, specimen size effects, and failure criteria (WP B3).
- Model systems of discontinuous prepreg have shown the potential for additional strain via slip at the interfaces (WP B4).
- A novel manufacturing process has been developed allowing high volume fraction highly aligned discontinuous fibre composites to be produced (WP B5.1, patent submitted). These have shown record modulus and strength for single materials, approaching those of continuous fibre composites. Pseudo-ductile strain of 1.1% has been demonstrated, with the ability to achieve novel architectures with different fibre types, lengths and distributions. Multiaxial composites have been produced which can deform during manufacture to produce complex shapes. High performance composites have been created from recycled fibres.
- Good progress has been made towards creating ductile nanotube fibres that show high initial modulus and high strains to failure (WP C2.1). Tensile strength of 1.4 GPa has been achieved with a strain to failure of 12.7%, with much higher strains at reduced strength.
- A new class of nacre mimic has been prepared (WP C2.2). Coated fibres showed reduced local stress concentrations arising from fibre breaks and increased slippage.
- Comprehensive modelling of the deformation and damage mechanisms has been undertaken, giving a good understanding of the factors controlling performance, for example the effect of constituents, variability and defects on the behaviour of discontinuous hybrids (WP B8).
- Over 50 journal papers and more than 60 conference papers have been published.

4. Summary of Work Packages

Table 1 below summarises the work packages undertaken. In bold are the completed WPs resulting in publications for which reports with details are given in the appendix. The table also lists a number of initial feasibility studies, or projects which were not continued. Missing numbers are due to projects that were not in the end undertaken, or were reorganised into other WPs.

	Work Package (Lead)	Key Challenge		
	A1 Angle plies (Wisnom)	To create optimised angle ply laminates achieving 'ductility'		
		through fibre rotation and matched matrix response.		
	A5 Microbraided ropes	To investigate micro-braided hybrid fibre architectures and to		
	(lannucci, Robinson)	assess the potential for creating pseudo-ductile response of		
ntation Concepts		resulting composites.		
	A7 Wavy ply sandwich	To demonstrate large deformations in a wavy-ply sandwich with		
	(Robinson)	composite skins and crushable core.		
	A8 Wavy ply discontinuities	To evaluate the potential of in-plane wavy ply composites for a		
	(Robinson)	more ductile behaviour.		
	A9 Ductile micro-braided helical	To establish the feasibility of creating micro-braided structures		
orie	structures (Weaver)	displaying favourable load-extension characteristics.		
Rec	A10 Friction mechanisms	To use of friction mechanisms in order to achieve pseudo-ductile		
e	(Robinson)	behaviour while keeping high modulus and strength.		
Е.	A11 Triaxial braided composites	To explore the potential of triaxial braided composites for		
A	(Pinho)	pseudo-ductility.		
ne	A12 Auxetic Architectured UD	To investigate auxetic slitting patterns to increase strain to failure		
her	hybrid composites (Scarpa)	of UD hybrid composites.		
F	A13 Hierarchical perforations for	To use controlled defects (in-plane fibre waviness and resin rich		
	enhanced ductility (Allegri)	areas) to promote pseudo ductility.		
	A14 Multi-scale multi-fibre	To achieve pseudo-ductility via micro-wrapped/ spread tows in		
	hybridisation (Wisnom, Potluri -	braided and 3D woven hybrid architectures.		
	Manchester)			
	B1 Hybrids – intermingled fibre	To manufacture hybrid composites using a range of fibre types		
	types (Bismarck)	and optimise to maximise pseudo-ductile composite response.		
	B2 Hybrids – modulated fibre	To develop modified high strength, high stiffness fibres with		
	properties (Bond, Bismarck)	properties modulated along the length and investigate their		
S				
che	B3 Hybrids - Thin prepreg	To optimize and exploit the pseudo-ductility of thin-ply hybrid		
roa				
dd	B4 Discontinuous pre-preg	interleaves in unidirectional composites with pro-defined cuts		
ce /	PE 1 Discontinuous fibro	To develop povel manufacturing methods for aligned chart fibro		
rfa	manufacturing (Potter)	composites for high performance ductile response		
nte	PE 2 Liquid crystalling	To create a liquid crystalling suspension of short carbon fibres at		
П рс	manufacturing (Bismarck)	a relatively high concentration		
e al	B5 3 Thermonlastic extrusion	To produce aligned short fibre thermoplastic composites with		
ibr	manufacturing (Bismarck)	high fibre volume fraction and low void content via twin screw		
Р Ч		extrusion of UD composite tapes.		
gne	B5.5 Laser patterned	To create discontinuous prepreg with controlled patterns using a		
Ali	discontinuous fibres (Bismarck)	laser.		
E E	B5.8 Laser modified fibres	To exploit pre-weakened and modified fibre shapes to create		
me	(Bismarck)	pseudo-ductility.		
The	B6 Hierarchical bundle	To create small sub-bundle composites and validate the model		
	composites (Pimenta)	for hierarchical ductile composites.		
	B7 Interlaminar and ply	To exploit interlaminar and ply weakening to generate pseudo-		
	weakening (Robinson)	ductility.		
	B8 Discontinuous composites	To use modelling to optimise the pseudo-ductile response of		
	across the scales (Pimenta)	discontinuous composites.		

	C2.1 High performance ductile fibres (Shaffer)	To fabricate high-performance ductile fibres based on internal strain hardening between nanostructured elements.	
Theme C – Ductile Constituents	C2.2 Nacre-inspired interphase (Shaffer)	To coat fibres with nanoplatelet layers that can shield fibre breaks and lead to a strain hardening interface.	
	C3 High Performance Ductile Matrices (Shaffer)	To develop new high strength, high stiffness polymeric matrices with large failure strain and low creep.	
	C4 Carbon Nanotube Fibres and Sheets (Rahatekar)	To use high performance carbon nanotube fibres as potential ductile constituents for composite reinforcement and control of crack propagation	
	C5 Realising the Potential of Carbon Fibre Composites in Compression (Wisnom)	To suppress the shear instability or control it to achieve ductile compression behaviour of carbon composites.	
	C6 Optimised Matrices (Hamerton)	To tailor composites with matrices optimised to meet the requirements in other WPs.	
	C7 Ductile Superlattice Nanoparticle Matrices (Shaffer)	To create ductility via dislocation-based deformation in an ordered nanoparticle supercrystal matrix.	
	C8 Carbonised Cellulose Fibres (Rahatekar – Cranfield, Eichhorn- Exeter)	To create higher strain fibres by controlled carbonisation of regenerated cellulose and cellulose nanocomposite precursors.	

Table 1. Summary of Work Packages

5. Added Value and Impact

5.1 Added Value

A key success has been the creation of a tightly integrated research team working both across work packages and institutions to provide a coherent and well-coordinated programme, with monthly meetings of the whole team held alternately at Bristol and Imperial throughout the programme.

A number of research workshops and brainstorming sessions were held to explore new and creative approaches to tackling the challenges of creating ductile composites. These workshops had the added benefit of bringing in other academics with additional expertise to explore how their research ideas could contribute to the programme and seed corn funding was provided to initiate a number of new projects.

In addition to the research directly funded under HiPerDuCT, twelve PhD projects were aligned with the programme, listed in Table 2.

Jonathan Fuller	Pseudo-ductility of thin ply angle-ply laminates	Bristol CDT, 2015
Hele Diao	Carbon fibre reinforced polymer composites with	Imperial PhD, 2015
	enhanced ductility	
Henry Maples	Shape changing composites	Imperial PhD, 2016
Stephano del Rosso	Micro-braided composites	Imperial PhD, 2016
James Trevarthen	Towards CNT Fibre/Polymer Composites	Bristol CDT, 2016
Francois de Luca	Fibre-Reinforced Composites with Nacre-Inspired	Imperial PhD, 2018
	Interphase: A Route Towards High Performance	
	Toughened Hierarchical Composites	

Xun Wu	Behaviour of pseudo-ductile thin-ply angle-ply laminates under different loading conditions	Bristol CDT
Putu Suwarta Pseudo-ductility of Unidirectional Thin Ply Hybrid Composites		Bristol PhD
Tamas Rev	Exploiting thin-ply hybrids to establish failure criteria under multi-axial loading	Bristol CDT
Jakub Rycerz	Realising the potential of carbon fibre composites in compression	Bristol CDT
James Finley	Developing ductile composite materials across a range of strain-rates and scales	Imperial PhD
Jingjing Sun	Pseudo-ductile composites with weakened plies	Imperial PhD

Table 2. PhD Projects Aligned with HiPerDuCT

5.2 Contributions to and beyond the discipline

Many contributions have been made to important topics in composites research such as failure mechanisms, novel modelling approaches, hybrids, short-fibre composites, interfacial characteristics, composites with controllable stiffness, bi-continuous aerogel matrices and nanotube fibres. In addition the thrust of the programme to create ductile composites has stimulated interest in this new direction, which is quite distinct from other current research, opening up a completely new research area. We organised dedicated sessions on the theme of ductile and pseudo-ductile composites at the International and European Conferences on Composite Materials, the leading conferences in the field. Sessions at ICCM21 in Copenhagen in 2015, ECCM16 in Munich in 2016, ICCM22 in Xian in 2017 and ECCM17 in Athens in 2018 have all been well attended and created significant interest.

5.3 Dissemination and Impact

Dissemination of results was mainly through publication of journal papers and presentations at major international conferences such as ICCM and ECCM. A number of invited presentations were given, including a plenary by Prof. Michael Wisnom at the ICCM21 conference in Xian in August 2017. A website <u>www.hiperduct.ac.uk</u>, provides information on the programme, including publications.

Two industry and academic engagement meetings were held at the National Composites Centre in 2015 and 2017 to share programme outputs, attract new partners and to get feedback on industry requirements.

A new £1.3M EPSRC project on "High Performance Discontinuous Fibre Composites: A Sustainable Route to the Next Generation of Composites" is taking forward this element of the programme, scaling up the HiPerDiF process and applying it to a range of applications, led by Prof. Ian Hamerton.

An impact acceleration project was also funded on "Application of the HiPerDiF (High Performance Discontinuous Fibres) manufacturing method for Quality Control of reclaimed carbon fibres in recycled composite materials" in collaboration with ELG Carbon Fibre Ltd, which demonstrated that the method could be successfully applied to the property measurement and quality control of reclaimed carbon fibres and recycled composites.

A second impact acceleration project was also funded on "Hybrid composite strain overload sensor concept in real-life applications – Market search and industrial partnership for exploitation of the invention". This identified a number of potential applications for the technology, and companies which could potentially benefit. Further activity is being pursued directly with some of these companies, and

a Technology Pull-Through project has been funded by the National Composites Centre to investigate the repeatability and environmental stability of the sensors, and look at scale-up and applications.

Further work is also being pursued on other potential applications, for example the use of hybrid composites for resisting impact in aero-engine components in collaboration with Rolls-Royce.

Another key impact is that six researchers including all the initial team members moved on to academic appointments and remained involved in HiPerDuCT in their new positions: Dr. Jonny Blaker, University of Manchester; Dr. Soraia Pimenta, Imperial College; Dr. Gergely Czel, Budapest University of Technology and Economics; Dr. HaNa Yu, Bath University; Dr. Meisam Jalalvand, University of Strathclyde; Dr. Mohammad Fotouhi, University of the West of England.

5.4 Patents

Six patent applications were filed during the course of the programme, listed in Table 2.

Reference	Title
GB1306762.4	Method and apparatus for aligning discontinuous fibres
GB1405824.2	Wavy Sandwich Structural composite material
PCT/GB2013/050826	Composite Material Suitable for a Morphing Skin
P120511GB	Visual Strain Sensor
1700641.8	Fatigue sensor
1621494.2	Nacre-like decorated fibre for hierarchical ductile composite
	Table 3. HiPerDuCT Related Patent Applications

6. Conclusion

This programme has shown that it is indeed possible to create high performance composites that fail gradually with ductile or pseudo-ductile response and notch insensitivity. A strong collaboration has been established between Bristol and Imperial, and a wide range of novel approaches investigated. A number of new ways of creating additional strain have been successfully demonstrated via fibre rotation, in-situ fragmentation, fibre ductility and slip in continuous and discontinuous composites. A whole new field of research has been initiated, with a large body of work published in top journals and conferences. This work is being continued through new grants, in conjunction with industry and the NCC, and through the research of the team of outstanding researchers who now have their own academic positions.









The Composites Centre for research, modelling, testing and training in advanced composites

HiPerDuCT Programme Grant

A1 Final report: Angle Plies

Ductility via fibre re-orientation

Using carbon/epoxy plies of only 0.03 mm thickness, matrix cracking and delamination can be completely suppressed in angle-ply laminates, allowing the fibres to rotate under tensile loading, creating additional strain and pseudo-ductility [1]. Angle plies of (±45) layup can produce strains of over 20% and necking behaviour despite the brittle nature of the matrix (see figure 1). There is a trade-off between the stresses and strains that can be achieved depending on the angle, and this has been investigated in modelling studies [2]. A good balance of properties has been achieved for example with thin ply (±25) carbon/epoxy laminates that gave a pseudo-ductile strain of 1.23% and a maximum stress of 927 MPa, figure 2 [1].



Figure 1. Necking of (±45) thin ply carbon/epoxy angle ply specimen



Figure 2. Pseudo-ductile response of thin ply (±25) carbon/epoxy laminate

Tests involving loading, unloading and then reloading have shown that the initial modulus is fully recovered, and so these laminates may be considered as ductile rather than pseudo-ductile, figure 3 [3].



Fragmentation in thin-ply angle-plies with 0° plies

Introducing thin 0° plies allows fragmentation to occur in these plies which together with the fibre rotation of the angle plies gives a highly non-linear response, as shown in figure 4, with a pseudo-ductile strain of 2.2% in this case [4]. On reloading, these laminates do show some loss of initial modulus due to the fragmentation of the 0° plies, and so are pseudo-ductile rather than fully ductile [3]. Gradual failure is maintained when loaded at small angles to the 0° plies, although with reduced pseudo-ductile strain. High strain rate testing has demonstrated that pseudo-ductile behaviour can be retained, with modified response [5]. The effects of different temperatures on response have also been assessed [6]

Different materials can be used for the 0° and angle plies. For example a (25/-25/0/-25/25) layup using thin ultra-high modulus carbon for the 0° ply but standard modulus thin carbon for the angle plies allows a pseudo-ductile sub-laminate to be produced which is only 0.15 mm thick and has a modulus of 135 GPa [5]. Multi-directional laminates of these pseudo-ductile plies can then be created. Modelling has shown that they give a pseudo-ductile response [7] and this has also been demonstrated experimentally.

Fragmentation of the 0° plies can also occur under compression, giving an analogous pseudo-ductile response to that in tension, although with lower strength [8].



Figure 4. Pseudo-ductile response of [±265/0]s laminate

Notch Insensitivity

The pseudo-ductility produced due to fragmentation and the associated non-linear response allows load redistribution to occur at stress concentrations, in a similar way to stress redistribution due to plasticity in ductile metals. Modelling has shown that a notch-insensitive response should be obtained provided the ratio of pseudo-ductile strain to pseudo-yield strain is sufficiently high [9].

Figure 5 shows the response of a $[\pm 25_2/0]_{s4}$ laminate with Intermediate modulus angle plies and high modulus 0° plies. The unnotched behaviour is compared with that of a specimen with a 16 mm wide specimen with a 3.2mm hole, showing that the open hole strength is similar to the net-section pseudo-yield stress [10].

Gradual failure with load redistribution has also been demonstrated in bolt bearing tests [11].



Figure 5. Open-hole tension response of [±25₂/0]_{s4} IM-HM carbon/epoxy laminates

Fatigue Behaviour

Cyclic loading of $[\pm 25_2/\underline{0}]_s$ carbon/epoxy laminates with intermediate modulus angle plies and ultra high modulus 0° plies has shown no damage up until 100,000 cycles at 80% of the pseudo-yield stress and up to 1000 cycles at 95%, Figure 6 [12].



Figure 6. Fatigue response of IM-HM [±25₂/0]_s laminates

Demonstrators

A number of demonstrators have been manufactured and tested showing that the concept can be applied to real components [13] including a skateboard [14] and a tubular tension member that successfully carried nearly 90kN with pseudo-ductile failure, Figure 7.



Figure 7. Pseudo-ductile response of tubular tension member

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HiPerDuCT Programme Grant

A5 Final report: Microbraided ropes

This study presents the manufacture and mechanical characterisation of hybrid microbraids made by braiding UHMwPE fibres over a unidirectional core of carbon fibres, and their direct use as the reinforcing phase within polymer composites. The main aim of this work is to investigate composite materials able to undergo large deformations, nevertheless having stiffness and energy absorption capabilities at least similar or better with respect "conventional" fibre reinforced polymer composites made of the same materials [1,2].

The manufacture of 2D hybrid microbraid was performed using a Herzog RU2/16-80 vertical braiding machine as shown in Figure 1. The braider was set to interlace eight DyneemaRSK75 yarns in a diamond fashion over a unidirectional core of carbon fibres (ToraycaR T300). Microbraids having different braid angle α were manufactured by changing the cogwheel ratio on the braiding machine.



Figure 1: Hybrid microbraids: (a) HMB1 α =14° (b) HMB2 α =33°; (c) HMB3 α =39°.

Quasi-static tensile tests on yarns and dry microbraids were performed at room temperature using an Instron 5969 universal testing machine equipped with a 50 kN load cell. In order to prevent fibre damage and a possible premature failure of the carbon filaments, rubber tabs were glued on the specimen ends using Araldite 2011.



Figure 2: Tensile stress vs. tensile strain of the investigated yarns and microbraids.

The mechanical behaviour of core-filled microbraid has three distinctive regions:

In Region 1, the tensile behaviour of the hybrid microbraids was mainly governed by the unidirectional core fibres and, to a lesser extent, by the bias fibres. The UD core eliminated the initial long plateau commonly seen when tensile testing coreless braids. The tensile stress increased in a linear fashion with increasing the load. The Young's modulus E_i and the strength at the first failure of the carbon fibres σ_{ic} decreased with increasing braid angle. When the strain reached the strain to failure of the carbon fibres, failure of the inner core occurred. The bias fibres not only contained the failed carbon fibres in the core of the braid, but also carried the load after failure of the core.

In Region 2, a series of peaks in the tensile stress was observed. As the bias fibres tended to scissor to the loading direction, the diameter of the microbraid reduced, squeezing and containing the failed core. The load was taken by the bias fibres and transferred to the inner fibres via friction. Although fractured, the carbon core was able to absorb the load until a subsequent failure occurred.

In Region 3, the bias fibres reached the jamming point and the tensile stress increased until a final catastrophic failure occurred. Even in this region it is possible to identify further ruptures of the carbon fibres in the core.

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HiPerDuCT Programme Grant

A7 Final report: Wavy-ply sandwich with crushable core

This workpackage focused on the design, simulation and testing of a sandwich structure (Figure 1) which exhibits large deformations (through unfolding of the wavy composite skins) and high energy-absorption (through crushing of the foam core cells) under tensile loading [1,2].

The wave geometry, foam material and shape of epoxy fillet were optimised through a combination of analytical modelling and Finite Element (FE) simulations. Abaqus' plasticity model for crushable foam with volumetric hardening was used to simulate the response of the core material; the initial yield surface of the foam was calibrated using the values of strength under uniaxial tension, uniaxial compression and shear loading provided by the manufacturer, and the assumed strain hardening behaviour was found to influence significantly the response and failure mode of the wavy-ply sandwich structure. Cohesive elements were used to account for the possibility of debonding near the bridging region, between the skin and the foam core / epoxy fillet.

Wavy-ply composite specimens were manufactured using machined foam cores and aluminium moulds. Experimental testing of the wavy-ply specimens demonstrated high strength, large deformations and high energy absorption (Table 1), as predicted by the FE simulations (Figure 2). Envisaged applications of the *wavy-ply sandwich with crushable core* concept include blast protection structures and casings.



Figure 1. Wavy-ply sandwich concept.

	Initial stiffness,	Ultimate strength,	Failure strain,	Energy dissipated,
Average		1570 MPa	<u> </u>	9.35 kJ/kg
Coefficient of Variation	2.9%	5.0%	2.1%	9.6%

Table 1: Mechanical properties measured for wavy-ply composites (from six successful tests).



Figure 2: Comparison between FE (left) and experimental (right) results.

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A8 Final report: Shifted in-plane waviness in UD laminates

'Brick and mortar' composite architectures (see Figure 1a) have been investigated to produce a ductile failure process [1, 2]. The pseudo-ductility of the architectures results from either complete yielding of the matrix in the overlap region, or from cracks initiated at the ends and growing inwards towards the centre of the overlap region. However, in a composite containing many overlaps, the failure tends to localise at one set of overlaps running across the thickness of the specimen.

A modified continuous unidirectional 'brick and mortar' composite architecture (Figure 1b) with inplane wavy connecting segments was investigated. The in-plane wavy segments (in the low initial stiffness state) extend and stiffen after failure of one overlap, so that the applied load is transmitted to the adjacent overlaps. In this way the localisation problem can be avoided and a pseudo-ductile stress-strain relationship permitted.

It is important to ensure that the in-plane wavy segments extend and carry the load after failure of the adjacent overlaps. To achieve this, the composite must fail by shear cracking between the fibres. This allows the composite to extend and stiffen as the fibres become straighter. An interleaved wavy unidirectional carbon fibre/epoxy composite has been manufactured containing polystyrene (PS) interleaves.

Preliminary investigation was conducted on flat unidirectional carbon fibre/epoxy composites of HexPly T300/914 with polystyrene interleaves ([0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°/PS/0°], nominal thickness = 1.7 mm). The PS interleaf can lose its stiffness when heated above the glass transition temperature and regain the stiffness when cooled down [3]. To take advantage of this, specimens were heated to 120°C and re-shaped by axially compressing to form the required wavy profile. They were cooled down to room temperature and tested under tension. The stress-strain relationship from a tensile test (see Figure 2) shows that delamination of the polystyrene interleaves occurred at around 0.8% strain and the composite continued to straighten and stiffen, and finally failed at around 2.7% strain. As expected, this significantly surpasses the failure strain (~1.3%) of the pristine unidirectional carbon fibre/epoxy.



Figure 1. Sketches of (a) 'bricks and mortar' composite architecture [1-2] and (b) modified 'brick and mortar' composite with in-plane wavy segments.



Figure 2. Resulting stress-strain relationship of an interleaved wavy specimen under tension at room temperature.

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A9 Final report: Pseudo-Ductility in Helical Lattice Structures

An analytical investigation of the non-linear elastic response of helical lattice structures demonstrated that pseudo-ductile responses can be obtained [1]. The feasible region of pseudo-ductility was determined based on the tunable parameters that govern the system response, including the form factor, geometric and stiffness ratios.



Figure 1. Helical lattice structure

Reference

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A10 Final report: Friction Mechanisms

The proposed mechanism relies on the geometry shown in Figure 1. The structure is composed of jigsaw like pieces, each of which is made of bow-tie shaped elements which are connected by infill regions. Each coloured jigsaw block is a separate block that is only connected to other blocks by the interlocking jigsaw shape. When this configuration is subjected to tension in the longitudinal direction, the blocks start to slide against each other. This sliding causes friction forces in the interlocking structure. The role of the infill region is to introduce the transverse forces thereby increasing the frictional force in the wedges and so leading to higher overall longitudinal stresses in the structure.



Figure 3: Geometry, a) unloaded specimen, b) loaded specimen

Ceramics

An investigation was performed on a model system made of MACOR, a brittle machinable glass ceramic [1]. The initial modulus, strength and failure strain were, 67 GPa, 34.5 MPa and 5.15e-4, respectively. Finite element models indicate that pseudo-ductility with hardening behaviours can be obtained in ceramics by optimizing the interlocking structure of the jigsaw blocks.

Figure 2 shows that pseudo ductile behaviour with long plateaus can be obtained by changing the geometry. The failure strain of brittle ceramics can be increased significantly.



Figure 4: Pseudo-ductile behaviour achieved in interlocking ceramic structure

3D Printed materials

3D printed internally and externally constrained interlocking specimens were investigated [2] to test different configurations leading to pseudo-ductile behaviour.

Stress-strain plots for the configurations made of 3D printed materials are shown in Figure 5. It can be observed the specimens show an initial response which is approximately linear. The stiffness of the externally constrained specimen is higher because, for a given strain, the extension of the central part of the dog bone shape is small compared to the extension due to sliding of the inclined surfaces. The tangent stiffness for both curves starts to reduce (at a stress between 0.75 -0.9 MPa) and finally levels out at a maximum stress of around 1MPa.

At this point the sliding has started to localize at one position and beyond this, the stress drops gradually until complete separation of the interlocking shapes occurs at some position.



Figure 5: Mechanical response of 3D printed bow-tie structures

Composites

Composite specimens (2) show a non-linear behaviour. It can be seen that the specimen with no preload reaches a strength of 24 MPa at a strain of 18 %. However, the geometrically identical but preloaded specimen achieves 27 MPa at 15 % strain. Between approximately 3% strain and the curve peak, the curve of the preloaded specimen is shifted to higher stresses than the curve of the specimen without preload by a relatively constant value of about 5 MPa. The composite assembly fails prematurely due to localization of the deformations, but prior to failure the behaviour shows good agreement with the prediction.



Figure 6: Mechanical response of composite bow-tie structure

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A11 Final report: Triaxial Braided Composites

Loading in transverse direction

Triaxial braided composites can show a highly non-linear stress-strain response. For example, the $[0/\pm30]$ carbon/epoxy braid shown in Figure 7(a) loaded in the transverse direction features a pseudoductile plateau due to the complex damage behaviour. Severe non-linearities are present in the stress-strain response for loading in the transverse direction. The $[0/\pm30]$ braid features a complex damage behaviour, which is shown in Figure 7(a). Here, the material response can be separated into three distinct domains up to final failure: an elastic domain, a damage progression domain and a saturation domain. When a critical load level is reached in the linear domain, matrix cracks form in the axial yarns and initiate further cracking inside the braid yarns. As the strain further increases, the load level exhibits a stable plateau. In the experiments, this behaviour is associated with the continuing development of inter-yarn and also intra-yarn cracks across the entire specimen length, originating from the initiation location. Final failure of the specimen occurs where multiple cracks coalesce across the specimen width.



Figure 7: Stress-strain curves for loading in transverse direction (a) [0/±30] (b) [0/±45]

Loading in braid fibre direction

The triaxial braided composite was also loaded in the braid fibre direction, experimental results are shown in Figure 8.



Figure 8: Stress-strain curves for loading in braid fibre direction (a) [0/±30] (b) [0/±45]

Up to a homogenized strain level of 0.6 %, all braid architectures exhibit approximately linear stressstrain behaviour. Distinct load drops after this can be attributed to a progressive mixed mode failure mechanism intrinsic to the textile architecture: as the external load is coincident with the braid fibre direction, the aligned yarns straighten along their longitudinal direction. Hence, intersecting axial yarns are subjected to an out-of-plane movement, which is at first inhibited by the overlying non-aligned braid yarns. As the load is increased, transverse cracks and delaminations develop in these areas. This process continues, until multiple inter and intra-yarn cracks appear in the braid yarns along the path of a single underlying axial yarn. Finally, the braid yarns lose their capability to resist the out-ofplane movement, resulting in a sudden delamination of individual axial yarns across the entire specimen width. This mechanism is repeated, until all axial yarns debond over the specimen length, causing global delamination of the coupon.

Meso-scale simulation framework

A meso-scale framework for predicting the non-linear mechanical response of triaxial braided composites was developed, which showed good correlation with the measured stress-strain curves and damage mechanisms [1, 2]. Based on a reduced unit cell concept which exploits symmetries to minimise computational expense, a compacted and interpenetration-free yarn geometry is created within a three-stage simulation process. In the first step, a nominal geometry is constructed from user-defined input parameters. Local volumetric interpenetrations present in the model are resolved in a subsequent fictitious thermal step. The unit cell is further compacted to the desired fibre volume fraction using flexible membranes Out-of-plane periodic boundary conditions allow an implicit consideration of the compaction of multiple braid plies in different nesting configurations, which further enables us to render high global fibre volume fractions (55-60%) using experimentally determined intra-yarn fibre volume fractions.



Figure 9: Roadmap and data flow for generating a realistic unit cell model

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A14 Final report: Micro-wrapped Hybrid Tows

Dry fibre tows consisting of high modulus fibres micro-wrapped with high strain-to-failure fibres have been developed as potential candidates for improving ductility in high performance composites (figure 1). These dry textile architectures have the potential to offer significantly lower-cost ductility solution in comparison to thin-ply prepreg-based laminates.



Figure1: a) micro-wrapping T700 carbon fibres with S glass fibres b) M55J fibres with T700 carbon fibres

Uni-directional (UD) woven fabric (figure 2a) laminates consisting of micro-wrapped tows have been compared with conventional hybrid laminates (with side-by-side configuration). While conventional hybrids exhibited a significant load-drop after the first failure, micro-wrapped laminates exhibited pseudo-ductility through stable fibre fragmentation/ pull-out (figure 2b).



Figure 2: a) Uni-directional weave micro-wrapped fabric b) micro-wrapped versus conventional side-by-side hybrid

Reference

 Islam MH, Koncherry V, Wisnom MR, Potluri P, Pseudo-ductile Composites with Microwrapped Hybrid Tow, ASC 33rd Annual Technical Conference, September 24-26, 2018, Seattle, USA.









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B1 Final report: Intermingled Fibre Composites

A gas-flow-assisted process was developed to spread and co-mingle fibres that was able to produce hybridisation of glass and carbon composites [1]. A method of quantifying the degree of hybridisation was developed and applied to characterising the composites, Fig. 1 [2]. The manufacturing process resulted in slight degradation of the fibres and a broadening of the fibre alignment distribution [3]. Non-constrained annealing of carbon fibre/PA-12 also broadened the fibre distribution, producing wavy fibre composites. Both methods gave a stepwise and more gradual tensile failure mode with the wavy composites having an ultimate failure strain of 2%, significantly higher than 1.6% of the control composite, Fig. 2 [4].



Figure 1: Analysing the degree of hybridisation of glass/carbon composites



Figure 2: Tensile stress–strain curves of (a) control, (b) gas-textured and (c) non-constrained annealed carbon fibre/PA-12 tapes

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B3 Final report: Thin-ply hybrids

Pseudo-ductility in Unidirectional Tension

A new failure mechanism of fragmentation in hybrid thin-ply laminates has been demonstrated, with multiple failures of the stiffer, lower strain to failure plies and stable pull-out of the fragments, producing a pseudo-ductile stress-strain response [1]. The use of thin plies suppresses overall delamination. Figure 1 shows a typical response for two 0.029 mm plies of high strength TR30 carbon sandwiched between single 0.155 mm S-glass epoxy plies on either side [2]. The striped pattern associated with the fragmentation is also shown.



Figure 1. Pseudo-ductile response of S-glass/high strength carbon hybrid [2]

Modelling has shown that both the relative thickness (i.e. proportion of carbon) and absolute thickness of the carbon plies are important in controlling the hybrid response [3-6]. With the appropriate thicknesses, premature brittle failure of the whole hybrid specimen and catastrophic delamination can be avoided.

Damage mode maps can be produced such as Figure 2, with the different failure mode regions in this case indicated approximately based on FE analysis [3]. However analytical models allow the boundaries to be calculated explicitly [4].

There is a trade-off between pseudo-ductility and yield stress [2,5]. A range of different glass-carbon hybrid configurations has been evaluated, and pseudo-ductile strains of up to 2.66% have been obtained with a plateau stress of 520 MPa, or 0.86% pseudo-ductile strain with a plateau stress of over 1300 MPa [2].



Figure 2. Damage mode map for E-glass/thin carbon hybrid composite [3]

Similarly pseudo-ductile response has been demonstrated with hybrids with different grades of carbon fibres. Figure 3 shows the response of hybrid specimens made from thin plies of Granoc XN80 ultrahigh modulus carbon between layers of T1000 intermediate modulus carbon fibres [7].



Figure 3. Stress-strain response for [T1000₂/XN80₂/T1000₂] carbon hybrid [7]

Loading-unloading-reloading tests show a reduction in initial modulus due to the damage, and so these laminates are pseudo-ductile rather than truly ductile [8,9].

It has been shown that acoustic emission can be used to detect fragmentation, with a direct correlation between acoustic and fragmentation events, allowing the technique to be used to detect fragmentation in opaque carbon/epoxy laminates [9,10].

Introducing cuts into the low strain plies is an alternative means of producing pseudo-ductility via controlled local delamination from the discontinuous plies before they fragment [11].

Pseudo-ductility in Multi-directional Laminates

Modelling showed how pseudo-ductile behavior could be achieved in multi-directional layups [12] and this has been demonstrated experimentally with quasi-isotropic laminates made from unidirectional carbon fibre hybrid sub-laminates [13]. An alternative concept with sublaminates formed by blocks of the same material with different ply orientations rather than blocks of different materials with the same orientation has also been shown to produce pseudo-ductile quasi-isotropic laminates whilst reducing the risk of free edge delamination [14], and giving similar response in all the fibre directions, Figure 4 [15]. Loading at small off-axis angles also produces pseudo-ductile response [16]



Figure 4. Pseudo-ductile response of glass/carbon hybrid loaded in four fibre directions

Notched Response of Pseudo-ductile Hybrids

The pseudo-ductility produced due to fragmentation and the associated non-linearity allow load redistribution to occur at stress concentrations, resulting in notch-insensitive response in a similar way to stress redistribution due to plasticity in ductile metals. This was investigated by modelling, which showed that the ratio of pseudo-ductile strain to failure initiation strain is a key parameter [17]. Notched quasi-isotropic pseudo-ductile laminates made from hybrid carbon sub-laminates similar to those shown in Figure 3 were tested with 3.2 mm holes [13]. Figure 5 shows the response, indicating that the failure stress of the specimens at the ligaments (net section stress) has actually exceeded the un-notched strength of the laminate. In addition, after the net section strength had been reached, they did not immediately fail catastrophically, but still showed some residual load carrying capacity, gradually reducing with further increasing strain. A similar notch-insensitive response was obtained with sharp notches [13]. Material blocked multi-directional glass/carbon hybrids were also shown not to be sensitive to sharp notches or open holes [18]. Gradual failure has also been found in bearing and bearing-bypass tests [19] and in compact tension tests as well [20].



Figure 5. Open-hole tension of QI hybrid carbon specimens

Pseudo-ductility in Other Loading Modes

Fragmentation can also occur in compression, for example in thin ply M55 carbon/ S glass laminates on the surface of specimens loaded in bending [21]. The carbon fibres break, and then some relative movement between the fracture surfaces allows further loading and failure elsewhere, leading to a similar fragmentation behaviour and associated change in stiffness to that observed in tension. The visually observable fragmentation pattern and a schematic of the failure mechanism is shown in Figure 6 [22]. Similar behaviour has also been obtained in direct compression tests, giving a significantly non-linear response [23].

In hybrids with higher strain carbon fibres near the surface loaded in bending, fragmentation occurred in tension before sudden failure in compression, but compressive strains as high as 2.5% were obtained in the carbon plies [21]. Specimens of this type which were fragmented first in tension and then loaded in compression, failed earlier, but still exhibited compressive strains in excess of 1%. Specimens hybridised all through the thickness similarly fragmented in tension before failing in compression [24].



a) Top view of fragmentation pattern



Figure 6. Fragmentation of S-glass/ M55 carbon hybrid in compression [22]

Tension fatigue testing on glass/carbon hybrids at 90% of the knee point strain which showed no fibre failure on first loading sustained no damage after 74000 cycles [25].

Specimens that were loaded statically until initiation of fragmentation and then fatigued at 80% of the knee point stress showed gradual growth of damage until they were fully delaminated after around 6000 cycles.

Tensile tests at high loading rates have shown that fragmentation and pseudo-ductility still occur [26]. Pseudo-ductility is also maintained at -50°C and +80°C [27]. Initial results under static indentation [28] and impact [29] show promising behaviour, with the ability to tailor the response and modify the failure mechanisms.

Overload Sensing

When fragmentation occurs in glass/carbon hybrids it causes a visible change in appearance on the surface with a striped pattern emerging as the strain increases, Figure 7. This can be calibrated and used as a simple overload detector, either forming the surface load bearing layer of the structure, or as a separate bonded-on sensor for composite or metallic structures [30-32]. It can also be incorporated into a smart self-warning repair patch [33].



Figure 7. Visualisation of overstrain in glass-carbon hybrid

By incorporating a pre-cut, the clearly visible extent of delamination can be used to estimate the number of fatigue cycles the structure has undergone, as shown in Figure 8 [34].



Figure 8. Fatigue sensor

Improved Testing and Understanding of Failure

Hybrid specimens allow improved tensile testing. The glass plies on the surface of glass/carbon hybrids can completely eliminate the stress concentration where the specimen is gripped, producing consistent gauge length failures [21]. This gives higher results than other test methods, and allows some fundamental effects to be explored that could otherwise be obscured by variability and premature failure.

When the low strain plies in the hybrid laminates are very thin, it has been shown that there is an enhancement in the strain to failure referred to as a hybrid effect [35]. This only occurs for plies less than 0.1 mm thick, and can be as high as 20% for a single 0.03 mm ply. The effect can be modelled, and has been shown to be due to the constraint on forming critical clusters of fibre breaks [35]. This means that as well as producing pseudo-ductile response, these hybrid laminates are able to take greater advantage of the intrinsic properties of the carbon fibres. Similar enhancements in the failure strain have also been measured with thin high modulus carbon sandwiched between intermediate modulus carbon plies [36].

Scaled hybrid specimens can be used to determine the size effect, whereby the tensile strain at failure decreases with increasing specimen volume [37]. Study of progressive ply fragmentation tests can be used to estimate the intrinsic variability of the material and deduce the Weibull modulus [37]. Hybrid specimens can also be used to study the delamination behaviour at the interface, and to deduce the mode II traction-displacement relation for use in cohesive finite element analysis [38,39].

Since thin plies suppress delamination, and tab failures can be avoided, hybrid specimens allow some innovative methods of investigating the interaction of different stress components on failure. For example, thin carbon-fibre angle plies sandwiched between glass have shown very limited effect of high in-plane shear stresses on fibre direction tensile failure [40]. Angle plies have also been used to investigate the interaction of large transverse compressive stresses on tensile failure of unidirectional plies [41].

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B4 Final report: Discontinuous Prepreg

Discontinuous Prepregs with Interleaves

The failure modes of fibre reinforced composites can be altered though the introduction of discontinuities in the fibres or through the inclusion of interleaved materials between the laminae. When prepreg plies are cut to produce a discontinuous architecture, the tensile properties of the resulting composites are strongly dependent on the overlap [1,2, 3] and the materials contained within the interply region. The insertion of tough thermoplastic interleaves in a unidirectional carbon fibre reinforced composite is expected to improve shear properties in tension in matrix dominated failures/configurations [4, 5]. The combination of a discontinuous composite architecture with an interleave region was explored.

A pre-cut unidirectional carbon fibre prepreg composite, with an overlapped finger-joint architecture, was produced with polyethersulfone (PES) interleaves, Figure 1 [6, 7]. When the tough thermoplastic interleaves spanned only the central portion of the overlap, a crack arresting failure mechanism was observed in tension. A pronounced plateau region or pseudo-ductile response was shown in conjunction with a strain hardening response after crack arrest (Figure 2). The local strain-to-failure of PES interleaved samples was increase by 85% compared to the pre-cut baseline. The interleaving process is simple to implement and can be used without changing the prepreg curing cycle. Further developments, for example, multiple overlaps for an increased strain-to-failure or the use of alternative interleave materials, could be exploited to yield a high performance yet (pseudo-)ductile composite response under tension.



Figure 1. Discontinuous composite architecture with a thermoplastic toughened interleave region with epoxy fillets.



Figure 2. Tensile tests of discontinuous unidirectional carbon fibre PES interleaved composites with a comparison baseline of discontinuous unidirectional carbon fibre composites with and without epoxy fillets.

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HiPerDuCT Programme Grant **B5.1 Final report: Aligned Discontinuous Fibres**

The High Performance Discontinuous Fibre (HiPerDiF) technology allows to produce Aligned Discontinuous Fibre Reinforced Composites (ADFRC) with mechanical properties comparable to those of continuous fibre composites manufactured with the same constituents [1,2,3]. The HiPerDiF discontinuous fibre alignment method exploits the sudden momentum change of a jet of fibres suspended in water directed in a narrow gap between parallel plates.



Figure 1. HiPerDif machine schematic and working method [4]

If the fibres are longer than the critical length, pseudo-ductility can be achieved in ADFRC through three different methods of hybridization: intermingled hybrids [5], intraply hybrids [6], and interlaminated hybrids [7]. In all cases, the ductile response is achieved through fragmentation and diffuse debonding or delamination of the low elongation reinforcement. Different types of hybrid can be organised in a hierarchical structure to further enhance the pseudo-ductile behaviour [8]. If the fibres are shorter than the critical length the pseudo-ductile behaviour is achieved through a fibre pullout mechanism [9].

Intermingled Hybrids

Intermingled ADFRC hybrids containing High Modulus low strain Carbon and Glass or High Strength Carbon fibres with high strain allow pseudo-ductile behaviour to be achieved through fragmentation of the low strain fibres [5,8]. This has been demonstrated also for guasi-isotropic laminates [10].



Figure 2. Pseudo-ductile intermingled ADFRC hybrids behaviour [8]

Interlaminated Hybrids

Interlaminated hybrids are made of a central thin layer of ADFRC sandwiched between continuous high elongation fibres, e.g. glass fibres. Through the use of Damage Mode Maps is possible to tailor the cross-section to achieve a pseudo-ductile behaviour [6,8,11,12].



Figure 3. Pseudo-ductile interlaminated hybrid behaviour [6]

Intraply hybrids

Thanks to its modular design the HiPerDiF machine allows manufacturing intraply hybrids able to provide pseudo ductile behaviour [7,13].



Figure 4. Pseudo-ductile intraply hybrid behaviour.

Further Industrial Relevance of the HiPerDiF technology

ADFRCs manufactured with the HiPerDiF method have been laid-up in a quasi-isotropic configuration demonstrating mechanical performance superior to that of randomly oriented short fibre composite with similar volume fraction [14].

Moreover, intermingled Carbon-Flax hybrids showing promising vibration damping properties have been successfully produced [15,16].

Finally, the HiPerDiF method allows the remanufacturing of reclaimed carbon fibres into a recycled material with one of the best mechanical properties ever reported in the literature [17,6]. It also allows the development of a closed-loop recyclable material [18, 19, 20] and for the quality control and property assurance of reclaimed carbon fibres and recycled composites [21]

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B5.8 Final report: Laser modified fibres

Unduloid-reinforcing carbon fibres were produced using controlled laser irradiation which resulted in expanded and ablated regions along the fibre axis, Figure 1 [1, 2, 3]. These modulated carbon fibres were created at predetermined regions, and their diameters were locally increased up to 53%, forming outward taper angles of up to 1.8° and tested mechanically demonstrating that the majority of the single fibre stiffness, in tension, was maintained. When modified single carbon fibres were embedded into a compliant matrix and pulled-out, extensive ploughing from the taper ends contributed to a ~7-fold increase in work of pull-out compared to the as-received carbon fibres (Figure 2).

Subsequently, fibres could be laser modified multiple times along the fibre axis and be used to increase the strain-to-failure of composites. Expanded fibre regions would break, and the tapered ends which are formed would then mechanically interlock with the matrix and resist fibre pull-out. High powered laser treatment is a promising route to generate tapered carbon fibres, translating a key geometric feature of natural composites to the current state-of-the-art structural materials.



Figure 1. Laser irradiated carbon fibres and the various shapes produced.



Figure 2. Modified single fibre pull-out tests from a compliant matrix.

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B6 Final report: Hierarchical Bundle Composites

New classes of materials are required if we are to mimic natural structures which not only demonstrate high strength but also deform in a non-brittle manner. Nature's hierarchy with combinations of different fibres, matrices and interfaces at different length scales are utilized to create materials like bone, tendon, and plants etc. These natural materials have excellent damage tolerance but are challenging to reproduce synthetically. Exploiting hierarchy in the micro-structure of unidirectional fibre composites to enable progressive tensile failure was investigated through modelling (Figure 1) [1, 2] and through testing small fibre bundles [3].

Currently, continuous fibre reinforced composites fail when a relatively small critical cluster of broken fibres is formed, even if most fibres in the composite are still intact. This process is due to the stochastic variation of strengths of those individual fibres in composites, and to the fact that clusters of broken fibres lead to stress concentrations around their neighbours, causing premature failure of the composite structure. If these weak fibres/bundles can be isolated such that there is a reduction in the likelihood of the nearest neighbour failing when the weak fibre(s) fail, a more progressive global failure may be achieved.

The formation of these hierarchical micro- as well as macro-scale fibre bundles, or bundles-of-bundles from standard tows, is non-trivial and requires a steady hand! The range of fibres in a bundle was varied from 3 to 12 000 through a bottom-up, and top-down approach. An example of 20 carbon fibres is shown in Figure 2. These bundles were tested to validate the model and further work on creating bundles of bundles is on-going. Ideally the process of fabricating these bundles may be carried out at the point of fibre manufacture. This as well as the potential for varying the interface between the bundle-of-bundles, or within the bundles, has an exciting future.



Figure 1. Schematic of hierarchically modelled system shown with a coordination factor of 7.



Figure 2. Small composite bundles produced from a bottom-up fabrication route.

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B7 Final report: Interlaminar and ply weakening

Controllable pseudo-ductile tensile behavior can be achieved by tailoring ply weakening in unidirectional (UD) CFRP laminate made of single carbon system (Hexcel® M21/35%/198/T800S prepreg). This has been investigated through FE modelling [1] and validated by experiments [2]. Delamination initiating from ply weakening (either cuts [2] or perforation) in the middle plies sandwiched between continuous plies of a unidirectional (UD) laminate promotes a plateau stage in the stress-strain curve (see Fig. 1). Pseudo-ductility was also achieved in quasi-isotropic (QI) [3] and 0-degree fibre dominated multidirectional (MD) laminates. A 4-ply UD cut-ply laminate (in which the middle two plies contained periodic ply cuts and the outer plies were pristine) was used as the 90-, 0-, +45- and -45-degree direction sub-laminates of the QI laminates (see Fig. 2). The MD laminates are made of two 0-degree plies containing ply cuts embedded in two blocks of QI ([90/0/+45/-45]) material (see Fig. 3). These QI and MD configurations are notch-insensitive, as shown in the open-hole tensile (OHT) behaviour (see Fig. 2 and Fig. 3).



Figure 1. Pseudo-ductile tensile performance of a UD 4-ply laminate containing 10 mm spaced ply cuts in the two middle plies



Figure 2. Schematics of two types of QI laminates containing 10 mm spaced ply cuts in the two middle plies of each sub-laminate and the corresponding tensile and open-hole tensile (OHT) behaviour



Figure 3. Schematic of 0-degree fibre dominated multi-directional (MD) laminates containing 10mm spaced ply cuts in the middle two 0-degree plies and corresponding tensile and open-hole tensile (OHT) responses

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B8 Final report: Modelling discontinuous composites across the scales

A semi-analytical virtual testing framework was developed to predict the response of aligned discontinuous composites with single [1] and hybrid [2-3] fibre-types, as produced by the HiPerDiF manufacturing process.

This framework uses a shear-lag model to predict matrix damage, and Weibull statistics to predict fibre fragmentation; moreover, it uses a non-linear fracture mechanics criterion to identify the location, size and shape of the critical weak region triggering failure of the entire specimen. These models are formulated analytically, and integrated in the numerical simulation of a composite specimen with individual fibres and matrix represented explicitly (see Figure 1).

Due to the combination of analytical and numerical methods, this framework can be used to model full specimens (with millions of fibres) within minutes. The predictions of this framework have been compared against experiments, both for non-hybrid, and hybrid composites (see Figure 2) [1-3]. The virtual testing framework was also used to explore the influence of fibre-type arrangements on the pseudo-ductility of hybrid composites; this work showed that failure can be delayed by promoting the intermingling of different fibre types (see Figure 3) [3]. Moreover, the virtual testing framework can provide further insight on the effect of different sources of variability and defects on the failure process of composites [4].



- a) Representation of the specimen modelled.
- (i 1, j) (i 1, j) (i, j 1) (c) H(i, j) (i, j + 1) (i, j) (i + 1, j) (i + 1, j)
 - b) Fibre interacting with its 4 nearest neighbours.



c) Shear-lag elements along the interaction between fibres.

Figure 1. Overview of virtual testing framework for aligned discontinuous composites [2].



Figure 2. Comparison between model predictions and experimental results, for High-Modulus Carbon (HMC) /Glass hybrids (at different volume rations between HMC and glass) [2].



 Random intermingling of the two fibretypes within the cross-section.



- b) Preferential clustering of HMC fibres at the centre of the cross-section.
- Figure 3. Comparison between model predictions and experimental results, for High-Modulus Carbon (HMC) / Glass hybrids (at different volume rations between HMC and glass) [3].

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C2.1 Final report: High Performance Ductile Fibres

Single-walled carbon nanotubes (SWCNTs) are envisaged to have superlative mechanical properties. However realising these nanoscale properties on the macroscale level is challenging. One such route is to spin fibres containing SWCNTs from solutions. These SWCNT-based fibres have shown promise in providing a route to strong, tough, low density and ductile materials. Functionalised, orientated nanotubes aligned in the fibre's direction should produce a strain hardening effect under tension. Manipulation of reductive chemistry on SWCNTs to produce individual species further improves stress transfer between neighbouring SWCNTs, improving the macroscopic properties.



Figure 1. Schematic of the fabrication of Single-walled carbon nanotube (SWCNT) ductile fibres from starting nanotube bundles. SWCNTs are individualised in solution and functionalized before being spun into fibres.

Controlling the nanotube loading tailors the properties of the resulting composite fibre. Fibres with a high loading of SWCNTs (40 wt%) showed a tensile strength of ~1.4 GPa with a strain to failure of 12.7%. Compared to a low SWCNT loading (3 wt%) fibre, a reduced tensile strength of ~0.7 GPa was measured but with a higher strain to failure of 62%. The cyclic tests of both the high strength and high strain fibres demonstrated excellent ductility across the complete range of strains. This bottom-up approach provides a route for producing ductile responses at a fibre level. This development of ductile constituents is required for the realisation of a truly ductile composite.



Figure 2. Left: Tensile stress-strain curve for the high strength SWCNT-PVOH based fibre compared to commercially available Kuralon fibre. Right: Tensile stress-strain curve for the high strain SWCNT-PVOH based fibre compared to commercially available Kuralon fibres.



Figure 3. Left: Tensile stress-strain curve for the high strength SWCNT-PVOH based fibre compared to a fibre produced with the same method tested cyclically. Right: Tensile stress-strain curve for the high strain SWCNT-PVOH based fibre compared to another SWCNT-PVOH fibre tested cyclically.

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C2.2 Final report: Nacre-inspired interphase

Mechanical properties of fiber reinforced composites are greatly influenced by the fiber-matrix interphase. A strong interphase can result in high strength, while a weak one enables debonding that leads to a high degree of energy absorption. Achieving both properties in one composite interphase is a great engineering challenge.

The biopolymer nacre exploits a combination of crack deflection and strain hardening and maximizes energy absorption, while simultaneously limiting delamination. The reason is its "brick-and-mortar" structure of brittle inorganic building blocks, which are "glued" together by a soft organic framework (Figure 1). In reference to this "brick-and-mortar" structure of natural nacre, a nanostructured coating was developed with high proportion (~ 90 wt%) of well aligned inorganic platelets embedded in a polymer matrix, but uniformly scaled down by more than 1 order of magnitude (Figure 2). [1] - [4]



Figure 1. Natural nacre (left) and "brick and mortar" structure of nacre (right) [5], [6]



Figure 2. Nanostructured coatings deposited on a flat substrate (left) and a fiber (right).

A Layer-by-Layer (LbL) assembly resulted in coatings with ordered and dense layered nanostructure with a platelet misalignment as low as 8°. Assessment of the coating properties showed the known toughening mechanisms of nacre, such as platelet sliding and interlocking, as well as three-dimensional crack deflection. A similar elastic modulus and hardness close to nacre could be achieved, additional to larger plastic deformation in the material upon loading. [1]

Optimization enabled the transfer of the architecture of the coating to the curved surface of fibers, which showed an improvement of up to 30% upon loading in shear. Bundles of fibers could be coated homogenously via LbL and a scale-up to coat continuous fibers by adding additional baths to the traditionally used sizing line should be easily feasible. The nanostructure coated fibers showed reduced local stress concentrations arising from fiber breaks and increased extend of fiber slippage compared to untreated ones. The results were in good agreement with the results on flat substrates. A balance of simultaneously strength and toughness could be achieved on a small scale and leads a promising way for future large-scale composite tests. [3][4]

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C3 Final report: High performance but ductile matrices

Aerogel modified matrices

Where existing materials are not adequate, our emerging understanding is that stiff, yet plastic matrices with strong strain hardening characteristics will be desirable. Bi-continuous hybrid aerogel-polymer matrices (Figure 1) may provide a nano-reinforcement which both reduces stress concentrations at broken primary fibres and a strain hardening response in shear [1, 2]. Fibre breaks should be localised but allowed to slide to a limited extent, to introduce (pseudo)ductility. It is envisaged that the highest benefit in use of aerogel-matrix reinforced fibre composites will be in compression where lateral stiffening to limit fibre micro-buckling is promoted [3, 4].



Figure 1. Schematic of woven fabric embedded in an aerogel.

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C4 Final report: Ductile Carbon Nanotube Fibre Composites

Carbon nanotube fibres can show high strains and ductility, with scope to create ductile composites. Single CNT fibre fragmentation tests with a model high strain matrix of polycarbonate were carried out. It was shown that it is possible to achieve a high performance CNTF/Polymer interface by careful consideration of interface morphology [1]. An interfacial shear strength of 50 MPa, with adhesion maintained at very large strains of up to 13% was successfully demonstrated, offering the possibility of exploiting the mechanical and functional performance of CNTF in high performance ductile polymer composites.



Figure 1. Birefringence patterns indicating continued adhesion at very high strain

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C6 Final report: Optimised Matrices

Compression

To inhibit shear instability of the fibres in a composite in compression, polybenzimidazole (PBI) was examined as a composite matrix, given its high modulus (5.9 GPa). Several processing methods were explored (solution processing and heat pressing proved difficult to mill using both jet- or cryo-milling) [1]. Ionic liquids were employed as a greener alternative to conventional solvents as 1-ethyl-3-methylimidazolium acetate has been reported in this context. PBI was dissolved successfully and cast on (sized-removed) bi-directional carbon fibre plies. A specimen was fabricated and tensile tested as 1 ply composite to demonstrate fibre-matrix consolidation. The elastic modulus was measured as (33.99 ± 3.51) GPa.





Using the electrospinning rig at the University of Surrey, PBI nanofibres were successfully fabricated. This was of interest as the fabrication of nanofibres allows easy and complete removal of solvent without further treatment (vacuum oven etc.). In addition, the nanofibres can be cast straight on to the CF ply.



Figure 2: SEM image of the PBI nanofibres.

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C7 Final report: Ductile Superlattice Nanoparticle Matrices

Ductility via Dislocation-based Deformation in an Ordered Supercrystal Matrix

In a metal, dislocations allow high levels of deformation and require significant atomic ordering but suffer from high densities. A similar but lightweight approach is the use of metal oxide nanoparticles coated with ligands. These structures are expected to yield under high shear forces whilst exhibiting high strength. To further improve the ductility (as well as allowing self-healing) of these materials, cross-linking is performed with a unit containing a central disulphide bond, which easily opens and closes.

Metal-oxide nanoparticles were synthesised and cross-linked to produce a composite then tested using a nanoindentator, and the indentation properties compared to a standard epoxy resin. The metal-oxide superlattice showed a similar mechanical response in terms of stiffness and strength compared to epoxy, however it is capable of significantly more plastic deformation, with plasticities of >0.80 and 0.41 respectively. Metal-oxide composites which fail with a truly plastic deformation similar to metals is a new class of materials that can potentially compete with commercially available materials that exhibit high strength and stiffness.



Figure 1. (a): Schematic of ligand-coated nanoparticle superlattice. (b): Schematic of cross-linked metal-oxide nanoparticles with central disulphide bond.



Figure 2. Comparison of an epoxy with the cross-linked metal-oxide superlattices.