CHARACTERISATION OF HIGHLY-ALIGNED, DISCONTINUOUS, FIBRE COMPOSITES FOR COMPRESSIVE PERFORMANCE

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ABSTRACT

A new generation of advanced composites, featuring three dimensional architectures which mimic the multi-scale reinforcement seen in biological composite materials such as bone and wood are currently being developed. As with their natural antecedents, these novel manufactured materials are being designed to incorporate hierarchical systems of discrete structural elements which couple mechanistic properties across length scales. The aim of this programme of research is to significantly improve the compressive load bearing characteristics of reinforced polymeric composite materials.

Highly aligned, discontinuous carbon-fibre composite tapes promise to be useful structural elements across both meso- and macro-levels within the architectures being considered. The high alignment of the fibres can allow mechanical properties comparable to continuous fibre tapes, whilst the intrinsic pliability of the tapes favours the formation of complex structural geometries. This paper presents a discussion of the challenges encountered in characterising these materials in compression using a novel four-point bending test, and outlines the remedial actions taken.

1 INTRODUCTION

Despite over 60 years of active research, and considerable improvements in properties relating to tensile strength and impact resistance, compressive strength levels in manufactured composites are still some 40% lower than measured longitudinal tensile values. This disparity represents a continuing barrier to further industrial adoption of these materials.

Hierarchically-structured, natural composite materials such as bone, Figure 1, wood, and shell feature discrete but complementary reinforcement systems across multiple length scales which allow load carrying capacities significantly above those of their intrinsically weak constituent materials [1]. The EPSRC supported 'Next Generation Fibre-Reinforced Composites' (NextCOMP) [2] programme grant is developing novel manufactured composite materials which mimic such architectures, with the aim to significantly improve measurable compressive performance.

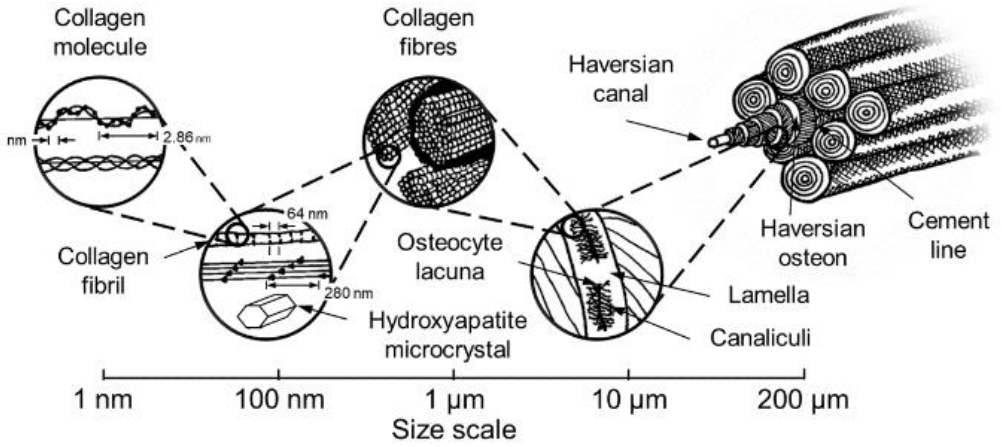


Figure 1: Illustration of the multiscale hierarchy present in human bone, a biological composite material. Adapted from Lakes [1].

Discontinuous fibre composite materials have processing and formability properties which could provide a useful element within such multi-scale, higher order hierarchical structures. As overall fibre alignment is a fundamental determinant of their mechanical performance, processing methods which can produce highly-aligned discontinuous fibre composites offer the opportunity of retaining a useful proportion of the strength of comparable continuous fibre materials [3]. The patented 'High-Performance Discontinuous Fibre' (HiPerDiF) [4] process developed at the University of Bristol is a proven method for producing composite tapes featuring highly-aligned discontinuous fibres of between 1 mm and 12 mm in length, utilising water as a carrying medium. Given a fibre aspect ratio high enough to allow load transfer without fibre pull out, the process produces materials with tensile properties approaching those of continuous fibre composites [5]. Aligned discontinuous fibre composites have also shown strong promise in overcoming certain limitations of continuous fibre materials when considering possible consolidation methods within three-dimensional architectures. Namely: tailoring and functionalisation of composite properties though the hybridisation of discrete fibre geometries; offering the potential for high volume, defect-free automated production of complex structural geometries, a consequence of the intrinsic pliability of the uncured tapes; and the opportunity to integrate reclaimed materials, a prerequisite of a sustainable, circular economy for manufactured composites [6].

2 METHODS AND MATERIALS

There is currently little published analysis of the failure characteristics of aligned, discontinuous, carbon-fibre reinforced polymer (ADCFRP) composites under compressive loading. As a first step in determining the utility of ADCFRP for the novel biomimetic composites discussed above, research is being undertaken which aims to characterise a range of ADCFRP composites.

Initial testing has focused on ADCFRP material produced on the HiPerDiF equipment located at the University of Bristol. This produces dry fibre preforms of aligned 3mm length, HTC-124 carbon fibres supplied by Toho-Tenax, Table 1 [7]. The preforms were cut to the required sample length and individually consolidated with Toray K51 [8] epoxy resin film using a heated belt system set to 80°C, Figure 2 (a & b). Test samples were then created within a bespoke mould by laying up eight preform / epoxy tapes per sample and consolidating with hand pressure, Figure 2(c). A cured sample laminate thickness of 0.8mm was selected for investigation with the samples cured by autoclave to the manufacturer's specifications, Figure 2(d).

Fibre Type	Fibre Length (mm)	Filament Diameter (µm)	Fibre Elastic Modulus (GPa)	Fibre Density (g/cm^3)	Fibre Failure Strain (%)	Fibre Tensile Strength (GPa)	Epoxy Resin	Lamina CPT (µm)
TohoTenax C124 (HSC)	3 (a)	7 (a)	225 (a)	1.82 (a)	1.93 (a)	4.35 (a)	K51	91 (b)

Table 1: Discontinuous fibre properties (a - adapted from Longana *et al* [9] , b - measured value).

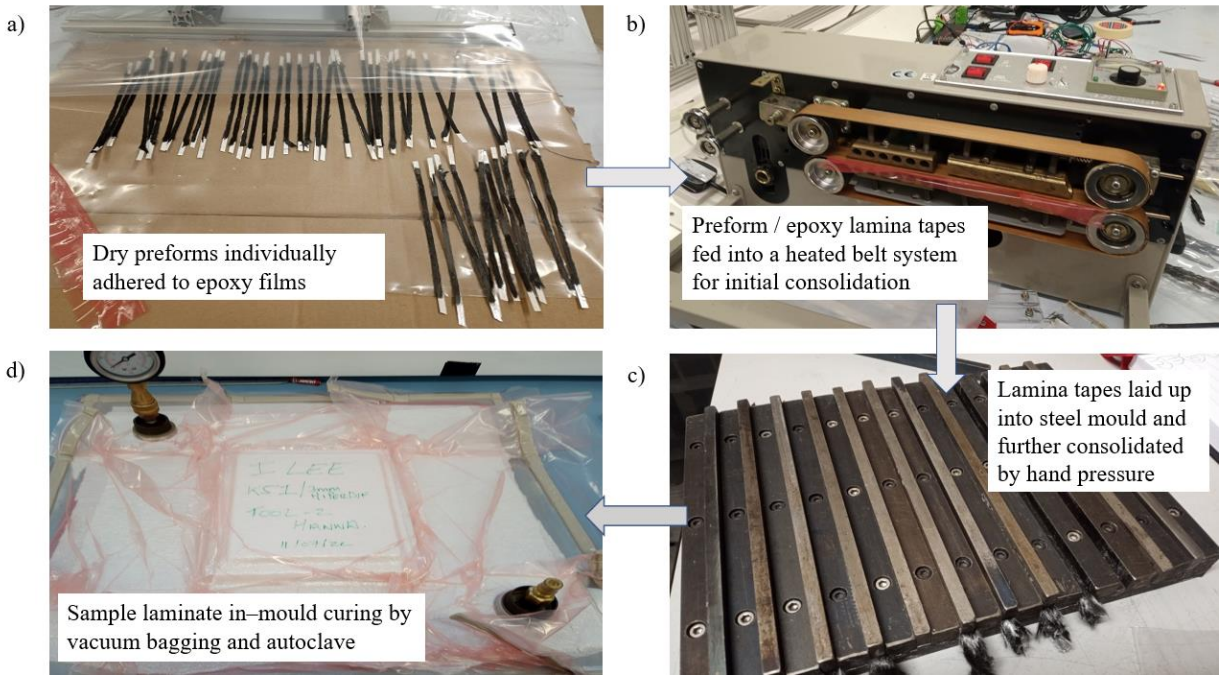


Figure 2: Discontinuous fibre sample preparation method, clockwise from top left – a) adhering dry preform to epoxy film, b) tape consolidation using heated belt system, c) lamina tape lay-up in bespoke steel mould, d) in-mould sample curing by autoclave.

To overcome known difficulties [10] in accurately determining compressive response in non-linear material systems such as ADCFRP tapes, a novel four-point bending test developed within the NextCOMP programme was selected as the characterisation method [11]. For this project cured discontinuous fibre samples with a geometry of 150 mm x 5 mm x 0.8 mm are individually adhered using an ethyl 2-cyanoacrylate adhesive [12] to test beams machined from poly(methyl methacrylate) (PMMA), Figure 3(a). PMMA was selected as the test beam material due to exhibiting strain characteristics (<2% of the sample material) which ensure it does not fail in preference to the samples during testing. The beam geometry and gauge length were determined through use of the modelling software Abaqus, based on the calculated critical bending loads for the materials, to promote sample failure within their unsupported sections.

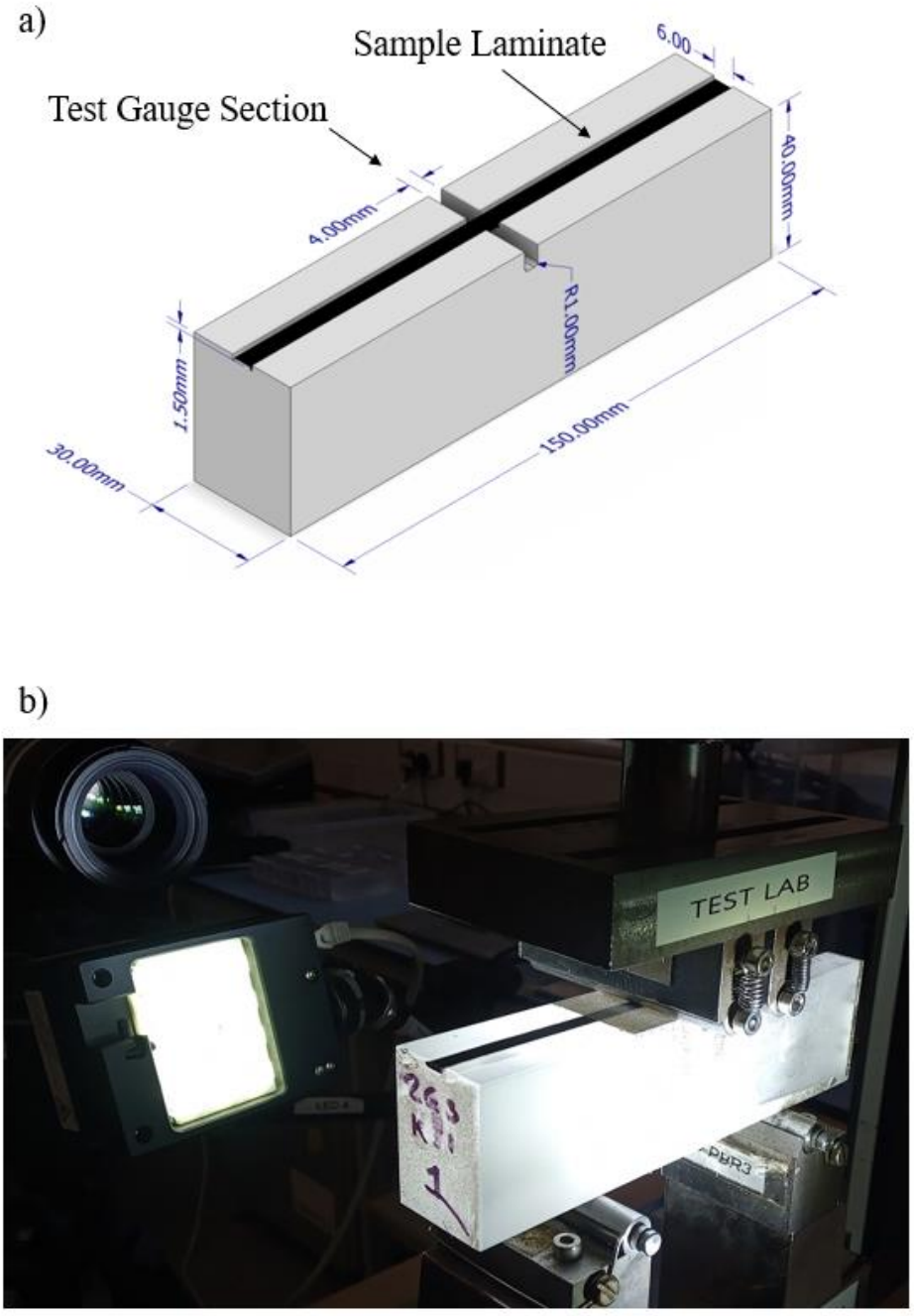


Figure 3: a) Schematic of PMMA test beam, showing sample (black) within a longitudinal machined channel above a 4mm width central gauge section; b) ADCFRP sample adhered to PMMA test beam, under four-point bending.

Testing was carried out on a Roell-Amsler universal test unit with a 25 kN load cell, and head displacement set to 1 mm / min, Figure 3(b). The strain rate for the samples was captured through use of LaVision Digital Image Correlation (DIC) equipment [13]. A 5-megapixel camera set-up was utilised, to image the samples longitudinal edges within their gauge sections. Ten samples of cured 3 mm discontinuous fibre ADCFRP laminates, generated on the second generation HiPerDiF unit (2G-3), were prepared for DIC imaging through the application of a base layer of white paint and a random black speckle pattern, with sample H16 left unpainted to allow for subsequent SEM imaging. Sample failure data was post-processed within the LaVision software.

3 RESULTS

Maximum strain (%) vs load (kN) data was gathered by virtual extensometer and charted for ten painted samples, Figure 4. Figure 5 is a scanning electron microscope (SEM) image of sample H16, which was left unpainted to allow improved post-failure surface imaging. A DIC image failure timeline for sample H6 is illustrated in Figure 6 (a-d), which is indicative of the samples tested.

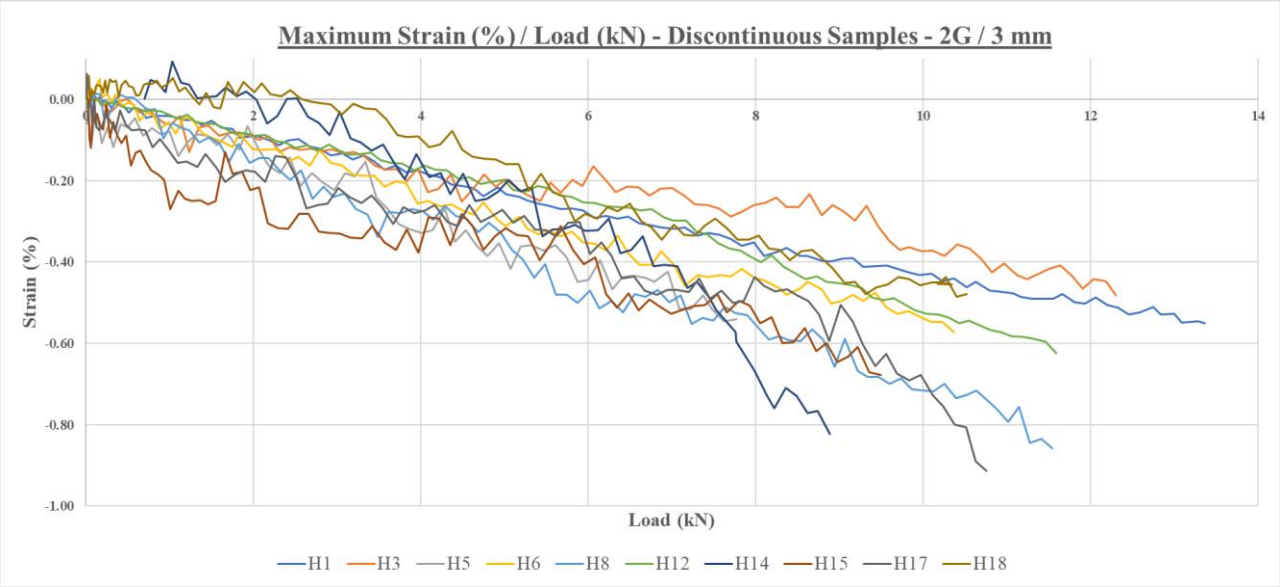


Figure 4: Maximum Strain (%) / Load (kN) for 2G-3 samples.

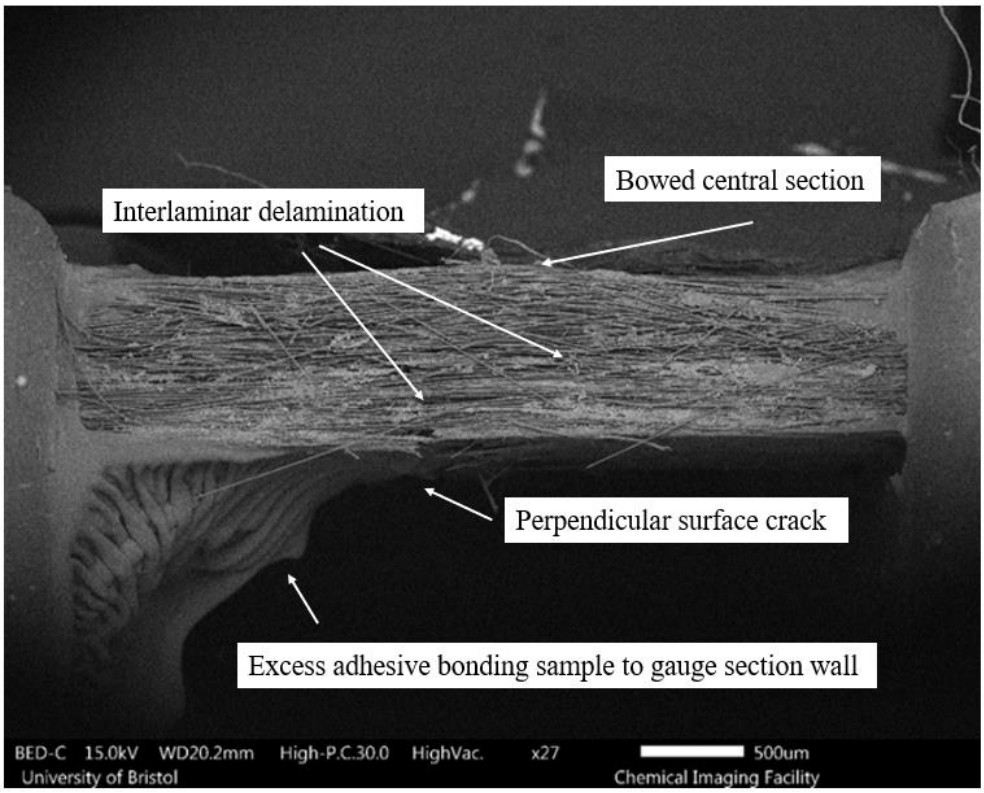
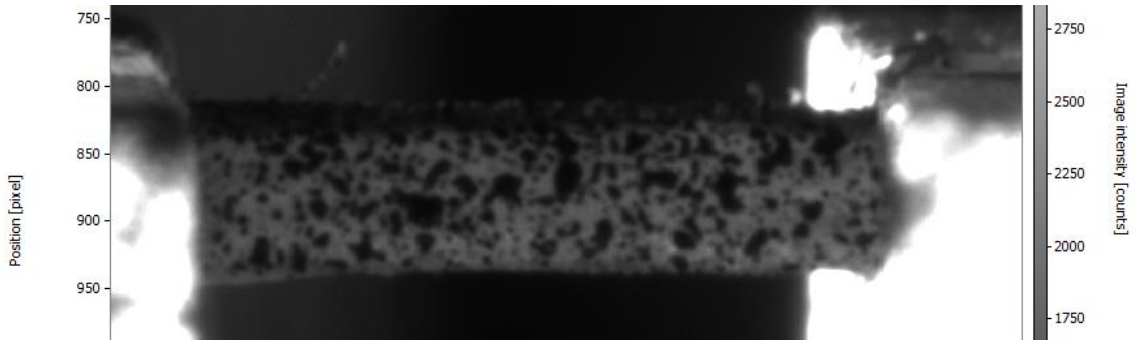
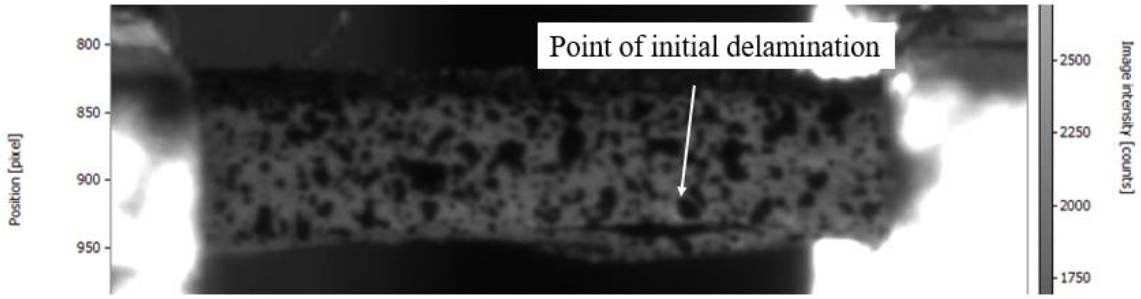


Figure 5: SEM micrograph of unpainted sample H16, scale bar length 500 μm.

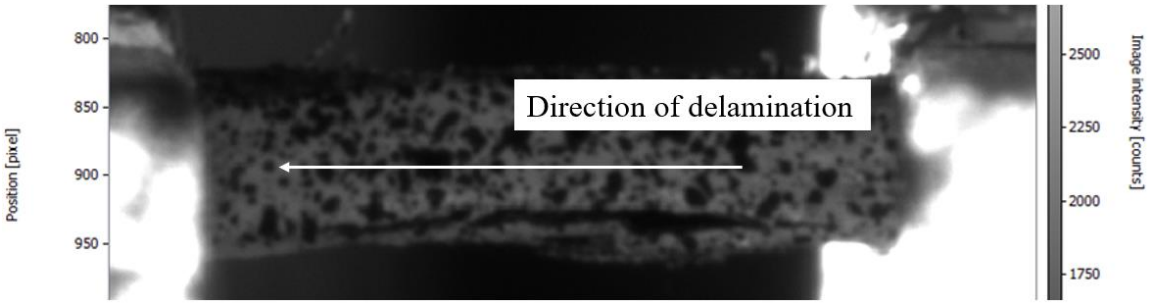
a) Sample H5 – Camera 1 – 22 secs



b) Sample H5 – Camera 1 – 32 secs



c) Sample H5 – Camera 1 – 39 secs



d) Sample H5 – Camera 1 – 41 secs

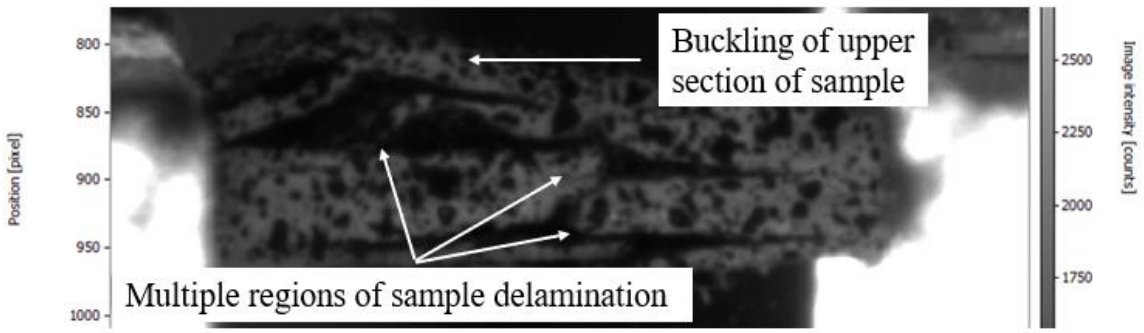


Figure 6: A timeline of failure for sample H5, illustrating progressive interlaminar delamination prior to an eventual buckling of the remaining material.

4 RESULTS DISCUSSION AND METHOD REFINEMENT

The data captured for the samples display a general linear response to loading, however there are notable disparities between samples both in terms of maximum recorded strain and load at failure. The range of sample ultimate strain is 0.47%, between -0.48 % and -0.96 %, with a mean value of -0.69 %, median value of -0.57 %, and standard deviation of 0.18 %. The range for load is 5.6 kN, between -7.8 kN and -13.4 kN, with a mean value of -10.7 kN, median value of -10.6 kN, and standard deviation of 1.7 kN. There is also considerable noise in the data due to the selection of a virtual extensometer to measure strain.

Investigation of the DIC images indicate that although all the samples were seen to fail within the test gauge length, the ultimate failure mode was not compressive but the compound effect of material delamination and buckling. As Figure 6 (b) illustrates, a single point of interlaminar debonding on the bottom right-hand side of sample H5 becomes the locus for a broader longitudinal delamination. The reduction in material integrity allows for a subsequent buckling in the upper section of the sample which ultimately causes the sample to fail, with the energy released at failure causing further delamination through the sample. Delamination prior to failure was evident to a greater or lesser extent in all the samples tested.

SEM imaging was carried out on a selection of the tested samples. Figure 5 illustrates the failure surface for the unpainted sample H16, displaying a through thickness crack on the bottom edge of the sample running perpendicular to the gauge section width. The left-hand side of the gauge length indicates an unwanted spread of the adhesive used to adhere the sample within its channel, which is anticipated to have affected the location of this crack. The bowed central section of the sample is evidence of the sample having buckled as in Figure 6. Post-loading relaxation within the sample has allowed closure of the longitudinal discontinuities, however delamination is still evident in the image, and the lack of a painted surface clearly identifies an overall lack of fibre-matrix consolidation in the sample.

Following investigation of the test data several remedial actions were identified to refine the testing methodology. Temporary masking of the gauge section will be carried out to control the spread of adhesive and a polymer self-adhesive tape applied perpendicularly around the jig and sample either side of the gauge section, will be used to contain any potential out of plane movement of the sample during loading. Future data capture will utilise a double camera set up, such that imaging both longitudinal edges of the gauge section will allow for determination of any out of plane buckling exhibited by the samples. The speckling pattern will be refined through use of an airbrush to apply black ink, which will improve contrast and control of speckle size, reducing data noise levels. It is anticipated that imaging whole strain field data for the sample rather than use of a virtual extensometer will also reduce noise levels. Finally, samples with the same geometry but machined from a material with well defined properties, such as a common aerospace aluminium alloy, will be tested alongside the ADCFRP material to provide a benchmark for the test method.

Several refinements to the sample processing method have also been implemented to improve sample properties for future testing. As the lack of matrix / fibre consolidation was determined to be the primary element promoting delamination, and to ensure full wetting out of the dry preform, the decision was taken to double the epoxy to fibre ratio per preform section. The steel mould was re-machined to improve fitting tolerances and an intermediate vacuum consolidation step (5 mins @ -1.0 bar) was included after laying up four lamina films. Figure 7 is an SEM micrograph of material created with this refined manufacturing method and the perpendicular cut surface indicates significantly improved fibre-matrix consolidation. It is hypothesised that the modifications identified and undertaken should allow for identification of a compressive failure mode in forthcoming testing.

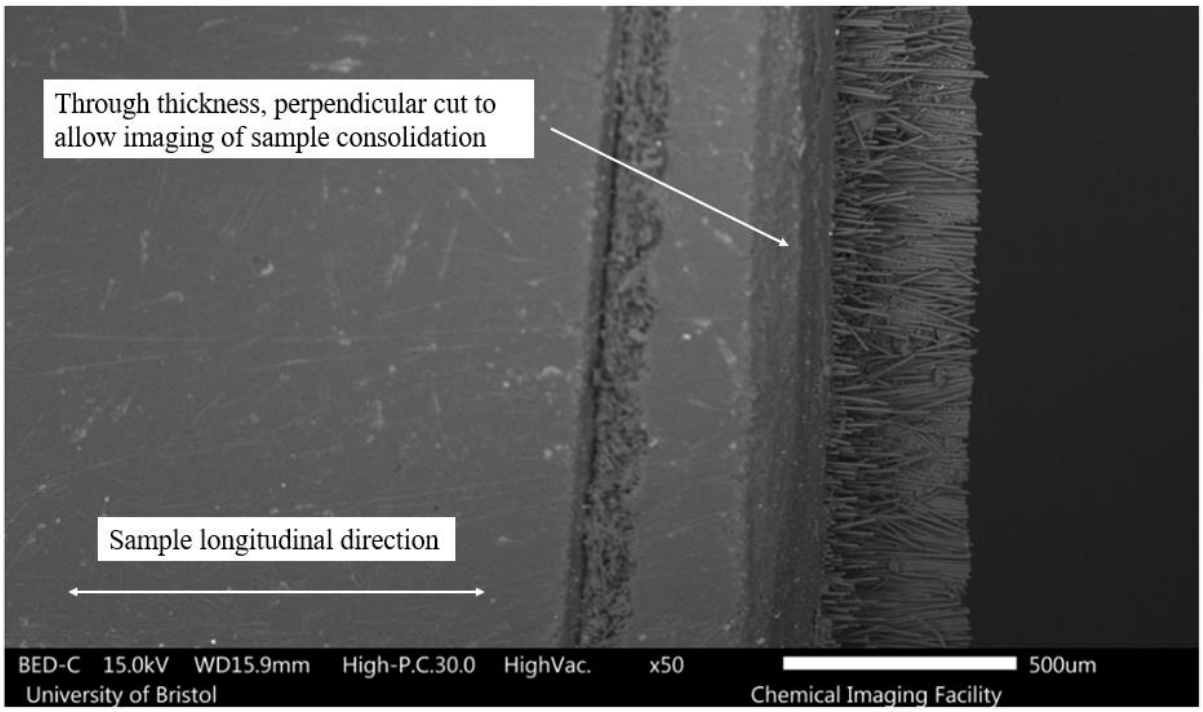


Figure 7: SEM micrograph of refined material, scale bar length 500 μm.

5 CONCLUSIONS

Initial testing has indicated the potential for a novel four-point bending test method to induce a compressive strain within highly aligned, discontinuous fibre composite samples. However, data for ultimate failure strain and failure load were unable to be generated due to delamination of the samples, a consequence of inadequate fibre / matrix consolidation. Several processing and methodological refinements have been identified and implemented which it is hypothesised will significantly improve the validity of future data.

The results described in this paper form part of a wider testing programme which will characterise a range of highly aligned, discontinuous fibre composites generated by the HiPerDiF process, and assess their suitability for the novel three dimensionally architectured advanced composite materials currently being developed by the NextCOMP research programme.

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REFERENCES

[1] R. Lakes, "Materials with structural hierarchy," *Nature* **361:6412**, 1993, pp. 511–515 (doi: 10.1038/361511a0)
[2] "NextCOMP." https://nextcomp.ac.uk/. Accessed April 20, 2023.
[3] M. Such, K. Potter, and C. Ward, "Aligned Discontinuous Fibre Composites: A Short History," *Journal of Multifunctional Composites* **3**, 2014, pp. 155–168 (doi:10.12783/issn.2168-4286/2/3/4/Such)
[4] "HiPerDiF | Bristol Composites Institute | University of Bristol." https://www.bristol.ac.uk/composites/research/hiperdif/. Accessed April 20, 2023.
[5] H. Yu, K. D. Potter, and M. R. Wisnom, "A novel manufacturing method for aligned discontinuous fibre composites (High Performance-Discontinuous Fibre method)," *Composites Part A Applied Science & Manufacturing* **65**, 2014, pp. 175–185, (doi: 10.1016/j.compositesa.2014.06.005)
[6] M. L. Longana, H. Yu, J. Lee, T.R. Pozegic, S. Huntley, T. Rendall, K.D. Potter, I. Hamerton "Quasi-Isotropic and Pseudo-Ductile Highly Aligned Discontinuous Fibre Composites Manufactured with the HiPerDiF (High Performance Discontinuous Fibre) Technology," *Materials* **12**, 2019, p. 1794 (doi: 10.3390/ma12111794)
[7] "Tenax® HT C124 - Toho Tenax Europe GmbH - PDF Catalogs | Technical Documentation | Brochure." https://pdf.directindustry.com/pdf/toho-tenax-europe-gmbh/tenax-ht-c124/37818-629939.html. Accessed April 20, 2023.
[8] "Thermoset Composite Materials | PRODUCTS | Carbon Fiber Composite Materials | TORAY." https://www.cf-composites.toray/products/thermoset/. Accessed April 20, 2023.
[9] M. L. Longana, H. Yu, K. D. Potter, M. R. Wisnom, and I. Hamerton, "The HiPerDiF (High Performance Discontinuous Fibres) technology for the manufacturing of pseudo-ductile quasi-isotropic aligned discontinuous fibre composites" in *ECCM 2018 - 18th European Conference on Composite Materials, Athens, Greece, June 25-28, 2018, Applied Mechanics Laboratory*, pp. 24–28.
[10] C. R. Schultheisz and A. M. Waas, "Compressive failure of composites, part I: Testing and micromechanical theories," *Progress in Aerospace Sciences*, **32**, no. 1, 1996, pp. 1–42 (doi: 10.1016/0376-0421(94)00002-3)
[11] G. Quino, P. Robinson, and R. Trask, "Design of a bending experiment for mechanical characterisation of pultruded rods under compression," in *Composites Meet Sustainability – Proceedings of the 20th European Conference on Composite Materials **3**, ECCM20, (Eds. A.P. Vassilopoulos, V. Michaud), Lausanne, Switzerland, June 26-30, 2022, CCLab, EPFL, Lausanne, p.167* (doi: 10.5075/epfl-298799_978-2-9701614-0-0)
[12] "Safety Data Sheet - Gorilla Super Glue". Available: www.gorillatough.com. Accessed: Apr. 13, 2023.
[13] "LaVision DIC Software." Available: https://www.lavision.de/en/products/strainmaster/system-components/dic-software/. Accessed Jul. 31, 2022.