

THE HIGH PERFORMANCE DISCONTINUOUS FIBRE (HIPERDIF) TECHNOLOGY FOR CONSISTENT QUALITY CONTROL OF RECLAIMED CARBON FIBRES

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Abstract

This paper describes the ongoing development of an alternative, improved method for the quality control of reclaimed carbon fibres based on materials manufactured with the HiPerDiF method. The HiPerDiF method, invented at the University of Bristol, allows remanufacturing aligned short fibre specimens that are representative of unidirectional composites manufactured from reclaimed carbon fibres (rCF). Two different specimen types are taken into consideration: 100% rCF and interlaminated hybrid specimens made of a layer of aligned short rCF sandwiched between continuous glass fibres. The obtained failure strain results are compared with results obtained from single fibre tensile test (SFTT): the interlaminated hybrid specimens allow avoiding premature failure caused by stress concentration in the end-tab region. Further work is needed to be able to efficiently retrieve the stiffness related properties.

1. Introduction

The wide spread of carbon fibres reinforced polymers (CFRPs) in various engineering and industrial sectors over the last decades poses the challenge of dealing with production waste and end-of life products. In the case of the thermosetting-based composites, research is moving towards recycling processes that comprise of a reclaiming stage, where the fibres are retrieved by degrading the matrix, and a remanufacturing stage to produce a reusable material. Pimenta and Pinho presented a complete review about the technologies to recycle CFRPs for structural applications [1]. Amongst the fibre reclamation processes, it is worth mentioning pyrolysis [2], oxidation in fluidised bed [3] and supercritical fluids [4]. Of greater interest for the presented work is, independently from the fibre recovery process, the remanufacturing of the reclaimed fibres into CFRP. The size-reduction of CFRP waste before reclamation, the fibre breakage during reclamation and the chopping of the fibres after reclamation lead to fibres that are averagely fragmented in short length. As a result, the only industrially relevant remanufacturing processes for reclaimed fibres so far are direct moulding techniques [5] and the compression moulding of intermediate random or aligned mats [6]. However, to deliver improved recycled materials, a high fibre alignment is the key factor to increase the fibre volume fraction, and consequently the performances of recycled composites [7]. Various techniques, already used for the alignment of short fibres, have been taken in consideration for the remanufacturing of reclaimed carbon fibres, such as modified papermaking technique [8], centrifugal alignment rig [9] and hydrodynamic spinning process [9].

The HiPerDiF (High Performance Discontinuous Fibre) method, invented at the University of Bristol [10], has proven to be an effective way to manufacture composite materials with high levels of alignment from short fibres. This unique fibre orientation mechanism uses the momentum change of a water-fibre suspension to align the fibres. It was previously noted that tensile modulus, strength and failure strain

of aligned discontinuous fibre composites produced with the HiPerDiF method were close to those of continuous fibre composites [11, 12]. The use of the HiPerDiF method not only has the potential for high volume production of high performance recycled carbon fibre composites from reclaimed short carbon fibres, but also can be used as a reclaimed fibres quality control tool.

Single fibre tensile testing is a laborious job that requires not only the correct preparation of a high number of specimens but also the need to accurately measure the fibres diameters to obtain reliable moduli and failure values. Several batches of different CFRPs can be processed daily in a recycling plant and a quality control process based on SFTT can hardly keep up with the production rate. The current paper suggests a new approach based on more commonly used and simple tensile tests on aligned short fibre specimens manufactured with the HiPerDiF method. Two approaches are presented here: one is based on tensile test of 100% rCF specimens and the other of interlaminated hybrid specimens where the rCF are sandwiched between layers of continuous glass fibres.

2. Materials and Manufacturing

2.1 Materials

Recycled carbon fibres (AS4, Hexcel) from a M56 resin composite with the pyrolysis “cycle B” process defined by Pimenta and Pinho in [13] have been used. The mechanical properties, measured with single fibre tests over a gauge length of 15 mm, are summarised in Table 1.

Table 1. Fibres properties

Fibre properties		
Diameter	[μm]	6.5
Length	[mm]	3
Density	[g/cm^3]	1.79
E_{11}	[GPa]	230
Failure σ_{11}	[MPa]	1110
Failure ε_{11}	[%]	0.5

The fibres are remanufactured in aligned short fibres preforms with the HiPerDiF method, described below, and impregnated using MTM49-3 epoxy resin. For the interlaminated hybrid specimens, S-glass epoxy prepregs (SG913, Hexcel) were used.

2.2 Fibre alignment process

In the HiPerDiF process for producing highly aligned discontinuous fibre preforms, invented at the University of Bristol, short fibres suspended in water are accelerated through a nozzle and directed in a gap between two parallel plates. The fibre alignment mechanism relies on a sudden momentum change when the suspension hits the plate. The aligned fibres fall on a perforated mesh conveyer belt and the suspension medium is removed by suction. The aligned fibre preform is finally dried with infrared radiation to allow the resin impregnation process. The HiPerDiF process allows the alignment of fibres between 1 and 6 mm in length. A schematic of the HiPerDiF short fibre alignment machine is shown in Figure 1.

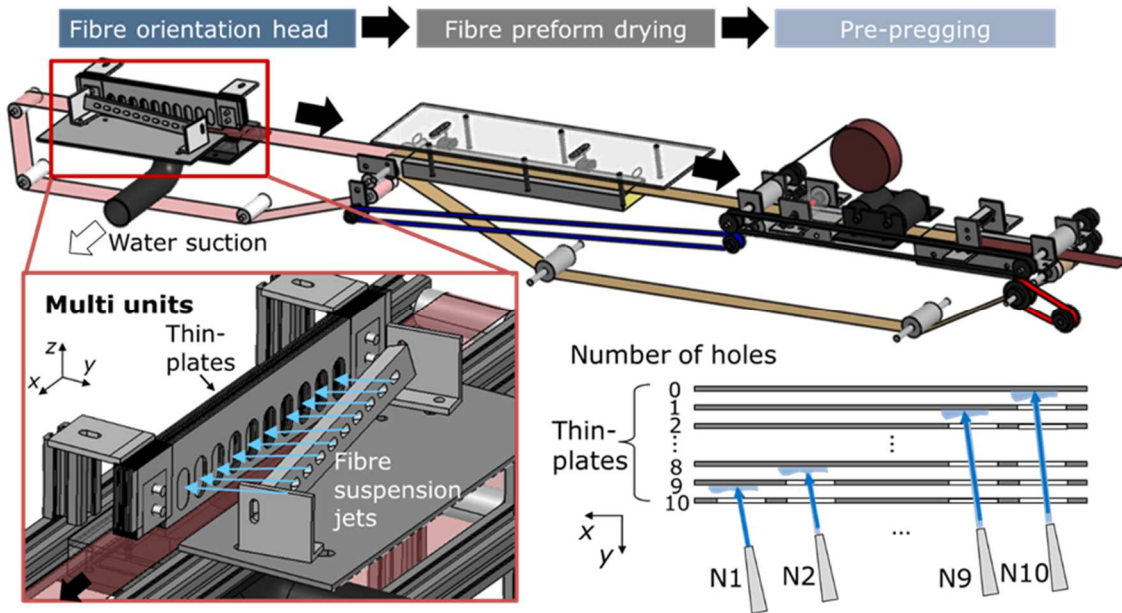


Figure 1. HiPerDiF fibre alignment machine.

2.3 Specimen manufacturing

The specimens were prepared by vacuum bag moulding. The 100% rCF composites were cured in an autoclave for 135 minutes at a temperature of 135°C and a pressure of 6 bar. The interlaminated hybrid composites were manufactured by coupling continuous S-glass fibre prepreg (ContG) with dry discontinuous fibre preform (rCF) following the stacking sequence [ContG₂/rCF₂/ContG₂], the reason of this choice is presented below. The specimens were cured according to the curing cycle of the continuous glass prepreg, i.e. 105 minutes at a temperature of 125°C and a pressure of 6 bar. Burrs at all edges of cured samples were gently removed. GFRP end-tabs were bonded with Huntsmann Araldite 2014-1 adhesive. A schematic of the specimen is shown in Figure 2a. The nominal thickness of the intermingled 100% rCF composites, manufactured with four plies of discontinuous carbon fibres, is 0.22 mm. The nominal thickness of the interlaminated hybrid composites is 0.56 mm. A view of the cross-section is shown in Figure 2b.

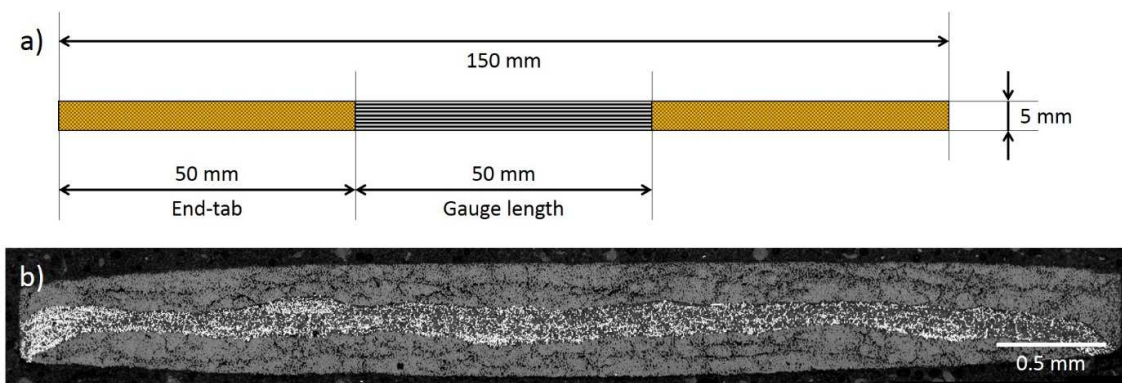


Figure 2. Specimen geometry:
 a) Top view;
 b) Interlaminated specimens cross-section.

3. Experimental Work

3.1 Interlaminated specimens design

The aim of the interlaminated hybrid specimens is to avoid the stress concentration caused by the end-tabs and be able to better define the failure strain of the internal layer of discontinuous rCF. Damage Mode Maps (DMM), proposed conceptually in [14] and further developed analytically in [15, 16], are an efficient design tool to tailor the material response and customise the stress-strain curve shape of interlaminated hybrid specimens. Since the failure strain of the low strain material, i.e. the aligned short rCF, could be only estimated by the data provided in [13] a new type of DMM with failure strain of the low strain material, i.e. the rCF, on the horizontal axis has been developed. In this case, the DMM are drawn to define the ratio between the rCF layer and the continuous S-glass layer that allows generating diffused fragmentation of the rCF, therefore, the ratio between the thickness of the carbon and the glass layer has been selected as vertical axis. To generate the DMM the thickness on the continuous glass layer is kept constant and the total thickness of the specimens is changed to consider the different thickness ratios. Similarly to the method presented in [14], the boundaries between different damage process zones are found by equating pairs of required stress for different failure modes of (i) high strain material failure, (ii) low strain material fragmentation and (iii) delamination. The DMM obtained considering the properties of the used S-glass is shown in Figure 3.

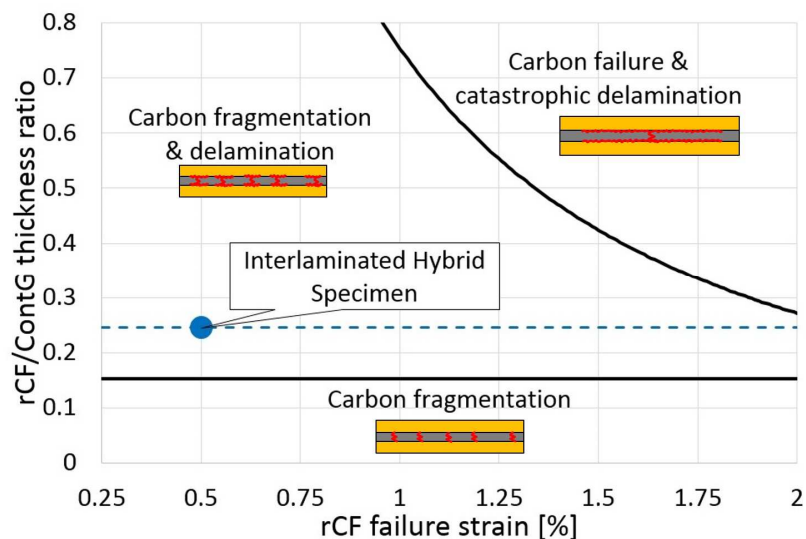


Figure 3: Damage Mode Map for interlaminated hybrids

From Figure 3 it is easily understandable that choosing a ratio of 0.25 allows obtaining fragmentation and diffused delamination, this not only allows identifying the rCF layer failure from the stress-strain curve but also visually observing the damage progression on the specimen surface during testing. Moreover, even if the rCF failure strain is higher than the one predicted by the SFTT this allows keeping the specimen in the same area of the DMM, as shown by the blue line in Figure 3. Observing Figure 3, it is easy to understand that, with the mechanical properties of the available materials it is not possible to achieve the configuration that allows single carbon failure.

3.2 Experimental methodology

Tensile tests were performed on an electro-mechanical testing machine with a cross-head displacement speed of 1 mm/min. The load was measured with a 10 kN load cell (Shimadzu, Japan) and the strain was measured with a video extensometer (Imetrum, UK). A white speckle pattern over a black

background was spray painted on the specimens to allow the strain measurement with the video extensometer. The gauge length for the strain measurement was around 40 mm.

3.3 Experimental results

The stress-strain curves obtained from the tensile tests are shown in Figure 4.

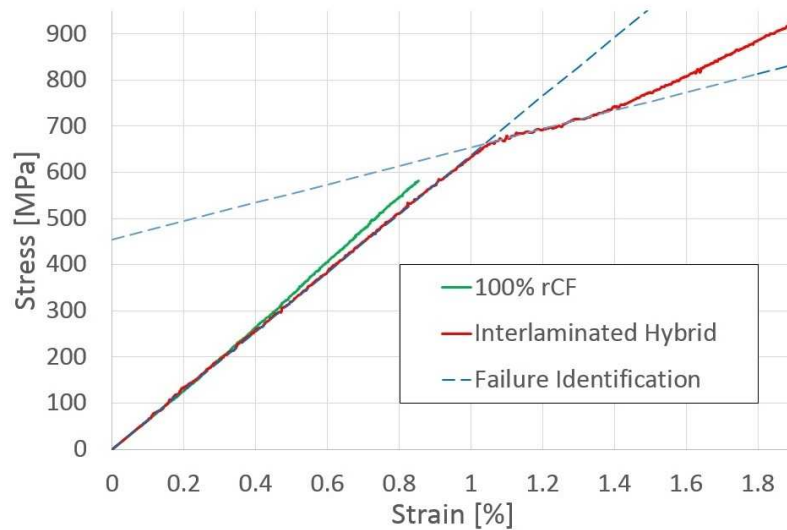


Figure 4. Stress-strain curves from tensile tests

It must be noted that the fibre volume fraction of the two specimens is different, 35% for the 100% rCF and 55% for the Interlaminated Hybrid, this explains why the two radically different sets of specimens have the same stiffness. It is straightforward to notice that the knee point of the interlaminated hybrid specimens, i.e. the beginning of the rCF fragmentation, is higher than the failure load of the 100% rCF specimens. This is believed to be because in the 100% rCF specimens the end-tab stress concentration causes premature failure. Figure 5 shows a specimen after it was subjected to a strain of 3%: in accordance with the prediction of Figure 3, the surface shows the striped pattern characteristic of fragmentation and the diffused delamination damage mode.

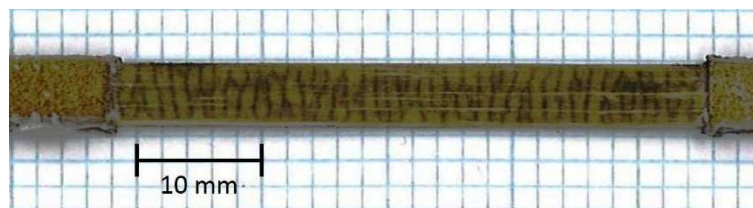


Figure 5. Interlaminated hybrid specimen with the characteristic striped pattern from the fragmentation and localised delamination.

The rCF failure for the Interlaminated Hybrid specimens was determined from the intersection of straight lines along the initial slope and the fragmentation plateau, as shown in Figure 4. A comparison of the obtained values is presented in Table 2.

Table 2. Measured rCF failure strain

	SFTT	100% rCF	Interlaminated Hybrid
Failure ϵ_{11} [%]	0.5	0.84	1.07
CV	N.A.	10.2	1.1

It is clear, observing Table 2, that there is high difference between the three measured values. The SFTT results present the lower value of failure strain this can be attributed to the high variability in strength along a single fibre and the nature of the test itself. For the 100% rCF specimen, the failure value is underestimated as the stress concentration generated in the end-tab region causes premature failure. The Interlaminated Hybrid specimens are the more representative of the fibre behaviour when those are embedded matrix to form a composite material. However, the definition of the failure strain for the Interlaminated Hybrid composite is, at the moment, arbitrary, as it depends on the identification of the fragmentation plateau slope. By changing the absolute thickness of the continuous outer layers and its mechanical properties, it will be possible to design the specimens to fall within the “carbon failure and catastrophic delamination” region. This would allow changing the shape of the stress-strain curve and unequivocally defining the carbon failure strain, as shown in Figure 6.

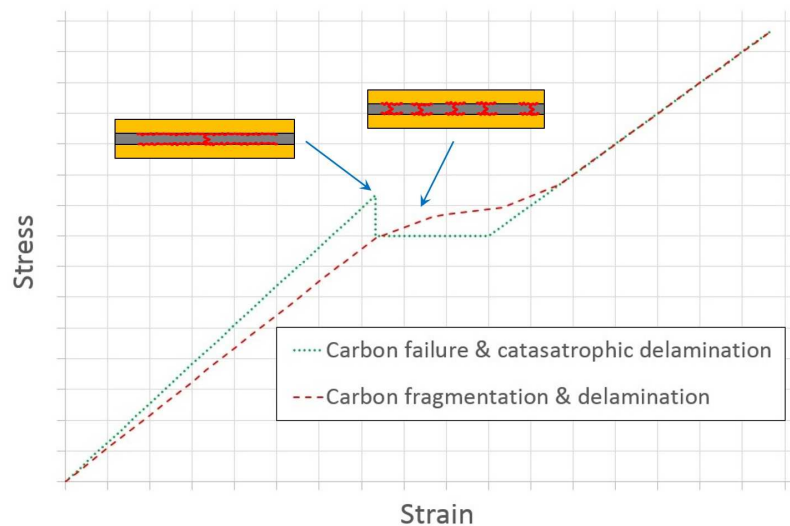


Figure 6: Shape of the stress-strain curves for different damage mechanism.

4. Conclusions and Future Works

Interlaminated hybrid specimens can be used to determine the failure strain of rCF avoiding the premature failure problems that affect the other testing methodologies. Changing the continuous fibre materials will change the shape of the Damage Mode Map and might allow obtaining a single fibre failure in the carbon, giving a clearly identifiable value.

For the, substantially indirect, measurement of the stiffness related properties, i.e. Young’s modulus and strength, it is necessary not only to take into account the fibre volume fraction but also the level of fibre alignment, as explained in Equation (1) below:

$$E_{Short\ fibre\ composite} = \eta v_{fibre} E_{fibre} + (1 - v_{fibre}) E_{matrix} \quad (1)$$

where E is the stiffness, v the volume fraction and η a correction factor that takes into account the discontinuity and the misalignment of the fibres.

To measure the fibre volume fractions various methods have been taken into account, i.e. resin burn off [17], acid digestion [18] and cross-section image analysis [19], but none of them has been deemed suitable to the high turnover required by quality control procedures. A methodology to directly relate the HiPerDiF machine manufacturing parameters with the areal weight of the preform and fibre alignment level and subsequently this information with the areal weight and the specimen curing parameters is underway; this will allow accurately estimating η and v_{fibre} and, solving Equation (1), obtaining the fibre stiffness and strength.

Acknowledgments

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