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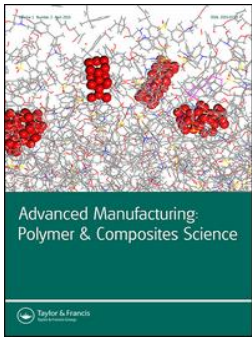
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# Development and application of a quality control and property assurance methodology for reclaimed carbon fibers based on the HiPerDiF (High Performance Discontinuous Fibre) method and interlaminated hybrid specimens

M. L. Longana<sup>a</sup> , H. Yu<sup>a,b</sup>, I. Hamerton<sup>a</sup> and K. D. Potter<sup>a</sup>

<sup>a</sup>Bristol Composites Institute (ACCIS, Advanced Composites Collaboration for Innovation and Science), University of Bristol, Bristol, UK;  
<sup>b</sup>Department of Mechanical Engineering, University of Bath, Bath, UK

## ABSTRACT

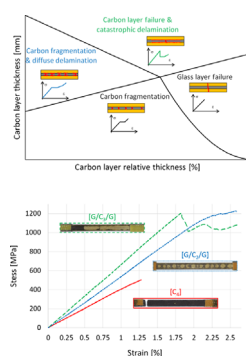
To promote the usage of recycled composite material, it is of paramount importance to develop quality control and property assurance methodologies compatible with the format of reclaimed fibers. In this paper, the concept of using interlaminated hybrid specimens, whose tensile response has been tailored with the aid of the Damage mode maps, is exploited to unambiguously identify the reclaimed fibers failure strain. The interlaminated hybrid specimens are manufacturing by sandwiching a layer of aligned discontinuous reclaimed carbon fibers produced with the HiPerDiF (High Performance Discontinuous Fibre) method between continuous glass fibers. The reliability of the obtained results is compared with results obtained with single fiber tensile tests. The developed methodology is then applied to the investigation of the strength retention of carbon fibers reclaimed through a solvolysis process and to the effects of the fiber length on the HiPerDiF alignment process.

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control



## 1. Introduction

The recycling of the composite material, and in particular of carbon fiber-reinforced thermosetting plastics, through the phase-separation route is a multi-step process: the collection and sorting of the waste, the reclamation of the fibers, and the remanufacturing to produce new intermediate materials.<sup>1</sup> Even if the development of solutions and technologies for fiber and matrix identification and grading is an interesting scientific problem that would further improve the effectiveness of reclamation processes and the reclaimed carbon fibers (rCF) properties, the collection and sorting of the waste can be essentially considered as an industrial issue.<sup>2</sup> Reclamation processes are currently attracting most of

the scientific interest in the composite recycling field.<sup>1,3</sup> Reclamation techniques can be categorized in two intersecting groups: the thermal processes, e.g. pyrolysis,<sup>4</sup> fluidized bed pyrolysis<sup>5</sup> and microwave-assisted pyrolysis,<sup>6</sup> and the chemical processes, e.g. solvolysis<sup>7</sup> and super-critical fluid solvolysis.<sup>8</sup> Independently from the reclamation technique, the rCFs are in a filamentized, randomly oriented, and low-density packing form.<sup>1</sup> The length of the rCFs is usually reduced by waste size reduction for transport and reclamation, by fiber breakage during and comminution after reclamation; moreover, the architecture and organization of the fiber are lost. The reclamation processes alter the mechanical performance of the fibers: in general, the stiffness is preserved

and the failure properties, i.e. strength and failure strain, are reduced. Finally, the fiber physical properties are affected: the sizing material is usually removed by the fiber surface,<sup>9</sup> the fiber surface topology, e.g. surface striation, roughness, and micro porosity, is altered by char formation or residues, and the surface activation energy and, more generally, the fiber–matrix adhesion properties are compromised.<sup>3</sup> These reclamation-induced alterations to fiber geometry, architecture, mechanical, and physical properties raise the need for specific remanufacturing processes and characterization procedures. In terms of remanufacturing, it is possible to use the rCFs as reinforcement in direct molding techniques, e.g. injection molding, bulk compound molding and extrusion molding, or to create non-woven or chop strand mats for injection molding<sup>1,3</sup>; however, this generates low-performance materials and parts that could be manufactured more easily and affordably with virgin glass fibers. The use of fiber alignment techniques, such as 3-DEP process,<sup>10</sup> centrifugal alignment<sup>11</sup>, and HiPerDiF (High Performance Discontinuous Fibre) technology,<sup>12</sup> allow achieving high fiber volume fractions and consequently good mechanical performance and economic value. The changes in fiber mechanical and physical properties pose a major challenge in terms of quality control and properties assurance. It was observed that residues on rCFs' surface preserve the mechanical properties but reduces the fiber–matrix adhesion,<sup>1</sup> therefore it is necessary not only to characterize the properties of the rCFs, i.e. stiffness and strength, but also their behavior when embedded in the matrix to generate composite materials. Therefore, to promote the industrial application of recycled composite materials, it is necessary to develop new quality control and characterization techniques able to provide reliable mechanical properties in a production-compatible timescale. Furthermore, the need for ad hoc characterization and quality control techniques for recycled composite materials is further justified by the fact that the waste, and consequently the rCFs, can come from multiple, and potentially unknown or scarcely documented, sources. This means that recycled materials will contain a blend of different fibers with a potentially wide distribution of size, mechanical, and physical properties.

This paper studies how the High Performance Discontinuous Fibre (HiPerDiF) technology, a water-based fiber alignment technique invented at the University of Bristol,<sup>12</sup> can be used to produce interlaminated hybrid specimens that allow users to quickly and reliably define the failure strain of rCFs imbedded in a composite material for quality control and properties assurance purposes. The HiPerDiF discontinuous fiber alignment method exploits the sudden momentum change of a jet of fibers suspended in water directed in a narrow gap between parallel plates.<sup>13</sup> Further details about the working principle and the mechanical properties achieved can be found in Refs. 13,14. In particular, it

has been demonstrated that, if the fibers are longer than the critical length, quantifiable in the order of 0.5 mm in the case of carbon/epoxy, the obtained aligned discontinuous fiber-reinforced composite (ADFRC) has mechanical properties compared with those made of continuous fibers.<sup>13</sup> The HiPerDiF method has also been used to remanufacture rCFs to obtain high mechanical properties in intermingled hybrids with virgin fibers and interlaminated hybrids with continuous glass fibers<sup>15</sup> and to demonstrate the possibility to maintain high performance after multiple closed loop recycling processes.<sup>16</sup>

This quality control and properties assurance methodology is then used to study the effects of using blends of different fiber lengths on the quality of the material produced with the HiPerDiF method.

## 2. Materials

To avoid any bias, the fibers used for the quality control methodology development, reclaimed through pyrolysis from an expired prepreg tape, were sourced in tow form without any additional information in terms of provenance, reclamation processes, and mechanical properties. The fibers were manually cut at 25 mm for the Single Fiber Tensile Tests and automatically chopped at  $3(\pm 1)$  mm to manufacture the Aligned Discontinuous Fiber and Interlaminated Hybrid tensile test specimens.

As the length distribution of rCFs is hardly measurable and controllable, the fibers used to study the effects of using blends of different fiber lengths on the quality of the material produced with the HiPerDiF method are chopped high tensile strength virgin carbon fiber (C124, TohoTENAX).

To manufacture the interlaminated hybrid specimens, the aligned carbon fiber preforms were coupled with S-glass epoxy prepreg (SG913, Hexcel), identified below as [G] in the lamination sequence, and characterized in Ref. 14. The mechanical properties of the used materials are summarized in Table 1.

## 3. Quality control methodology development

### 3.1. Methodology

The quality control and property assurance methodology developed in this paper is based on the use of interlaminated hybrid specimens to identify the failure strain of rCFs. Czel et al.<sup>17</sup> demonstrated that the use of glass/carbon interlaminated hybrid specimens for

**Table 1.** Material properties.

Properties		C124 carbon fiber	S-glass prepreg
Diameter	[ $\mu$ m]	7	–
Length	[mm]	3 & 6	–
Modulus	[GPa]	225	~58
Strength	[MPa]	4350	~1900
Failure strain	[%]	1.8	3.7
Ply thickness	[mm]	~0.07	~0.13

the characterization of carbon fibers allows the stress concentrations in the end-tab and gripping region to be suppressed, protecting the central carbon layer from premature failure.

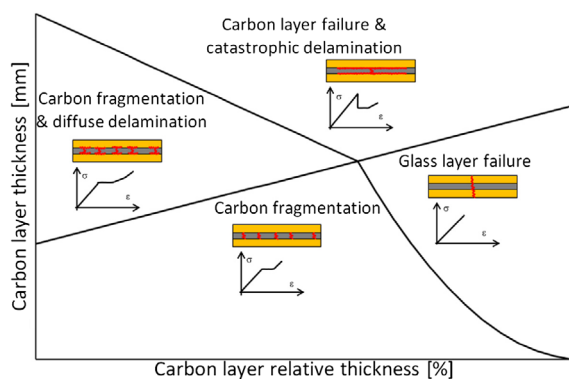
Jalalvand et al. in Ref. 18 introduced the concept of controlling the failure behavior of thin-ply interlaminated hybrid composites by changing the absolute thickness and thickness ratio of the constituent materials. Jalalvand et al. 19,20 visualized this dependence through the development of the Damage Mode Maps (DMM). Figure 1, adapted from Ref. 19, show how the different combinations of total carbon proportion and absolute carbon thickness generates four different failure modes, i.e. “carbon fragmentation”, “carbon fragmentation & diffuse delamination”, “carbon fragmentation & catastrophic delamination” and “catastrophic glass failure”.

Using interlaminated hybrid specimens not only makes it possible to avoid premature specimen failure but also to design the specimen to achieve the “carbon fragmentation & catastrophic delamination” failure mode. This allows to unambiguously identify the rCFs’ failure strain on the stress strain curves, as suggested in Ref. 21. To draw the DMM, it is necessary to know the failure strain and the stiffness of the intermingled hybrid laminate constituent materials. This basic information can be obtained through a limited number of single fiber tensile tests or aligned discontinuous fiber specimen tensile tests, as detailed below.

### 3.2. Baseline results

#### 3.2.1. Single fibre tensile test

Individual fibers were extracted from the reclaimed fiber tows, cut to length, and used as received. Each fiber was stretched taut between end-tabs to achieve a gauge length of 12 mm. Photocurable resin was then applied to the end-tabs and left to cure under a UV light source for 1 h. The samples were tested with a Dia-Stron LEX810 High Resolution Tensile Tester at a rate of 0.6 mm/min (strain rate  $0.05 \text{ s}^{-1}$ ) until failure, in accordance with ASTM D3822/D3822M-14.22 Load and extension (strain) were recorded excluding any samples that failed prematurely



**Figure 1.** Damage mode map and failure mechanisms schematics.

at the end-tabs. A total of 50 samples were successfully tested. An average fiber cross section has been obtained from optical microscope measurements. The obtained results are summarized in Table 2.

#### 3.2.2. Aligned discontinuous fiber specimens tensile test

The most immediate application of the HiPerDiF technology in terms of testing method is based on the conventional tensile test standard for composites<sup>23</sup> performed with aligned discontinuous fiber-reinforced composite specimens, as in Refs. 15,16. The HiPerDiF method was used to manufacture aligned discontinuous rCF preforms that were subsequently impregnated with MTM49-3 epoxy resin. Four layers,  $[C_4]$ , of the obtained prepreg tapes were placed in a semi-closed mold and consolidated at  $135 \text{ }^\circ\text{C}$  and 6 bar for 135 min. Considering the HiPerDiF manufacturing parameters and the areal densities of the aligned discontinuous rCF preforms and of the resin film, the fiber volume fraction of the obtained ADFRC material is estimated to be 30%. After curing glass fiber-reinforced epoxy end-tabs were bonded with Huntsmann Araldite 2014-1. The nominal specimen geometry is shown in Figure 2.

A total of seven samples were manufactured and tested in a Shimadzu servo-electric tensile test machine at a cross-head speed of 1 mm/min. The stain was measured with an Imetrum video gauge over a gauge length of approximately 45 mm. The obtained results are summarized in Table 3.

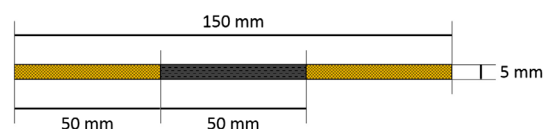
Taking into consideration the fiber misalignment and length factors,<sup>13</sup> the obtained stiffness is compatible with the estimated fiber volume fraction. A representative stress–strain curve for an aligned discontinuous fiber specimens tensile test, denoted as  $[C_4]$ , is shown below in Figure 4.

#### 3.3. Interlaminated hybrid specimens tensile test

To manufacture the interlaminated hybrid specimens, dry aligned carbon fiber preforms were placed between layers of continuous unidirectional S-glass prepreg with the lamination sequence defined above and cured in a semi-closed mold at  $125 \text{ }^\circ\text{C}$  and 6 bar for 150 min. The

**Table 2.** Single fiber tensile test results.

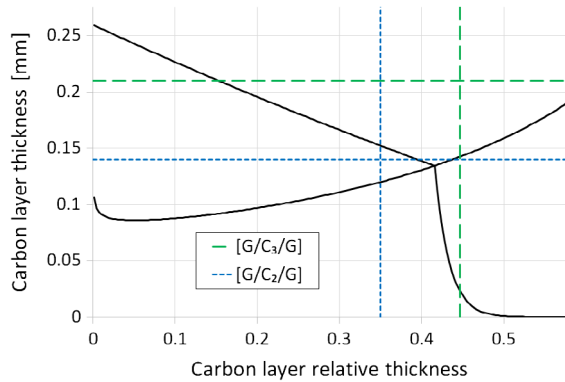
Properties		Value	CV [%]
Stiffness	[GPa]	204.7	10.7
Strength	[MPa]	4078	20.8
Failure strain	[%]	2.003	18.8



**Figure 2.** Tensile test specimen.

**Table 3.** Aligned discontinuous fiber specimens tensile test results.

Properties		Value	CV [%]
Stiffness	[GPa]	41.5	12.1
Strength	[MPa]	565	19.6
Failure strain	[%]	1.38	9.3

**Figure 3.** Damage mode map for different carbon fiber failure strain.

resin excess from the S-glass prepreg impregnates the central layers of dry discontinuous fibers. After curing, GFRP end-tabs were bonded with Huntsmann Araldite 2014-1. The nominal specimen geometry is the same as the one for aligned discontinuous fiber specimens shown in Figure 2. A total of 20 samples were manufactured and tested with the same equipment and settings used for the aligned discontinuous fiber specimen tensile tests.

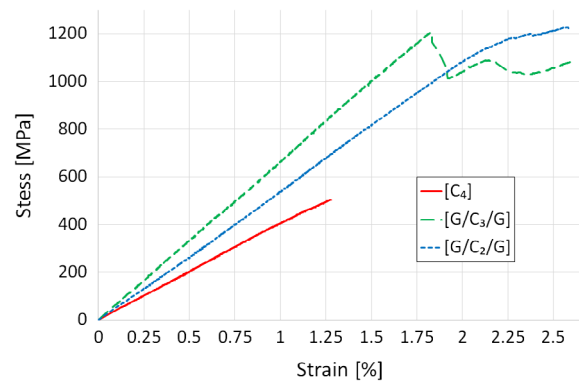
The DMM shown in Figure 3 is drawn accordingly to the mechanical properties of Tables 1 and 3. The single fiber tensile test allowed to estimate a failure strain between 1.6 and 2.4%; the first value, that results in more restrictive lay-up conditions, is chosen to draw the DMM. Two layups,  $[G/C_2/G]$  and  $[G/C_3/G]$ , have been taken into consideration, as identified on the DMM of Figure 3.

The  $[G/C_2/G]$  layup falls in the “Carbon Fragmentation & Diffused Delamination” failure mode area of the DMM while the  $[G/C_3/G]$  layup in the “Carbon Failure & Catastrophic Delamination”, this is reflected in the stress–strain curve shape, as shown below in Figure 4.

### 3.4. Results and discussion

Representative stress–strain curves of the aligned discontinuous fiber,  $[C_4]$ , and interlaminated specimen,  $[G/C_2/G]$  and  $[G/C_3/G]$ , tensile tests are shown in Figure 4.

Observing Figure 4, it appears evident that, contrarily to what one would expect, the  $[C_4]$  stiffness is lower than the interlaminated specimens, this is caused by the different manufacturing procedures: impregnating the aligned fibers preforms with the resin excess from the continuous glass fiber prepreg allows a higher fiber volume fraction, and therefore stiffness, to be obtained. The

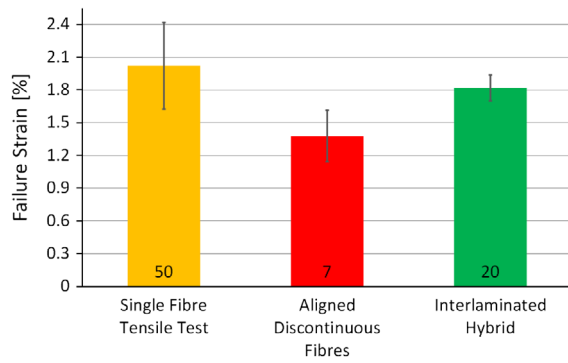
**Figure 4.** Stress–strain curves for aligned discontinuous fiber and interlaminated specimen tensile tests.**Figure 5.** In-plane view of aligned discontinuous fiber and interlaminated specimens.

difference in fiber volume fraction of the carbon layer and the different ratio between carbon and glass in  $[G/C_2/G]$  and  $[G/C_3/G]$  explains the difference in stiffness between these two sets of results. It is straightforward to notice that the failure strain of the aligned discontinuous fiber specimens is lower than the load drop that correspond to the carbon failure in the  $[G/C_3/G]$  and the knee point of the  $[G/C_2/G]$  specimens, i.e. the beginning of the rCFs’ fragmentation. This is believed to be due to the stress concentration in the end-tab region that causes premature failure, as also supported by Figure 5.

The benefits of designing specimens that fail with the “Carbon Failure & Catastrophic Delamination” appear clear when comparing the stress strain curves for the  $[G/C_3/G]$  and the  $[G/C_2/G]$  of Figure 4: the latter lay-up does not allow to unequivocally identify the inner carbon layer failure as no univocally definable feature appears on the stress–strain curve. Moreover, the manifestation of the carbon fragmentation, i.e. the gradual slope change in Figure 4, is delayed by the so called “hybrid effect”.<sup>24</sup> The sharp load drop on the  $[G/C_3/G]$  stress–strain curve is a clear feature that allows to identify the failure of the inner carbon layer.

The failure strain obtained with the three considered methods are summarized in Figure 6, along with the standard deviation and the number of tested specimens for each set.

At first, it is important to underline that the standard deviation values are affected by the number of tested specimen for each type of test or specimen. However, it is possible to observe that, for the reasons explained above, the aligned discontinuous fiber test underestimates the



**Figure 6.** rCFs' failure strain measured with the different methodologies.

fiber failure strain. It is important to underline that a direct comparison of the single fiber tensile test, whose results are dependent the fiber gauge length and affected by high scattering, and the interlaminated hybrid specimen test, influenced by the matrix and fiber–matrix interfacial properties, is not possible. However, taken into consideration the different nature of the two tests, the obtained results are consistent with each other.

The interlaminated hybrid specimen test is, in general, faster and less labor-intensive than the single fiber tensile test and gives more consistent results, i.e. with a lower standard deviation. Moreover, it is more representative of the rCFs' behavior when imbedded in the composite. Finally, the rCFs' layer is “protected” by the continuous glass layer and its failure is not affected by end-tab stress concentration.

#### 4. Methodology application

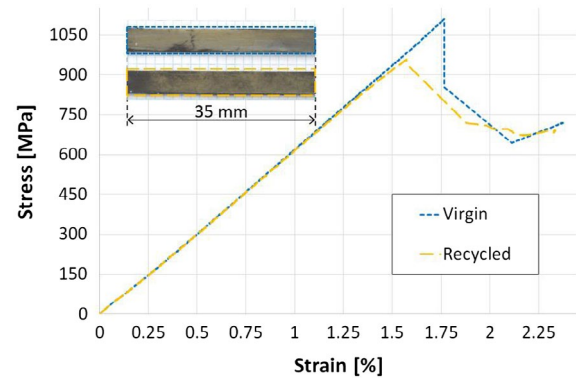
The methodology developed above have been validated by applying it to two different scenarios, i.e. the evaluation of a reclamation process on the carbon fiber strain and the possibility to process fibers of different lengths with the HiPerDiF method.<sup>25</sup>

##### 4.1. Effect of a solvolytic reclamation process on carbon fibers

For the methodology development the amount of available fibers was enough to perform single fiber tensile tests to measure fiber stiffness and failure strain to design the specimen. As such amount of fiber might not be always available or the fiber length might not allow to obtain this information, it is necessary to prove that the methodology can be applied “blind”. Two sets of 3-mm fibers, virgin and after a solvolytic reclamation process,<sup>26</sup> received without any further information, have been tested.

##### 4.1.1. Specimen design and manufacturing

In this case the lamination sequence [G/C<sub>4</sub>/G] has been chosen following a conservative approach, it was believed that this lay-up would allow to achieve the



**Figure 7.** Stress–strain curves from tensile tests on interlaminated specimen manufactured with virgin and recycled carbon fibers.

“Carbon Failure & Catastrophic Delamination” failure mode.

The specimens were manufactured as described above in Section 3.3 to ensure the full fiber impregnation two film of the same resin used in the S-glass prepreg has been used to impregnate the two central carbon fiber layers.

##### 4.1.2. Results and discussion

With the available amount of fiber, it was possible to produce two batches of specimens, one for the virgin and one for the reclaimed fibers, of four specimens each that were tested as described in Section 3.2.

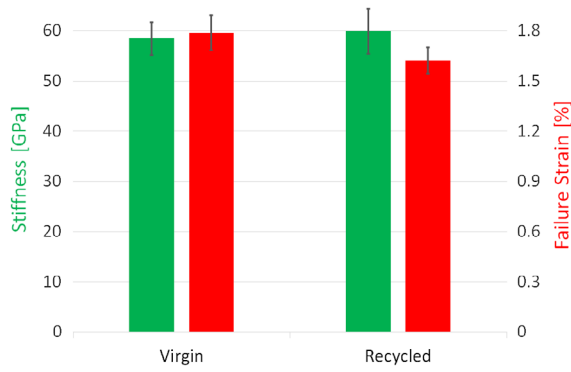
Stress–strain curves representative of the virgin and reclaimed fiber specimens' behavior are shown in Figure 7 along with an in-plane view of the failed specimens.

From Figure 7, it can be observed that the chosen lay-up allowed to obtain the desired failure mode, it is therefore possible to unequivocally identify the failure strain of the virgin and recycled carbon fibers. The specimen stiffness and failure strain of the carbon fibers, i.e. the strain correspondent to the load drop in the stress–strain curves of Figure 7, obtained from the tensile test are presented in Figure 8.

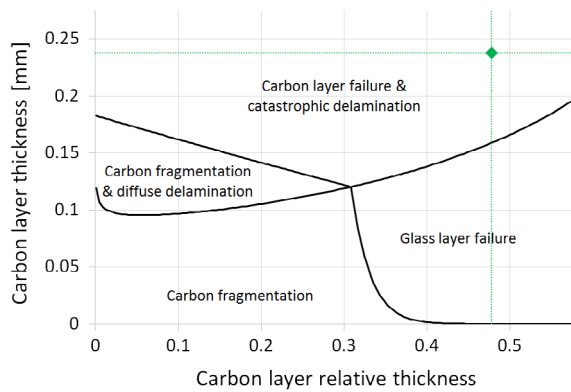
From Figures 7 and 8, it appears evident the stiffness of the specimen, and therefore of the fiber, is not affected by the reclamation process, this is in accordance with the literature about carbon fiber reclamation with pyrolytic processes.<sup>1,3</sup> As much as regards the fiber failure strain, this is reduced by 10% by the reclamation process.

##### 4.2. Effect of fiber length on the HiPerDiF process

All the research work on the use of the HiPerDiF method to remanufacture rCFs mentioned above was conducted using a single fiber length: this is not representative of the real rCF feedstock form that is constituted of discontinuous fibers with a variable length distribution. The quality control methodology developed above is used to investigate the possibility to process rCFs of different length: five batches of intermingled length hybrids specimens were manufactured with different 3- and 6-mm



**Figure 8.** Specimen stiffness and carbon layer failure strain from tensile tests on interlaminated specimen manufactured with virgin and recycled carbon fibers.



**Figure 9.** DMM for intermingled length hybrids.

fiber (properties in Table 1) length ratios (100% 3 mm, 75% 3 mm–25% 6 mm, 50% 3 mm–50% 6 mm, 25% 3 mm–75% 6 mm, and 100% 6 mm) are manufactured and tested.

#### 4.2.1. Specimen design and manufacturing

Using the information of Table 1, it is possible to draw a new DMM, shown in Figure 9, and to define the thickness ratio between the rCF and the continuous S-glass ratio.

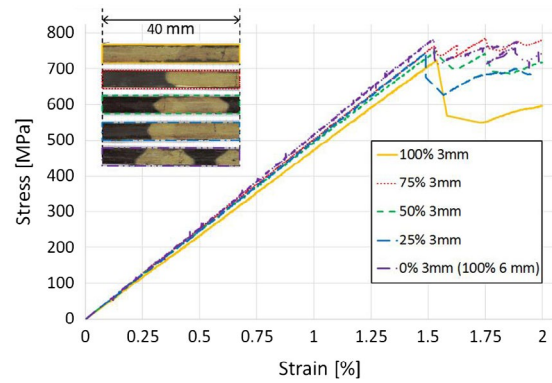
The desired failure mode, i.e. “Carbon Failure & Catastrophic Delamination”, can be achieved with the  $[G/C_4/G]$  lamination sequence.

The specimens were manufactured as described above in Section 3.3 to ensure the full fiber impregnation two films of the same resin used in the S-glass prepreg has been used to impregnate the two central carbon fiber layers.

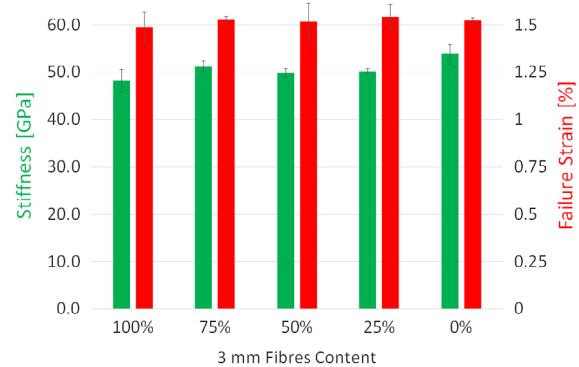
#### 4.2.2. Results and discussion

The specimens were tested as described in Section 3.2.

Stress–strain curves representative of the specimens’ behavior with different fiber length ratios are shown in Figure 10 along with an in-plane view of the failed specimens.



**Figure 10.** Stress–strain curves from tensile tests on interlaminated specimen manufactured with different carbon fiber lengths.



**Figure 11.** Specimen stiffness and carbon layer failure strain from tensile tests on interlaminated specimen manufactured with different carbon fiber lengths.

From Figure 10, it can be observed that the specimens failed as predicted with the DMM of Figure 9. It is therefore possible to unequivocally identify the failure strain of the aligned discontinuous carbon fibers layer. The specimen stiffness and failure strain of the carbon fibers, i.e. the strain correspondent to the load drop in the stress–strain curves of Figure 10, are presented in Figure 11.

From Figure 11 it can be observed that the different sets of specimens present the same stiffness and failure strain. Both stiffness and strength are dependent of the fiber alignment level; it is therefore possible to conclude that processing fibers of different length do not affect their level of alignment. In this case, the interlaminated hybrid specimen tensile test allows to test not just the tensile properties of the fibers, that are known, but the performance of the recycled composite overall.

## 5. Conclusions

The use of interlaminated hybrid specimens made of a layer of aligned discontinuous carbon fibers manufactured with the HiPerDiF method have been developed as a quality control and property assurance methodology



for rCF and of the remanufactured material. Damage mode maps allow the failure modes of the interlaminated hybrid specimen to be tailored; by achieving the “Carbon Failure & Catastrophic Delamination” failure mode it is possible to clearly identify the failure strain of the inner layer. The developed methodology has been applied to two case studies achieving satisfactory results. In the case of the carbon fibers reclaimed with a solvolytic process, it has been possible to demonstrate that, in accordance with<sup>26</sup> such process do not affect the fibers stiffness and only marginally affects the fiber and remanufactured material strength. In the case of the effect of the fiber length on the HiPerDiF process, it has been possible to demonstrate that the ADFRC materials manufactured with different ratios of 3- and 6-mm fibers do not present significant differences.<sup>25</sup>

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All data required for reproducibility are provided within the paper.

### Disclosure statement

No potential conflict of interest was reported by the authors.

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### ORCID

M. L. Longana  <http://orcid.org/0000-0002-9111-6038>

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