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# A novel powder-epoxy towpregging line for wind and tidal turbine blades

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Keywords: Powder-epoxy, towpregging, joule heating, carbon fibres, basalt fibres, hygrothermal ageing

Highlights:

- The powder-epoxy towpregging process was described in detail for both carbon fibres and basalt fibres.

- Excellent static mechanical properties were reported for unidirectional carbon-fibre/powder-epoxy composites and the influence of fibre volume fraction (FVF) was analysed.

- Two types of basalt fibre were tested, showing comparable mechanical properties to glass fibre composites.

- A hygrothermal study of both carbon-fibre composites and basalt-fibre composites showed that the latter had a greater sensitivity to water uptake and immersed ageing.

Abstract

A novel material and process was developed using fibre-reinforced powder-epoxy to produce unidirectional towpreg with a pilot-scale towpregging line, for cost-effective production of large composite structures for the renewable energy market, specifically for wind and tidal turbine blades. Electrostatic attraction was used to coat fibre tows with powder epoxy and either joule or radiant heating employed to heat and melt the polymer, followed by consolidation between rollers. Unidirectional carbon-fibre and basalt-fibre reinforced polymer laminates (UD-CFRP and UD-BFRP, respectively) were manufactured from the towpreg. Tensile test results showed that the towpregging process could be employed to achieve high performance UD-CFRP with 0° tensile properties that are similar or better than commercially-available UD-CFRP systems. The competitive advantages of the powder-epoxy towpreg system include lower cost, better overall manufacturing control for vacuum-bag-only manufacturing and the ability to co-cure parts together at a later stage. Mechanical test results showed some variation between two types of UD-BFRP, but the results compared well with published data on UD-BFRP and equivalent glass-fibre reinforced polymer (GFRP) systems. Finally, the influence of hygrothermal ageing due to water immersion on the tensile properties of the materials was investigated, with tests revealing that the water ageing effect was more severe in the case of UD-BFRP than for UD-CFRP.

## 1 Introduction

The International Energy Agency has reported an increasing rate of electricity generation worldwide of 8.28% per year over the last 50 years [1], which has been accelerating in the last two decades to sustain emerging economies. Despite a rapid expansion of renewable energy sources in the past 20 years [2], the overall contribution of renewable energy is still modest (1.8% for wind and solar, 2.5% for hydro) in comparison to global energy consumption. In this regard, optimisation and diversification of renewable energy sources and technologies have become paramount. Ocean energy potential, although very substantial at an estimated 8,000 TWh/year [3], is still largely under-exploited.

To exploit the full potential of offshore energy, in particular, several significant challenges must be addressed for wind and tidal turbine composite structures. These structures operate in severe environments undergoing high fatigue loading, water ingress, erosion, immersed ageing, bio-fouling, etc. [4–8]. Accordingly, the composite structures must have high mechanical properties, which cannot degrade significantly over the lifespan of the device (approx. 20 – 25 years). GFRP has been used predominantly in the manufacture of wind and tidal turbine blades due to its good mechanical performance, relatively low cost, and formability for complex geometries. As these blades get larger, however, the demand on mechanical performance increases, along with the need to lower the levelised cost of energy (LCOE).

For several years, basalt fibres have been investigated as a potential alternative to glass fibres; in particular, for E-glass fibres, which are widely used by the composites industry [9,10]. Basalt fibres are reported to have similar mechanical properties to S-glass fibres, however, the basalt fibre production requires less energy and less additives than glass fibre production [11], meaning that it costs less than S-glass [10]. In general, it has been shown in the literature that BFRP has mechanical properties which are comparable to, if not higher than, GFRP [11,12]; albeit, with considerable variation in reported properties. Some of this variation may be linked to basalt being from a natural source with a chemical composition that depends on location, as well as the typical variations between suppliers ascribed to their production methods [10]. In this regard, BFRP has not had the same level of evaluation as GFRP and CFRP to build a comprehensive knowledge of its mechanical behaviour. In the context of this paper, there are few studies which have documented the immersed performance of BFRP for marine applications [13–15]. Nevertheless, basalt fibre technology continues to advance, and the aforementioned studies reported sufficiently promising results to warrant further investigation as a potential alternative to GFRP for wind and tidal turbine blade manufacture.

Where higher performance materials are required, CFRP has also been investigated as an alternative to GFRP due to its high specific strength and stiffness. To maintain a low LCOE, however, careful consideration of blade design is required to optimise the cost effectiveness of introducing CFRP [16]. Many wind turbine blade manufacturers have implemented hybrid composite designs to offset the associated costs [4,6,17–21] (e.g. a CFRP spar with a GFRP root, shear web, and skins). As the main load bearing sub-structure along the span of the blade, the spar must withstand significant tensile and compressive stresses. Consequently, spars are very thick unidirectional laminates. Depending on the blade size and design, the spar may taper from 150 mm thickness down to less than 5 mm [6,22]. For wind turbines, this occurs over a larger span (e.g. 70 m) than for tidal turbine blades (e.g. 8.5 m). The spar caps for a 2 MW tidal turbine blade can taper from 104 mm thickness to 25 mm [23]. Manufacturing such laminates poses several processing challenges including maintaining fibre alignment and straightness [24], difficulty infusing with conventional methods (such as vacuum-assisted resin transfer moulding (VaRTM)) [6], and difficulty in controlling the exothermic curing reaction of the resin matrix [25]. Accordingly, several material suppliers have developed UD-CFRP vacuum-bag-only (VBO) prepregs specifically aimed at manufacturing spars and similar structures for

1 wind and tidal turbine blades; Gurit's SparPreg™, Hexcel's Hexply M9.6 series and M79 prepreg, and  
2 Cytec's (formerly ACG's) VTM 260 series [4]. The advantages of these VBO prepregs is that they greatly  
3 reduce the complexity of the infusion process as they are supplied in a partially or fully impregnated  
4 form. Their disadvantage is that they typically require temperature-controlled storage to prevent  
5 premature curing of the resin system (i.e. out-time effects [26]), and they generally do not address  
6 the issue of high exotherms, with the exception of Hexcel's Hexply M79 resin [27]. One alternative  
7 that allows blade manufacturers to avoid infusing dry fabric and controlling large exotherms, is the  
8 use of pultruded UD-CFRP [18,20,28], which has even been considered for offshore wind turbine tower  
9 structures [29]. Pultruded sections are fully infused (usually to a relatively high fibre volume fraction)  
10 and they are typically pre-cured, so they have no exotherm, they don't require temperature-  
11 controlled storage, and their high degree of fibre alignment is "locked in". The major disadvantage  
12 with these fully-cured pultruded sections is that they are difficult to form to complex geometries, such  
13 as the double curvatures in turbine blades, and they must be adhesively bonded together to form a  
14 single spar.

15 The technology described in this paper aims to address the aforementioned disadvantages of  
16 conventional VBO prepregs and pultruded sections by utilising the unique properties of a novel  
17 powder-epoxy matrix. In relation to fibre-reinforced composites, this powder-epoxy has been the  
18 subject of several studies in recent years which have investigated its processing behaviour [30–32], its  
19 mechanical behaviour [33–35], its behaviour in immersed conditions [36], and its application in wind  
20 [17] and tidal turbine blades [23,37]. Powder-epoxies, along with other thermosetting powders, are a  
21 commodity product from the coatings industry, which are widely available in bulk from a range of  
22 suppliers (e.g. FreiLacke, DSM, AkzoNobel, 3M, etc.). These powders tend to have excellent storage  
23 stability due to a high initial glass transition temperature (around 40°C) [38] and the frequent use of  
24 heat-activated latent curing agents in their formulation [39]. This means they can be used to make  
25 VBO prepregs which do not require temperature-controlled storage and generate significantly less  
26 exotherm than conventional epoxy systems [30,32]. Moreover, due to the processing stability of the  
27 powder-epoxy [30], it has been shown that sub-components (e.g. turbine blade skins) can be infused  
28 at 120°C, cooled to ambient conditions to form solid  $\beta$ -staged preforms (i.e. partially cured),  
29 assembled together, and then co-cured at 180°C [40]. The ability to store powder-epoxy preforms for  
30 use at a later stage is a strong competitive advantage of this technology. The ability to then co-cure  
31 these pre-formed parts is also very interesting, especially for the wind industry where blades are  
32 usually manufactured in sections and then adhesively bonded. The new process can thus avoid an  
33 extra processing step while maintaining the mechanical properties at the joining line, which can  
34 sometimes be design-critical.

35 The powder-epoxy is used in this study for the production of 'towpreg' a.k.a. pre-impregnated tows.  
36 In short, this production process consists of spreading the fibres of a tow, depositing powder between  
37 the fibres, heating the coated fibres, and then consolidating them. Spanning the last four decades,  
38 towpregging has been investigated by several authors for both thermoplastics and thermosets [41–  
39 45]. It has been shown that this production route has benefits over hot melt prepregging and solution  
40 prepregging in terms of cost and environmental impact; including little or no volatile organic  
41 compounds (VOCs) and little waste disposal due to powder reclamation and reuse [46]. Towpreg can  
42 be used for automated tape placement (ATP); either as a single towpreg (a.k.a. automated fibre  
43 placement (AFP)) or several consolidated towpreg (a.k.a. automated tape laying (ATL)) [44,47]. It can  
44 also be used in pultrusion [42,48] or filament winding [45]. This versatility lends itself to many  
45 industrial applications, however, to the authors knowledge there is little or no evidence to suggest it  
46 has been applied to the production of wind or tidal turbine blades or structures.

1 Initial work has been carried out on developing a pilot-scale powder towpregging for the  
2 aforementioned application [49,50]. In this study, a more mature version of the pilot powder  
3 towpregging line is described. Both carbon-fibre towpreg and basalt-fibre towpreg are investigated as  
4 alternatives to GFRP systems. Mechanical testing is carried out on UD-CFRP laminates made from the  
5 CF towpreg and compared with existing data to determine if the towpregging process has an adverse  
6 effect on the mechanical performance of the carbon fibres. The influence of fibre volume fraction, and  
7 its variability with respect to the towpregging line, is also studied for carbon-fibre towpreg. UD-BFRP  
8 laminates are made using basalt fibres from two different suppliers. These laminates are mechanically  
9 tested to study the variation between suppliers. Mechanical results for UD-BFRP are also compared  
10 with published data on equivalent material systems. Finally, a hygrothermal ageing study is carried  
11 out on UD-CFRP and UD-BFRP to assess the performance of both materials in immersed conditions.

12

## 13 2. Materials

14

### 15 2.1. Terminology

16 In the literature relating to material development and material production, the term ‘towpreg’ has  
17 been used commonly to describe tows that are impregnated with a thermoset powder or a  
18 thermoplastic powder [44,51,52]. For clarity, in this work, ‘tow’ is used to refer to the unimpregnated  
19 tow, and ‘towpreg’ is used for one or more tows that have been impregnated with powder and then  
20 sintered and consolidated, but which are still uncured. This terminology is chosen because ‘towpreg’  
21 represents a highly versatile precursor material which can later be developed for numerous  
22 manufacturing processes including pultrusion, ATP, filament winding, etc.

23

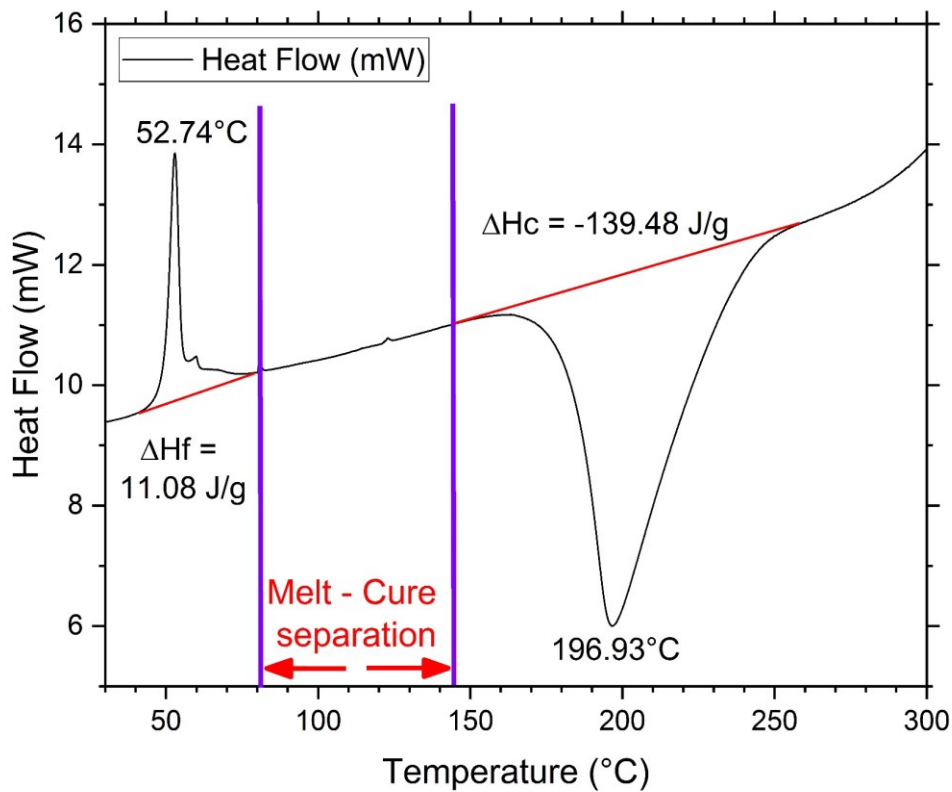
### 24 2.2. Powder-epoxy

25 All the composites in this study were manufactured using a novel powder-based epoxy matrix. The  
26 powder-epoxy (PE6405, density 1220 kg/m<sup>3</sup>) was engineered by Swiss CMT and then produced by  
27 FreiLacke. The processing characteristics of the powder-epoxy can be defined by two steps, which are  
28 relevant to both towpreg production and laminate manufacturing:

- 29 a) The powder-epoxy melts and sinters at around 40-60°C, reaching a minimum viscosity for  
30 infusion around 120°C.
- 31 b) The curing is then carried out through a heat activated catalytic process where the curing  
32 agent requires a temperature of at least 145°C for reaction initiation, as described by Maguire  
33 et al. [30] and Mamalis et al. [33] and shown by differential scanning calorimetry (DSC) in  
34 Figure 2.

35 The details of the chemistry involved are proprietary to the manufacturers of these commercially  
36 available products. The powder-epoxy has numerous advantages compared to standard VBO prepreg  
37 systems when building large, thick -section structures such as the latest wind turbine blades (over 100  
38 mm thickness at the blade root). Firstly, the low melt viscosity (minimum of 1.26 Pa.s [30]) and low  
39 rate of cure below 120°C allows more time to fully infuse the section [30–32]. Secondly, as shown in  
40 Figure 1, the powder-epoxy, PE6405 displays a curing exotherm of approx. 140 J/g, which is less than  
41 a third of the enthalpy of standard liquid resin epoxy systems (about 500 J/g) [30]. This feature is  
42 crucial in reducing the risk of thermal runaway for thick section composites manufacturing which  
43 allows for quicker production rates [31,32].

1 Figure 2 also highlights the powder-epoxy's ability to separate the melting/sintering phase (ending at  
 2 80°C) from the curing phase (starting at 145°C). Since the PE6405 melting/sintering temperature is  
 3 considerably lower than the curing temperatures, composite parts can be impregnated in one stage,  
 4 and then cured in another. For this reason, a two-stage heating profile is typically used as presented  
 5 in Figure 2. As previously mentioned, this feature gives manufacturers the ability to reform and co-  
 6 cure complicated parts as necessary. This strongly increases the cohesion of the different parts and  
 7 removes the need for an extra adhesion step, thus allowing 'one-shot' manufacturing, as shown in  
 8 Figure 3 [40,53,54].

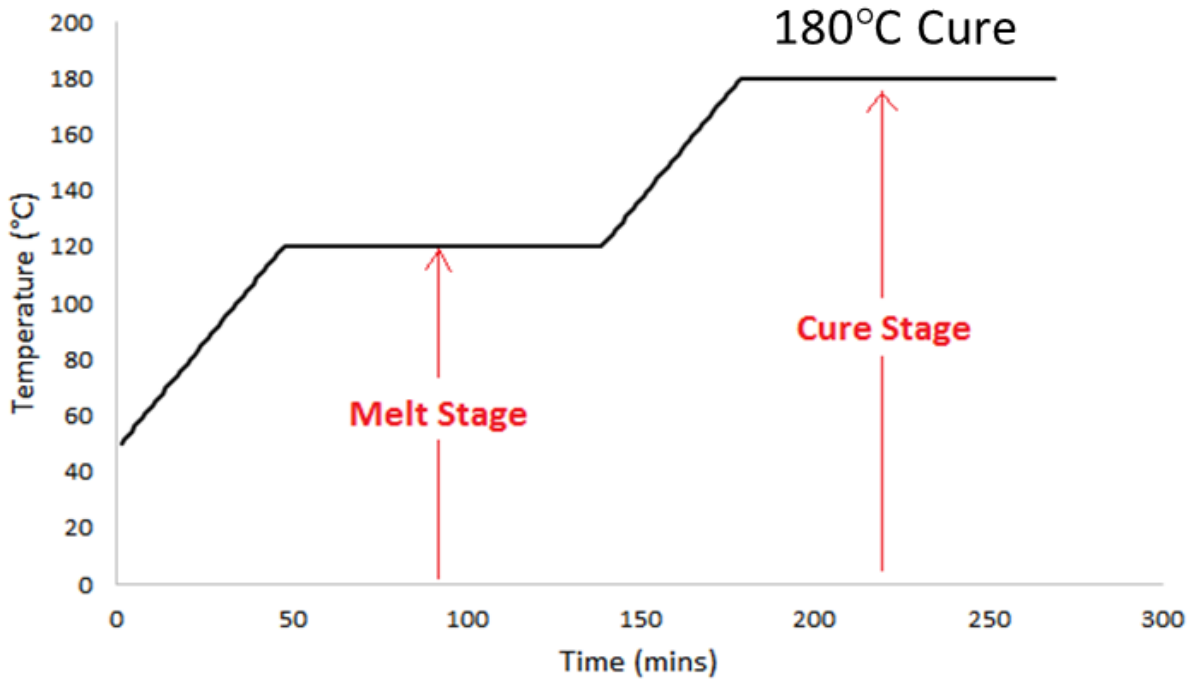


9  
 10 **Figure 1: DSC data showing a temperature sweep (20°C/min) of neat powder-epoxy sample,**  
 11 **exhibiting peaks due to a reversal of enthalpic relaxation (at 52.74°C) and curing (at 196.93°C). The**  
 12 **temperature gap between the end of the melting/sintering and the beginning of the cure is**  
 13 **represented by the purple lines. Note: exotherm is downwards.**

14  
 15 In recent times, 'one-shot' manufacturing processes have been targeted by both the wind and tidal  
 16 energy industries due to the added cost and weight of adhesives, as well as the potential for adhesive  
 17 bondlines to act as 'weak spots' in the structure [5,55]. These reported technologies all utilise some  
 18 form of VaRTM process to manufacture their 'one-shot' structures, but as Harper et al. [5]  
 19 acknowledge, it is unclear whether such processes can be used to successfully thick sections in 'one-  
 20 shot' (due to exotherms and difficulty infusing), or whether thick-section parts must be prefabricated  
 21 and later introduced during infusion of the skins [55]. In contrast, the low-exotherm powder-epoxy  
 22 'one-shot' technology can be used with VBO prepreg materials, thus reducing the risk of poor infusion  
 23 or 'thermal runaway'.

1 One limitation to the powder-epoxy technology is that the heat-activated curing agent requires higher  
2 temperatures for curing (> 160°C) than what is typical for conventional processes like VaRTM.  
3 Nevertheless, integrally-heated ceramic tooling has been developed to achieve the required  
4 processing conditions, and was used to produce “one-shot” 12.6m wind turbine blades, shown in  
5 Figure 3, using glass-fibre/powder-epoxy VBO prepregs [40].

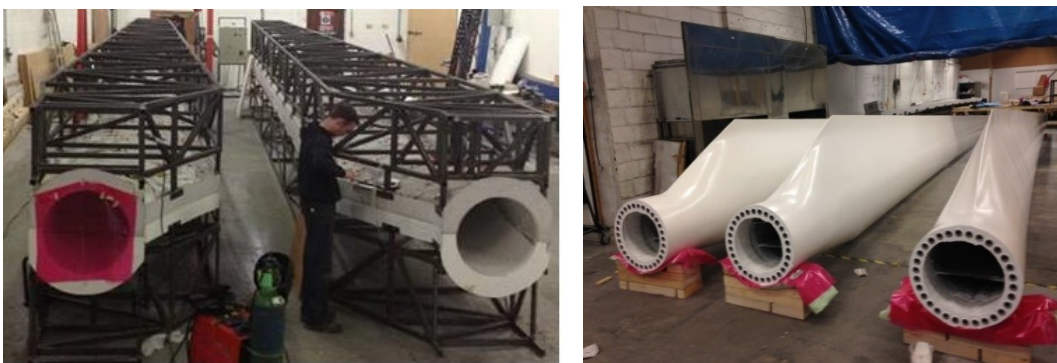
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7

8 *Figure 2: Typical melting and cure cycle for processing of powder-epoxy composites. Powder-epoxy*  
9 *composites can be melted and consolidated at 110-120°C without any significant curing, thus*  
10 *allowing individual components to be consolidated and assembled for a final co-cure at 180°C.*

11



12

13 *Figure 3: Ceramic heated tools (left); and 12.6 m blades made from powder-epoxy based VBO*  
14 *prepreg (right) [39]*

15

### 16 2.3. Carbon fibres

17 Mamalis et al. investigated the influence of sizing and fibre straightness on the mechanical properties  
18 of PE6405 powder-epoxy reinforced with Toray T700S-24K carbon fibres (CF) [33,35]. In their studies,

1 it was found that fibre straightness had a significant effect on mechanical properties, and the 50C sized  
 2 CF were found to be superior compared to two other fibre sizings. As such, T700S-24K-50C fibres  
 3 (equivalent to 1650 Tex) from Toray Industries Inc. [56] were chosen for this study; with individual  
 4 fibres having a diameter of 11  $\mu\text{m}$ .

#### 7 2.4. Basalt fibres

8 Variation in basalt fibre (BF) quality between different suppliers is common, as basalt is naturally  
 9 sourced from volcanic rocks, producing different chemical compositions when forming [11]. The  
 10 precise details of fibre manufacturing and sizing processes are often kept confidential, which leads to  
 11 greater uncertainty on the quality and reproducibility of the fibres. To address these concerns, the  
 12 properties of two different commercially available basalt fibres were investigated and compared in  
 13 this study; BF from Basaltex [57,58], and BF from Mafic [59]. Both Mafic BF and Basaltex BF had a fibre  
 14 diameter of 13  $\mu\text{m}$ , and were supplied in the form of 2400 Tex assembled rovings. The fibres were  
 15 both coated in alkoxysilane sizing, usually used in glass fibres for optimal cohesion with epoxide  
 16 systems [58,59]. A comprehensive summary of fibres and matrix mechanical properties are presented  
 17 in the table below.

18 **Table 1: Individual mechanical properties of fibres and powder epoxy matrix**

|  | $\sigma_M$ (MPa) | $E$ (GPa) | $\epsilon_f$ (MPa) | Diameter ( $\mu\text{m}$ ) | Density ( $\text{g}/\text{cm}^3$ ) |
|--|------------------|-----------|--------------------|----------------------------|------------------------------------|
| T700s carbon fibres [56]                 | 4900             | 230       | 2.1                | 7                          | 1.81                               |
| Mafic <sup>®</sup> direct roving [59]    | 3100             | 88-92     | 3.5                | 13                         | 2.63                               |
| Basaltex <sup>®</sup> direct roving [58] | 2900-3100        | 85-89     | 3.5                | 13                         | 2.67                               |
| PE6405 from Freilacke <sup>®</sup> [35]  | 73.1             | 3.0       | 2.43*              | N/A                        | 1.22                               |

20 \* linear elastic behaviour assumed

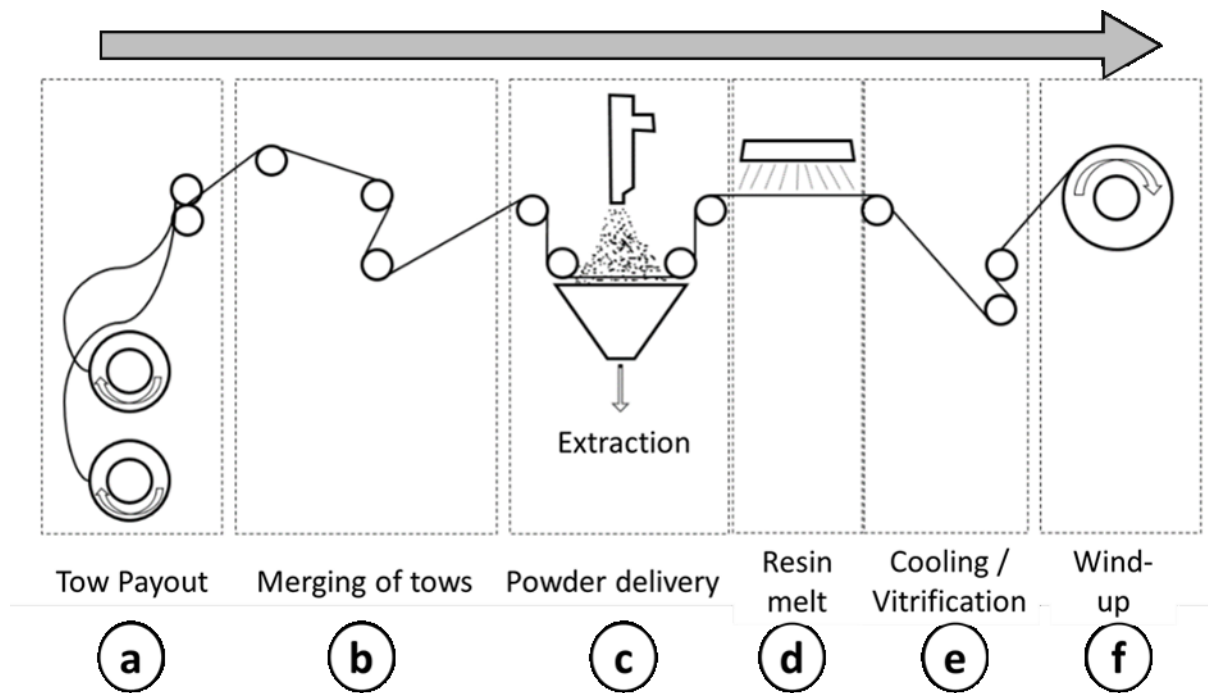
### 22 3. Towpregging process

#### 24 3.1. General description of the towpregging line

25 The pilot towpregging line was designed and constructed based on existing designs from the literature,  
 26 such as the thermoplastic towpregging developed by Edie et al. [41]. Figure 4 represents the different  
 27 steps of the process; all of which have an effect on the final properties of the towpreg. These steps  
 28 are briefly described below to give a general understanding of the towpregging line, prior to going into  
 29 further detail in the next sections.



- 1 a) The tows were guided through steel rings and tensioned with two interlocked
- 2 Polytetrafluoroethylene (PTFE) rollers.
- 3 b) Extra tension was applied using two additional PTFE rollers and the tows were merged by a
- 4 chamfered roller.
- 5 c) The fibres were pulled through the deposition chamber where an electrostatic spray gun
- 6 deposited powder particles onto the fibres at a 90° angle.
- 7 d) Heat was applied to the powder-coated fibres via an energy source tailored to the fibre type
- 8 in order to melt the powder.
- 9 e) The towpreg was cooled in ambient air under tension.
- 10 f) Finally, the vitreous towpreg was wound-up on a drum, which was motorised to keep the
- 11 whole line under tension.



12  
13 **Figure 4: Towpregging line schematic. The arrow indicates the direction in which the tow is**  
14 **travelling.**

15  
16 **3.2. Tow payout**

17 Tow payout for this study involved two tows being unwound from the as-supplied material drum while  
18 under tension. The payout design included grooves to guide the tensioning belts, a tapered internal  
19 cap diameter which could work with different drum sizes, and a ball bearing system for smoother tow  
20 payout. This system provided stable tension, which allowed for homogenised merging of the two tows  
21 on a grooved PTFE roller.

22  
23 **3.3. Powder deposition**

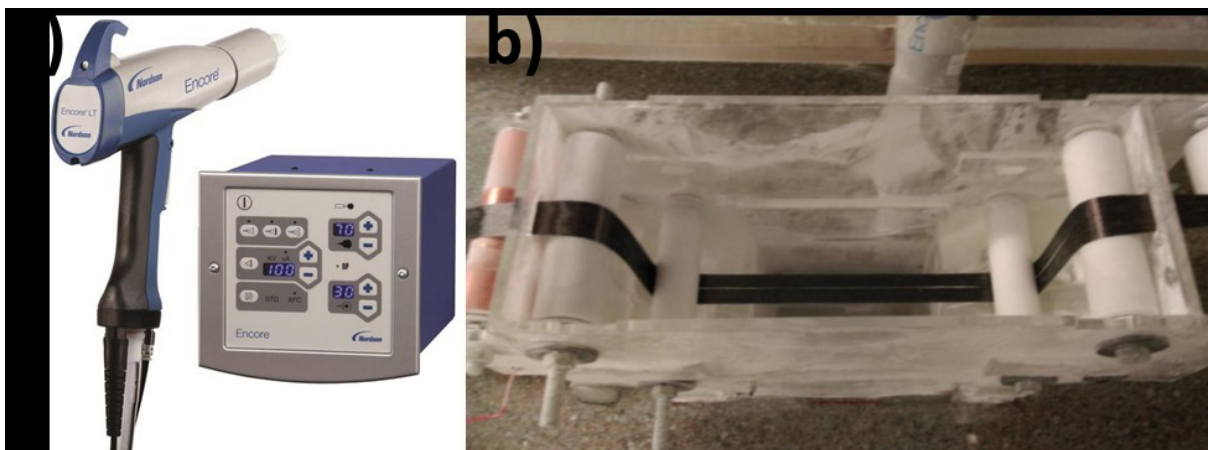
24 There are numerous methods of powder deposition for the towpregging process. Several of these  
25 methods were reviewed by Padaki and Drzal [60] including four types of fluidised bed deposition,  
26 powder slurry deposition, and electrostatic spraying. Another method is 'powder curtain' deposition  
27 which uses an auger to disperse powder across the tow as it passes underneath [44,61]. Early

1 iterations of the pilot towpregging line (presented herein) implemented a form of ‘powder curtain’  
2 deposition; using a vibrating hopper and mesh to create the ‘powder curtain’ [62]. This method,  
3 however, resulted in inconsistent powder dispersion along the towpreg.

4 For this study, electrostatic spraying was chosen as the method of powder deposition. A 60 W Encore  
5 LT electrostatic spray gun, from Nordson, was used to coat the fibres within an enclosed acrylic spray  
6 box (Figure 5). The gun was set to 100 kV at 60  $\mu$ A, using the smart flow setting for both the air and  
7 the powder flows, which helped with adjusting the flow to maintain the same powder velocity. The  
8 pressure was set to 5 bar. The surplus powder was evacuated from the spray box using a Nilfisk VHS-  
9 110 ATEX extraction unit with High Efficiency Particulate Air HEPA filter (1.1 kW), kept at maximal flow  
10 throughout the process to avoid clogging. The powder was recycled to avoid any waste.

11 The electrostatic attraction between the powder-epoxy and the tows was due to the powder being  
12 negatively charged by a corona electrode as it passed through the spray gun, while the tows were  
13 grounded. This phenomenon has been modelled for towpregging by Woolard and Ramani [63]. They  
14 demonstrated that electrostatic spray can coat both sides of the tow due to a ‘wrap-around’ effect,  
15 but that the strength of this effect was dependent on the spread tow width. Tow width was constant  
16 during this study so as not to introduce variability in powder deposition. This was achieved using  
17 grooved rollers at various points in the line.

18



19  
20 **Figure 5: a) Encore LT electrostatic spray gun and its control board (© Nordson). b) Plan view of the**  
21 **spray box where a cloud of negatively charged powder-epoxy coats the merged tows.**

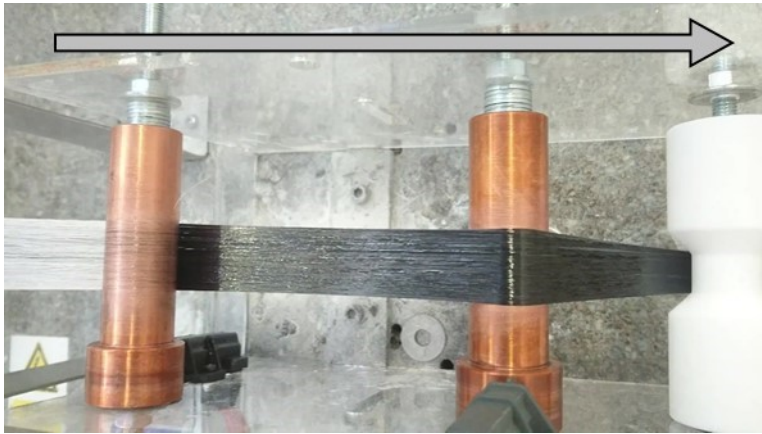
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### 23 3.4. Electrical heating of carbon fibre towpreg

24 As carbon fibres are electrically conductive, it has been shown by others that passing a direct current  
25 (DC) through the fibres can be used to heat towpreg [41,64] or even cure composite structures [65].  
26 This resistive heating is due to the Joule effect, and can achieve rapid increases in temperature while  
27 requiring less energy than conventional heating methods [65]. For this reason, resistive heating  
28 technology was employed in this study when producing CF towpreg. As shown in Figure 6, two copper  
29 rollers were connected to a DC generator using carbon brushes and a differential of 12 volts was set.  
30 As the CF passed between the rollers, they closed the circuit and generated heat. The current drawn  
31 oscillated between 140 and 160 mA, for a power consumption of only 1.68W to 1.92W. The current  
32 fluctuation was due to contact resistances, because of changes in both tow tension and powder  
33 concentration during processing.

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The temperature was monitored with an FLIR TG54 Spot IR Thermometer and was controlled to reach 120°C. The powder in contact with the fibres melted rapidly (Figure 6), demonstrating the ability to work at high speed (up to 15 metres per minute for a 50% fibre volume fraction), while keeping a constant temperature profile between the copper rolls. The copper rollers were coated with a release agent (Frekote 55-NC) and cleaned after each use to avoid any contaminants and inhomogeneity due to excess resin. The first two metres of towpreg were discarded for each run because, at start up, the towpregging line required some time to reach steady state conditions. Removing the first two metres also reduced the risk of any contamination due to Frekote residue.



11  
12 *Figure 6: DC electrical heating using copper rollers (plan view). The powder-epoxy coated tows (white) approach the first copper roller on the left. As the tows pass between the rollers, the powder-epoxy melts and sinters onto the carbon fibres (black). The arrow indicates the direction in which the tow is travelling.*

16  
17

### 3.5. Infrared heating for basalt fibre towpreg

18 Due to basalt fibres (BF) being non-conductive, an alternative heat source was required for processing  
19 the BF towpreg. From the literature, possible alternatives included tunnel ovens [66–68], convection  
20 ovens [41,69,70], hot gas torches [67,69], infrared lamps [61,68,70], and microwave ovens [41]. In this  
21 study, the towpregging line used a 1kW infrared (IR) lamp. This lamp had an adjustable power, was 30  
22 cm long, and had a reflector to focus radiated heat towards the substrate surface. Again, the  
23 temperature was monitored using an IR thermometer throughout the process. To maintain a  
24 homogenous heating profile, the maximum line operating speed was set at 5 m/min. The  
25 temperatures, however, were initially found to be lower on the non-exposed side. As such, an  
26 additional reflective system (a copper sheet) was added to improve the temperature homogeneity on  
27 both sides of the BF tow.

28  
29

### 3.6. Cooling and recovery

30 After the powder was melted, the towpreg proceeded to a grooved roller to maintain a constant  
31 width. It was then cooled quickly (in about five seconds) by convection and radiation to the  
32 surrounding atmosphere, prior to being wound up onto the winding drum. The towpreg temperature,  
33 located immediately before the winding drum, was consistently between 30°C and 40 °C, which was

1 lower than the melt temperature of the epoxy. Hence, the towpreg was wound in a vitreous state and  
2 maintained optimal straightness due to the tension applied to the fibres throughout the process. The  
3 rotating drum itself was powered by a Mellor electric 24 V DC Brushed DC Geared Motor with a  
4 maximum output speed of 5.3 rpm. As the drum itself had a diameter of 40 cm, the maximum  
5 towpregging process speed was 6.66 m/min. As the processing speed wasn't a constraint in this study,  
6 the motor speed was the main parameter used in order to control the fibre volume fraction (FVF) of  
7 the towpreg. In this study, all towpreg samples were manufactured using the same powder-epoxy  
8 (PE6405).

9

#### 10 4. Composite plate manufacturing and testing

11

##### 12 4.1. Manufacturing overview

13 The overall manufacturing system, illustrated in Figure 7, is described briefly below. Further details  
14 are given in subsequent sections.

15 Stages 1 – 2: Towpreg was produced on the towpregging line.

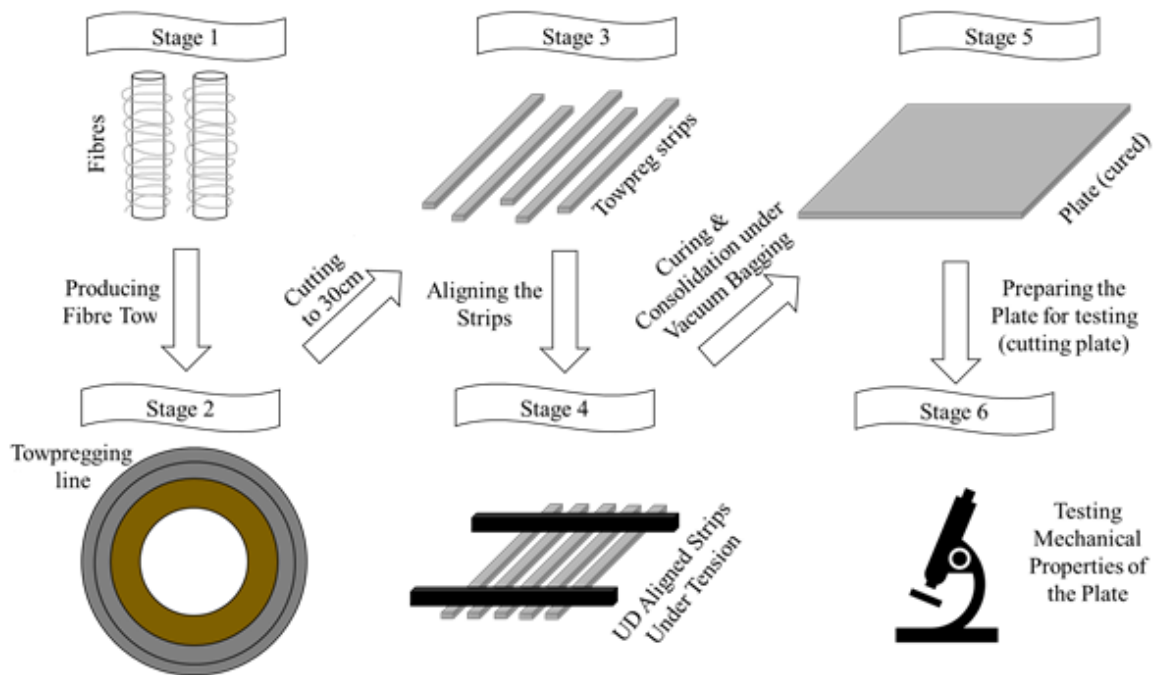
16 Stages 2 – 3: The towpreg was cut into strips of exactly 30 cm length using a guillotine system.

17 Stages 3 – 4: The towpreg strips were aligned and clamped in the fibre tensioning apparatus.  
18 This is used to ensure tow straightness in the unidirectional plates during cure.

19 Stages 4 – 5: The tensioned preform was cured under vacuum to produce a unidirectional  
20 composite plate.

21 Stages 5 – 6: The composite plate was tabbed and cut, and the resulting specimens were  
22 tested and characterised.

23

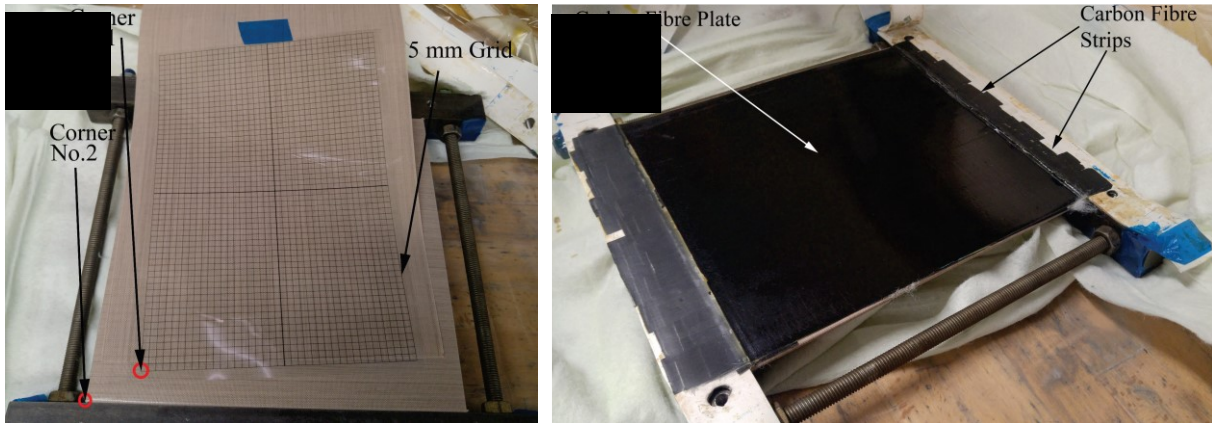


1  
2 **Figure 7: Manufacturing overview - from tows to plate manufacturing and testing.**

3  
4 **4.2. Plate manufacturing**

5 It has been shown that fibre misalignment and fibre ‘waviness’ can affect the mechanical performance  
6 of FRPs [24,71,72]. The results presented by Mamalis et al. [33,35] confirmed that tensioning  
7 unidirectional CF/powder-epoxy improved the mechanical properties of the material by increasing the  
8 fibre straightness and alignment. Using the towpregging line, highly aligned CF/powder-epoxy may be  
9 realised through automation, however, additional handling and processing of tows can lead to  
10 increased fibre breakage [43,44]. Therefore, the composite plates for this study were manufactured  
11 using the same tow tensioning apparatus developed by Mamalis et al. [33] so that a direct comparison  
12 could be made between the two sets of results. By comparing the two, it would be possible to  
13 determine if the towpregging process caused any significant fibre damage or breakage.

14 For the plate manufacturing, 30 cm long towpreg ‘strips’ were cut and placed on a PTFE coated flat  
15 steel plate; alignment was aided using a plastic grid (see Figure 8a). Once all the strips were placed  
16 correctly, they were locked in place by the tensioning apparatus (described further in [33]). Five layers  
17 of strips were necessary to obtain 1 mm thick plates. After the alignment grid was removed, a  
18 tensioning force of approx. 3.0 kN was applied. This was the force chosen by Mamalis et al. [33], to  
19 achieve optimal alignment of the T700S-24K-50C tows. An extra PTFE coated plate was placed on the  
20 top of the tensioned towpreg, and the whole tensioning apparatus was then vacuum bagged. The  
21 system was kept under vacuum at all times during processing. The curing cycle was set at 40 °C for 12  
22 hours to remove all form of moisture from the strips. The temperature then ramped to 130°C for 2  
23 hours, melting the powder and consolidating the system. Following this, the plate was cured at 185°C  
24 for 2 hours and then cooled to ambient temperature. This methodology was used to produce both  
25 UD-CFRP and UD-BFRP plates.



1  
2 **Figure 8: Tensioning rig system: a) Strip alignment using a grid, b) Cured plate.**

3  
4 **4.3. Quality control - weight measurements and void analysis**

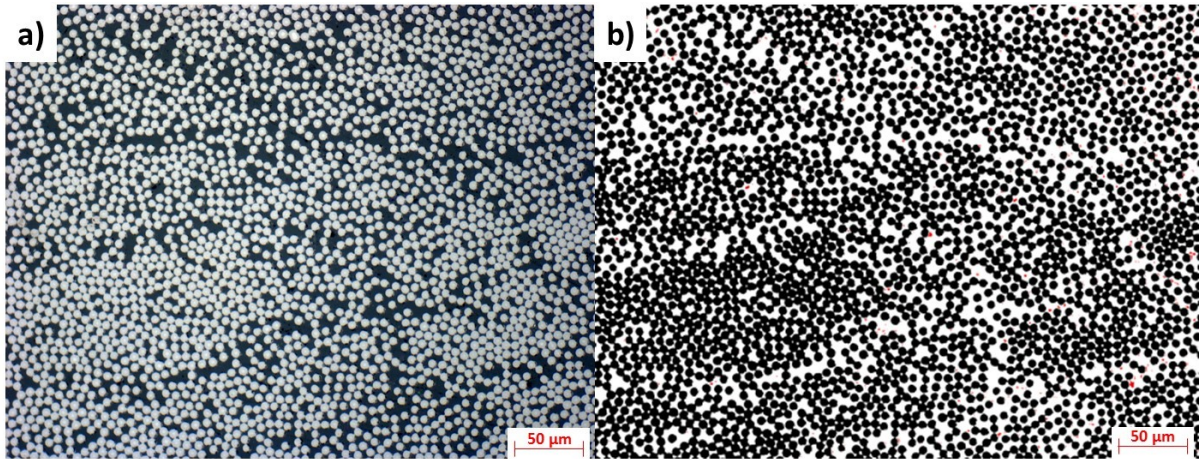
5 Fibre volume fraction has an important influence on the mechanical properties of the composite  
6 materials. As powder deposition may vary during towpreg production [61], it was important to  
7 understand what influence this may have on the mechanical properties of the cured laminates.  
8 Different FVF towpreg were produced by slightly modifying the line speed. For each line speed setting,  
9 30 cm strips of towpreg were cut and weighed, and the FVF of each strip was determined based on  
10 the known volume and mass of the virgin fibre tows. Resin burn-off was also performed on random  
11 samples to cross-check their FVF; ASTM D3171-15 – Procedure G – kept at 550°C for 60 min in high  
12 temperature furnace.

13 To investigate the influence of FVF over a wide range, the strips were filtered according to four FVF  
14 sub-groups: 45% to 50%, 50% to 55%, 55% to 60% and 60% to 65%. A plate for each FVF sub-group  
15 was manufactured using the methodology described in the previous section.

16 Optical microscopy was also used in this study. A UD-CFRP specimen was cut into 10 samples and  
17 embedded in epoxy. The 10 embedded samples were polished, and their cross sections were observed  
18 using an Axioskop® 2MAT optical microscope (Figure 9a). 50 photos (5 per sample) were taken with  
19 Axio vision SE64® software, and a FVF analysis was performed on ImageJ®, with 8 bits grayscale  
20 settings (Figure 9b). Two filters were used to differentiate the resin, fibres and porosity (fibres contrast  
21 threshold: 117 to 255, voids contrast threshold: 0 to 14). The fibre to resin to void surface ratio  
22 obtained on each individual photos, combined with a large set of 50 samples, allowed for a FVF  
23 estimate. This quantitative method allowed not only to crosscheck the FVF, but also to detect and  
24 determine the sample's porosity, as shown in Table 2. The table displays a low porosity (0.437%)  
25 compared to other VBO systems and tapes [73]. It is worth noticing that the FVF was found slightly  
26 below target (52.07% instead of 52.50%).

27





1  
2 **Figure 9: a) Optical microscopy of a powder-epoxy UD-CFRP cross section in reflection mode. b) Resin**  
3 **(white), fibres (black), and voids (red) identified using volume fraction analysis with ImageJ software**  
4 **filters.**

5  
6 **Table 2: Quantitative study of porosity and FVF along a UD-CFRP specimen length, extracted from a**  
7 **plate targeting 52.5 % FVF.**

|            | Porosity (%) | FVF (%) |
|------------|--------------|---------|
| Mean       | 0.44         | 52.07   |
| St. Dev.   | 0.13         | 4.03    |
| C.o.V. (%) | 29.54        | 7.74    |

8  
9 **4.4. Mechanical test methodology**

10 The UD-CFRP and UD-BFRP plates were prepared and cut into specimens according to the mechanical  
11 test standards used in this study. For UD-CFRP, tensile tests were performed in the longitudinal  
12 direction (i.e. 0° direction) according to BS EN ISO 527-5 [74]. 10 specimens were tested for each of  
13 the four FVF sub-groups. For UD-BFRP, 0° & 90° tensile tests, 0° & 90° flexural tests, and 0°  
14 compression tests were performed according to BS EN ISO 527-5, BS EN ISO 14125 [75], and ASTM  
15 D6641/D6641M-16E1 [76], respectively. In each type of test, 8 specimens were tested.

16 Additional 0° tensile tests (BS EN ISO 527-5) were carried out for aged specimens which had been  
17 immersed in water. For UD-CFRP, specimens were immersed at room temperature (approx. 21°C),  
18 50°C, and 75°C for 100 days. For UD-BFRP (Basaltex only), specimens were immersed at room  
19 temperature (approx. 21°C) and 55°C for 60 days. The specimens were periodically weighed using a  
20 microbalance to determine the water uptake over the duration of immersion. Tensile tests were also  
21 performed periodically to determine the influence of water uptake on the tensile properties of the  
22 specimens. In each case, 10 specimens were tested. Tensile testing was carried out when the  
23 specimens were still wet.

24 Tensile tests were performed using an MTS Criterion® Model 45 (C45.305) with a 300 kN load cell. A  
25 crosshead speed of 2 mm/min was used for all tensile tests. In all tensile test cases, the test specimens  
26 were end tabbed with ±45° GRFP and clamped in hydraulic grips with a clamping pressure of approx.  
27 100 bar. A speckle pattern was applied to the surface of each specimen and 2D strain was measured  
28 with digital image correlation (DIC).

1 Compression tests were performed using an MTS Criterion® Model 45 (C45.305) with a 300 kN load  
2 cell and a fixture specially designed by Wyoming Test Fixtures for this specific type of test. Specimens  
3 were clamped by applying a torque of 4 N m. A crosshead speed of 1.3 mm/min was used for testing.  
4 Again, DIC was used to measure strain for all compression tests.

5 Flexural testing was performed using an Instron 3369 Dual Column Tabletop Test System with a 50 kN  
6 load cell. A four point bend fixture was used with an outer span of 45 mm (Class III, Table 4 of BS EN  
7 ISO 14125). Testing was carried out with a crosshead speed of 2.15 mm/min. DIC was used to track  
8 the midspan deflection of the test specimens.

9

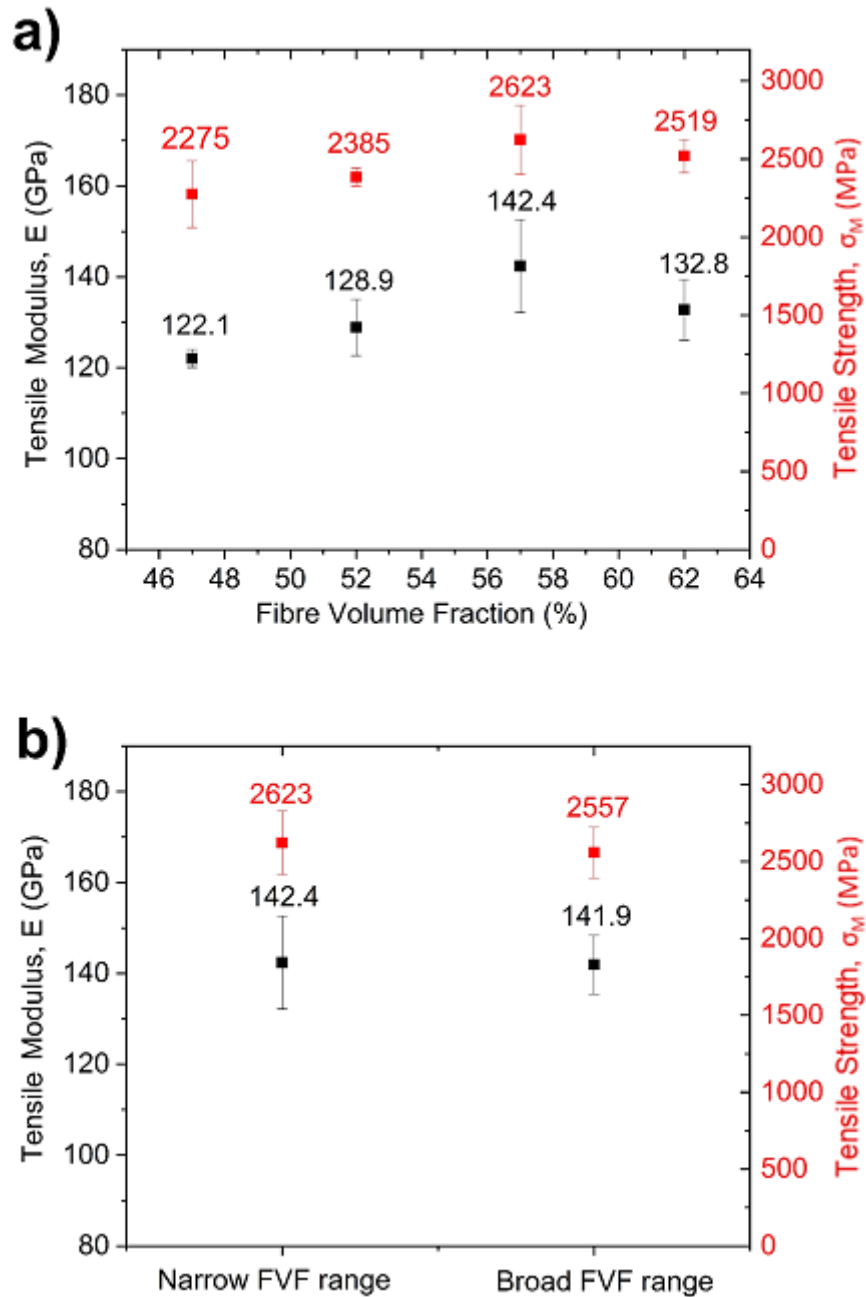
## 10 5. Results and discussion

11

### 12 5.1. Fibre volume fraction investigations of UD-CFRP

13 Figure 10(a) shows the influence of FVF on the tensile properties of the UD-CFRP. The general trend  
14 was that the tensile properties (tensile strength,  $\sigma_M$ , and tensile modulus,  $E$ ) improved with increasing  
15 FVF before decreasing above 57.5%. In addition to the void content analysis presented in Section 4.3,  
16 these results suggested that excellent consolidation was achieved at 47.5% FVF and 52.5% FVF. While  
17 the mean tensile properties of the 57.5% FVF specimens were the highest of the four sub-groups, the  
18 standard deviation (SD) was also the highest for both  $\sigma_M$  and  $E$ . This suggested that some specimens  
19 were well consolidated while others were not, due to insufficient wet-out and, consequently, poor  
20 load transfer between fibres and matrix. At 62.5%, this latter effect worsened and the mean tensile  
21 properties decreased. This upper limit in FVF was in agreement with what has been demonstrated for  
22 VBO prepregs previously [77]. At higher FVFs, consolidation was limited by the pressure that can be  
23 applied i.e. VBO pressure is 1 atm max. In this regard, Gutowski et al. [78] showed that FVF  
24 asymptotically approaches a maximum as a fibre bed compresses and resists the applied pressure. In  
25 the context of this study, higher FVF could be achieved via pultrusion of the towpreg through a die  
26 [69]. Further investigation of this concept was outside the scope of current work, however, it may be  
27 explored in future work (see Section 7). Alternatively, it is likely that additional autoclave or heated  
28 press processing would be required to achieve better quality and reliability above 60% FVF.





1  
2 **Figure 10: a) Influence of average FVF on 0° UD-CFRP mechanical properties.. b) Influence of strip**  
3 **FVF dispersion at 57.5 % FVF on 0° UD-CFRP mechanical properties.**

4

5 To further study how mechanical properties may be affected by the FVF variation of towpreg, Figure  
6 10(b) shows the results of two sets of specimens (both with an average FVF of 57.5%) which were  
7 manufactured using two different FVF ranges of towpreg. The original plate with 57.5% FVF contained  
8 strips of the same range (55% to 60%), called narrow FVF range. For the second plate, strips of various  
9 FVF were used, altering between high and low FVF layers of strips, but still overall reaching the same  
10 FVF as the first plate (i.e. broad FVF range). The results showed very similar values of  $\sigma_M$  and  $E$ . This  
11 suggested that the low viscosity of the powder-epoxy allowed for flow from resin-rich regions to resin-  
12 poor regions, leading to similar homogeneity for the two plates.

1 As previously mentioned, Mamalis et al. have studied the same powder-epoxy system with T700S-  
 2 24K-50C fibres [33,35]. In their studies, plates were manufactured in a tensioning apparatus with  
 3 powder manually dispersed onto the fibres. By using the same tensioning apparatus in this study, it  
 4 was possible to directly compare the 0° tensile properties and determine whether the towpregging  
 5 process had any adverse effect on the fibre properties. As shown in Table 3, the 0° tensile strength  
 6 ( $\sigma_M$ ) for 57.5% FVF specimens was almost identical to that which was reported by Mamalis et al. (for  
 7 59% FVF), while the 0° tensile modulus ( $E$ ) was slightly higher. This confirms that the towpregging line  
 8 does not have any significant adverse effect on the CF, such as fibre breakage.

9 Table 3 also shows the 0° tensile properties of other equivalent material systems of interest. As can  
 10 be seen, the 57.5% FVF specimens were stronger and stiffer than the data reported by Toray for UD  
 11 epoxy composites reinforced with T700s carbon fibres [56] while having a slightly lower FVF (57.5%  
 12 compared to Toray's 60%). Comparatively, normalised data shows a substantial increase in both  
 13 strength (+7.33%) and stiffness (+10.06%). This improvement was attributed to the improved fibre  
 14 alignment achieved with the tensioning apparatus. This highlighted the technological potential for  
 15 industrial manufacturing processes where carbon fibres are kept under tension, such as pultrusion,  
 16 filament winding, and, to a lesser extent, ATP. For further comparison, Table 3 includes the 0° tensile  
 17 properties of two flat-section pultruded CF/epoxy systems, both used for manufacturing turbine blade  
 18 spars [79,80]. Both pultruded systems show higher FVF, but only the tensile modulus of Röchling EP-  
 19 CFK (66% FVF) is higher than the 57.5% FVF UD-CFRP produced in this study. The lower 0° tensile  
 20 strength can be attributed to a difference in fibre type and fibre quality e.g. the individual tensile fibre  
 21 strength of T700S fibres [56] are markedly higher than Zoltek's PX35 fibres [81].

22  
 23  
 24

**Table 3: Comparison of the 0° tensile properties for powder-epoxy UD-CFRP and equivalent material systems of interest.**

|                                  | FVF (%) | $\sigma_M$ (MPa) | SD (MPa) | $E$ (GPa) | SD (GPa) |
|----------------------------------|---------|------------------|----------|-----------|----------|
| Towpreg line                     | 47.5    | 2275.0           | 212.3    | 122.1     | 1.9      |
|                                  | 52.5    | 2385.0           | 56.5     | 128.9     | 6.1      |
|                                  | 57.5    | 2623.0           | 220.8    | 142.4     | 10.2     |
|                                  | 62.5    | 2519.0           | 106.7    | 132.8     | 6.7      |
| Mamalis et al. [33]              | 59.2    | 2650             | -        | 134       | -        |
| Toray T700S [56]                 | 60      | 2550             | -        | 135       | -        |
| Gurit CF SparPreg [82]           | 56*     | 2234             | -        | 140       | -        |
| Zoltek PX35 Pultruded Plate [83] | 62      | 2350             | -        | 140       | -        |
| Röchling EP-CFK [79]             | 66      | 2000             | -        | 150       | -        |

25 \* Normalised to 56% in the technical data sheet

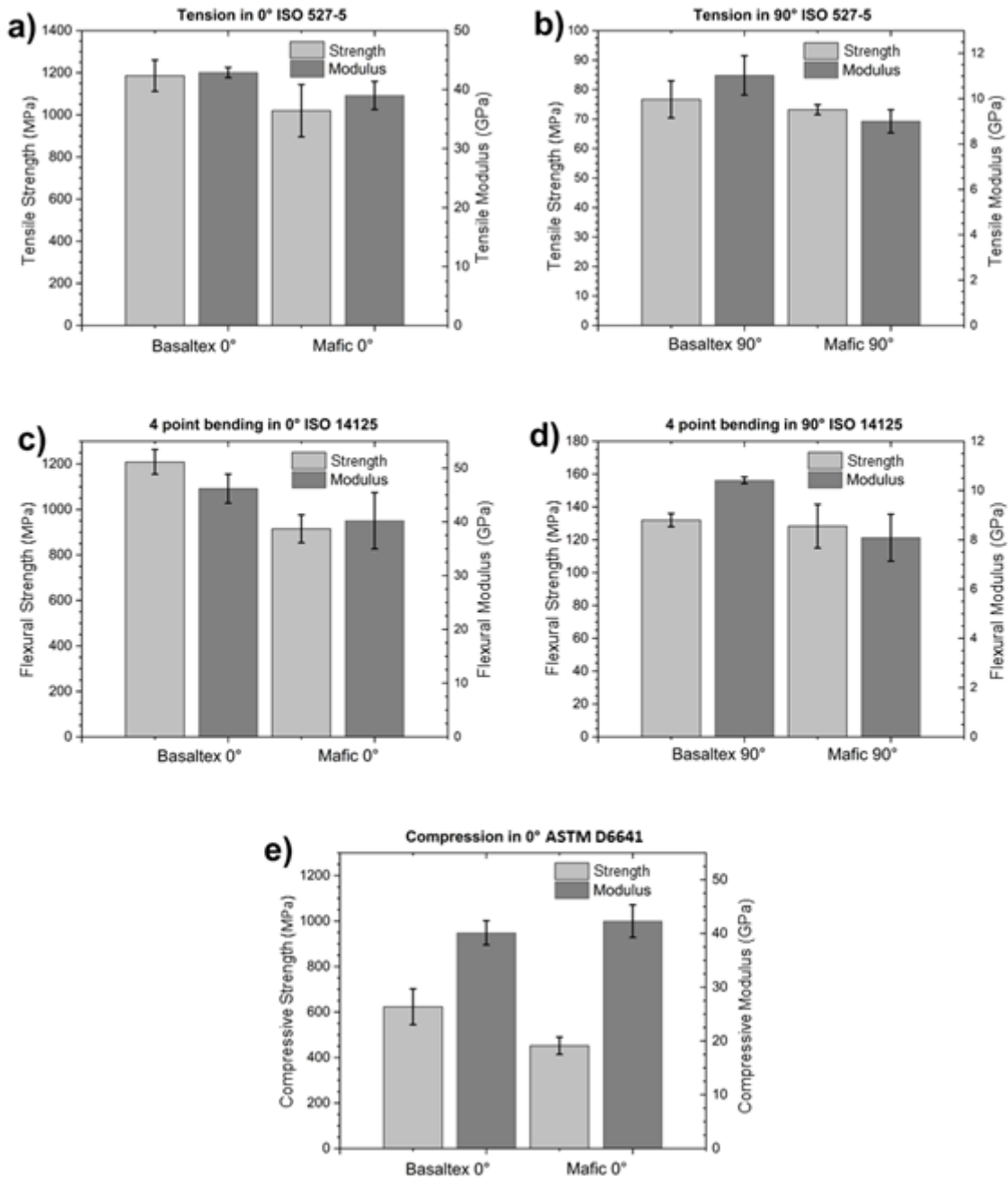
26

## 27 5.2. Comparison of UD-BFRP plates

28 Powder-based UD-BFRP plates were manufactured to compare Mafic and Basaltex basalt fibres. A FVF  
 29 of 50% was targeted to ensure that homogeneous samples were processed. The results of the various

1 tests are plotted in Figure 11. Overall, the Basaltex fibre composites exhibited better mechanical  
 2 properties than those with Mafic fibres. The mean strength of the Basaltex composite specimens was  
 3 superior in 0° tension (+14%), 0° flexure (+24.3%) and 0° compression (27.4%), when compared to the  
 4 Mafic composites. Basaltex composite specimens were also stiffer in 0° tension (+9.1%) and 0° flexure  
 5 (+12.9%) but not in 0° compression (-5.5%). This important mismatch in properties is not unexpected  
 6 as the basalt fibres are naturally sourced, and will display different properties as their chemical  
 7 compositions depends on their extraction location [11].

8



9

10 **Figure 11: Mechanical properties of Basaltex and Mafic basalt towpreg based composites in tension**  
 11 **(a & b), flexure (c & d) and compression (e).**

12

1 The mechanical properties in the transverse direction allow a direct comparison of the interfacial  
 2 properties, as the powder-epoxy is the same in both cases. Basaltex and Mafic BFRP displayed very  
 3 similar strength in both 90° tension (+4.6% for Basaltex) and 90° flexure (+3% for Basaltex)  
 4 configurations. As the tensile strength in transverse direction is largely matrix and fibre interface  
 5 dominated, it was not surprising to obtain similar properties. Basaltex samples still exhibited much  
 6 better transverse stiffness properties than Mafic specimens, doubling the difference already obtained  
 7 longitudinally. It is likely that this is a result of a more effective fibre sizing.

8 For further comparative purposes, the results are reproduced in Table 4, Table 5, and Table 6 alongside  
 9 mechanical properties for similar material systems. Direct comparison with other BFRP studies is  
 10 difficult because much of the existing literature has focussed on the mechanical properties of bi-  
 11 directional or multi-directional laminates [11,12]. Wang et al. [84] studied mechanical properties of  
 12 UD-BFRP (epoxy matrix) in the 0° direction, comparing it to UD-GFRP in tension, compression, and  
 13 flexure. They reported that the 0° tensile strength of the UD-BFRP (640 MPa) was much lower than  
 14 UD-GFRP (1029 MPa), and attributed this to a variation in the tensile strength of individual fibre  
 15 strands i.e. large variation in basalt fibre quality. Table 4 confirms that their values were much lower  
 16 than the tensile strengths reported herein for Mafic fibres (37% lower) and Basaltex fibres (46%  
 17 lower). Nevertheless, their 0° flexural strength was similar to the values for Mafic and Basaltex fibres,  
 18 and notably, their 0° compressive strength was higher in both cases.

19

20 **Table 4: Comparison of the tensile properties for powder-epoxy UD-BFRP and equivalent material**  
 21 **systems of interest.**

|                            | FVF (%) | Test direction | $\sigma_M$ (MPa) | SD (MPa) | $E$ (GPa) | SD (GPa) |
|----------------------------|---------|----------------|------------------|----------|-----------|----------|
| Mafic towpreg              | 50      | 0°             | 1020.0           | 124.0    | 39.0      | 2.4      |
|                            |         | 90°            | 73.2             | 1.7      | 9.0       | 0.5      |
| Basaltex towpreg           | 50      | 0°             | 1186.0           | 74.3     | 42.9      | 0.9      |
|                            |         | 90°            | 76.7             | 6.3      | 11.0      | 0.9      |
| Wang et al. [84] (UD-BFRP) | -       | 0°             | 640              | -        | 40.8      | -        |
| Gurit GF                   | 56**    | 0°             | 1225             | -        | 48        | -        |
| Sparpreg* [82]             | 50      | 90°            | 42               | -        | 12        | -        |
| Röchling EP-GZUV [79]      | 64      | 0°             | 1500             | -        | 45-50     | -        |

22 \* Values for 1600 g/m<sup>2</sup>

23 \*\* Normalised to 56% in TDS

24

25 Davies and Verbouwe [15] investigated the mechanical properties of a stitched BFRP fabric with 357  
 26 g/m<sup>2</sup> in the 0° direction and 50 g/m<sup>2</sup> in the 90° direction (ratio of 7:1). Their quasi-UD BFRP fabric was  
 27 made using Basaltex fibres sized for epoxy with alkosilane agent, which are believed to be very similar  
 28 to the Basaltex fibres used in this study. The flexural strength reported in their work was 698 MPa,  
 29 which is 42.3% lower than the value obtained in this study. Likely, this was due to the presence of

1 fibres in the transverse direction; typically used to stabilise the fabric while being laid up. Having less  
 2 fibres aligned with the loading direction will reduce the strength in that direction, and will also  
 3 increase the crimp of the BFRP, thus reducing the ultimate strength of composite materials [85].

4 Table 4, Table 5, and Table 6 also display values for commercially available material systems that are  
 5 used for turbine blade spar caps; Gurit GF Sparpreg [82], a UD-GFRP prepreg system, and Röchling EP-  
 6 GZUV [79], a UV-cured GF/epoxy pultruded section. Both UD-BFRP systems from this study compare  
 7 favourably with the 0° tensile properties of the commercially available UD-GFRP (when factoring in  
 8 the higher FVFs reported for the latter). Notably, the 90° tensile strengths were markedly higher for  
 9 the powder-epoxy systems than for Gurit GF Sparpreg. These values were equal to the tensile strength  
 10 of the neat powder-epoxy matrix (73.1 MPa [35]), which was atypical for conventional epoxy systems.  
 11 Often some strength reductions are expected for transverse UD composite properties compared to  
 12 neat matrix properties. These reductions are usually attributed to the presence of defects (such as  
 13 voids), which form during composite processing, and aid crack initiation and propagation through the  
 14 resin matrix. This is a particular issue for conventional epoxies, which are brittle, however, toughened  
 15 epoxy systems have reported significantly better 90° tensile strengths: 90 MPa for Solvay’s CYCOM®  
 16 5276-1 (G40-800 Tape) [86], 77 MPa for Hexcel’s Hexply® 8552 UD CF prepreg [87], and 64 MPa for  
 17 Hexcel’s Hexply® M73/IM7 UD tape. In the latter case, the 90° tensile strength of the composite  
 18 approached the tensile strength of the neat M73 epoxy i.e. 77 MPa). The improved performance of  
 19 toughened epoxies may be attributed to their greater ability to resist crack growth compared to  
 20 conventional systems. For the powder-epoxy in this study, it has been shown to have relatively ductile  
 21 behaviour with exceptionally high mode I fracture toughness; between 1,100 and 1,900 J/m<sup>2</sup>,  
 22 depending on the fibre and sizing [35,88]. It should be noted that the ductile behaviour of the powder-  
 23 epoxy is also evident in the tensile failure strain values for the 90° specimens: 0.92 ± 0.14%, which is  
 24 high, compared to standard epoxy UD materials.

25

26 **Table 5: Comparison of the flexural properties for powder-epoxy UD-BFRP and equivalent material**  
 27 **systems of interest.**

|                            | FVF (%) | Test direction | $\sigma_M$ (MPa) | SD (MPa) | $E$ (GPa) | SD (GPa) |
|----------------------------|---------|----------------|------------------|----------|-----------|----------|
| Mafic towpreg              | 50      | 0°             | 915.0            | 61.3     | 40.2      | 5.2      |
|                            |         | 90°            | 128.0            | 13.3     | 8.1       | 0.9      |
| Basaltex towpreg           | 50      | 0°             | 1209.0           | 54.3     | 46.2      | 2.7      |
|                            |         | 90°            | 132.0            | 4.0      | 10.4      | 0.1      |
| Wang et al. [84] (UD-BFRP) | -       | 0°             | 1115             | -        | 43.7      | -        |
| Gurit GF Sparpreg* [82]    | 50      | 0°             | 1347             | -        | 42        | -        |
| Röchling EP-GZUV [79]      | 64      | 0°             | 890-1020         | -        | 35-45     | -        |

28 \* Values for 1200 g/m<sup>2</sup>

29

1 The strong transverse performance of the UD-BFRP was confirmed by the 90° flexural properties which  
 2 were similar to those reported by Mamalis et al. [33] for powder-epoxy UD-CFRP. 0° flexural properties  
 3 were also similar to the UD-GFRP systems shown in Table 5. For 0° compressive properties (Table 5),  
 4 however, the UD-BFRP again performed poorly compared to UD-GFRP. Failure was predominantly due  
 5 to kinking of the fibres, despite the high degree of fibre alignment, which suggests that the ductile  
 6 nature of the powder-epoxy played a role in the comparatively low 0° compressive properties.

7

8 **Table 6: Comparison of the compressive properties for powder-epoxy UD-BFRP and equivalent**  
 9 **material systems of interest.**

|                         | FVF (%) | Test direction | $\sigma_M$ (MPa) | SD (MPa) | $E$ (GPa) | SD (GPa) |
|-------------------------|---------|----------------|------------------|----------|-----------|----------|
| Mafic towpreg           | 50      | 0°             | 452.0            | 38.0     | 42.3      | 3.0      |
| Basaltex towpreg        | 50      | 0°             | 623.0            | 78.9     | 40.1      | 2.2      |
| Wang et al. [84]        | -       | 0°             | 836              | -        | -         | -        |
| Gurit GF Sparpreg* [82] | 56**    | 0°             | 950              | -        | 47        | -        |

10 \* Values for 1600 g/m<sup>2</sup>

11 \*\* Normalised to 56% in TDS

12

13 5.3. Hygrothermal aging

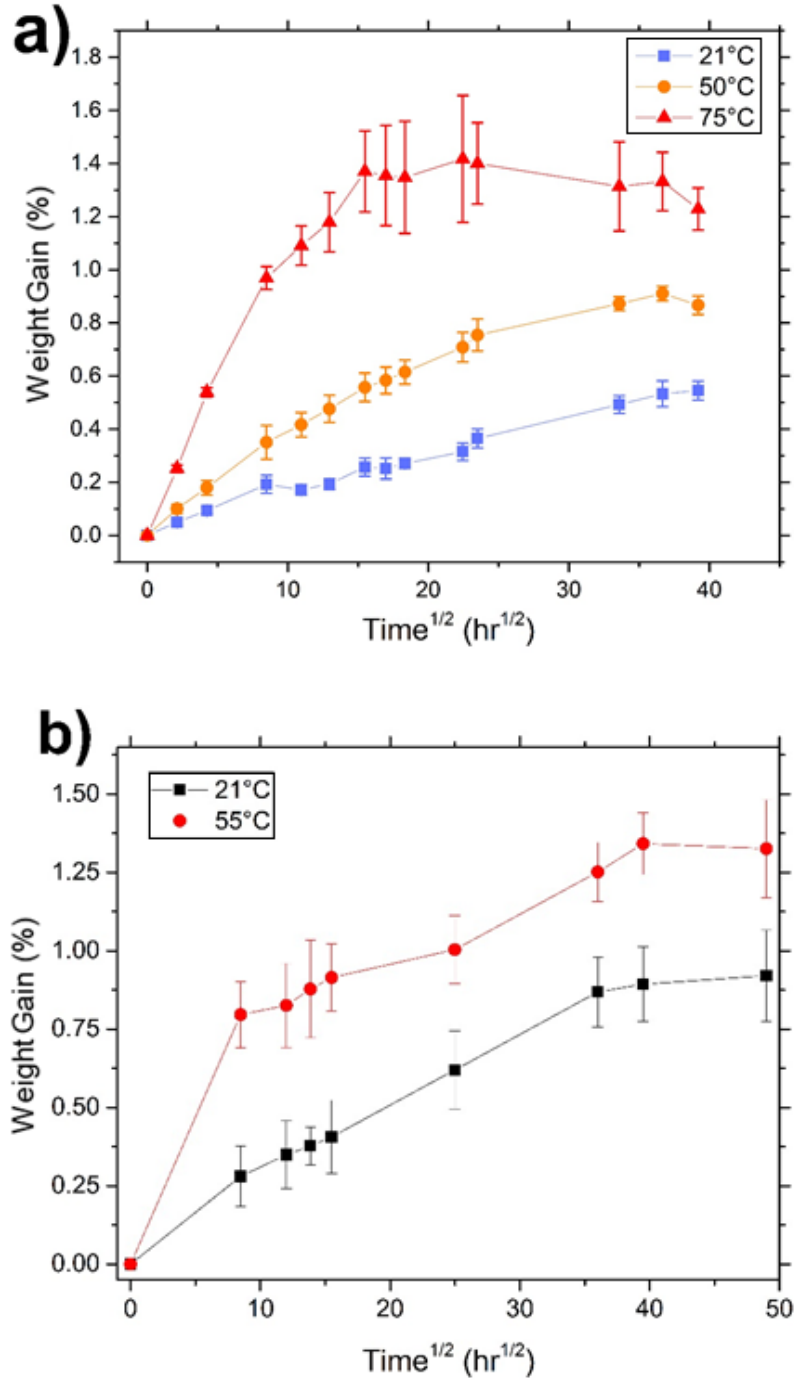
14 The results for 0° tensile testing of UD-CFRP (Figure 10 (a) and Table 3) show that the standard  
 15 deviation was the lowest at 52.5% FVF; highlighting better reproducibility. As such, this FVF range was  
 16 selected for a subsequent hygrothermal ageing study. In addition, Basaltex UD-BFRP was selected for  
 17 the hygrothermal ageing study as it displayed better mechanical properties than Mafic UD-BFRP. UD-  
 18 CFRP and UD-BFRP specimens were immersed in water as described in Section 4.4. In both cases, the  
 19 temperature had an influence on the absorption rate and the water saturation level (see Figure 12).

20 As expected, rates of water absorption were highly temperature dependent. In the case of UD-CFRP,  
 21 the specimens reached saturation within approx. 15 days of immersion at 75°C, but showed a high  
 22 degree of scatter after that point with a potential loss of mass. Such a phenomena is not uncommon  
 23 due to leaching of soluble components of the polymer matrix, which is exacerbated at higher  
 24 temperatures [36]. At lower immersion temperatures, the UD-CFRP and UD-BFRP displayed similar  
 25 characteristics, but differed in the quantity of water absorbed. Grogan et al. [36] reported similar  
 26 differences in weight gain for powder-epoxy CFRP and GFRP specimens, despite the specimens having  
 27 similar FVFs. Assuming that the water was predominantly absorbed by the resin matrix, they  
 28 attributed this discrepancy to the difference in resin mass fraction (i.e. the resin mass fraction in the  
 29 GFRP was lower due to the higher density of glass fibres (approx. 2520 kg/m<sup>3</sup>) compared to carbon  
 30 fibres (approx. 1800 kg/m<sup>3</sup>)). They stopped short, however, of calculating the weight gain with respect  
 31 to the resin mass fraction,  $W_r$ , using the simple equation,

$$W_r = \frac{W_c}{m_r} \quad (1)$$

1 Where  $W_c$  is the weight gain of the composite material, and  $m_r$  is the resin mass fraction of the  
 2 composite material.

3



4  
 5 *Figure 12: Composite weight gain ( $W_c$ ) due to immersion of powder-epoxy based UD-CFRP (a) and*  
 6 *UD-BFRP (b) against the square root of time at different temperatures.*

7

1 Using Equation 1, it is possible to quantify the weight gain with respect to the resin mass fraction and  
2 compare it to the equivalent value for neat resin. For example, after 60 days at 50°C and ambient  
3 pressure, Grogan et al. [36] reported a weight gain of approx. 0.8% for GFRP (reported resin mass  
4 fraction of 33%), and approx. 1.0% for CFRP (reported resin mass fraction of 42%). Using Equation 1,  
5 this equates to 2.42% weight gain w.r.t to the resin mass fraction of the GFRP, and 2.38% weight gain  
6 w.r.t to the resin mass fraction of the CFRP. In comparison, under the same immersed conditions, they  
7 reported that the neat epoxy powder (density of 1220 kg/m<sup>3</sup>) had a weight gain of approx. 2% with  
8 respect to its initial weight. This shows that both composites absorbed similar quantities of water, and  
9 both were slightly higher values than the weight gain of the neat resin. Applying the same calculation  
10 to the UD-CFRP in the current study (resin mass fraction of 38%), after 60 days at 50°C and ambient  
11 pressure, the specimens had a weight gain of approx. 2.26% w.r.t to the resin mass fraction of the  
12 CFRP. This shows excellent consistency between the two studies, and offers a simple, yet powerful  
13 method for analysing the influence of the polymer matrix on water absorption.

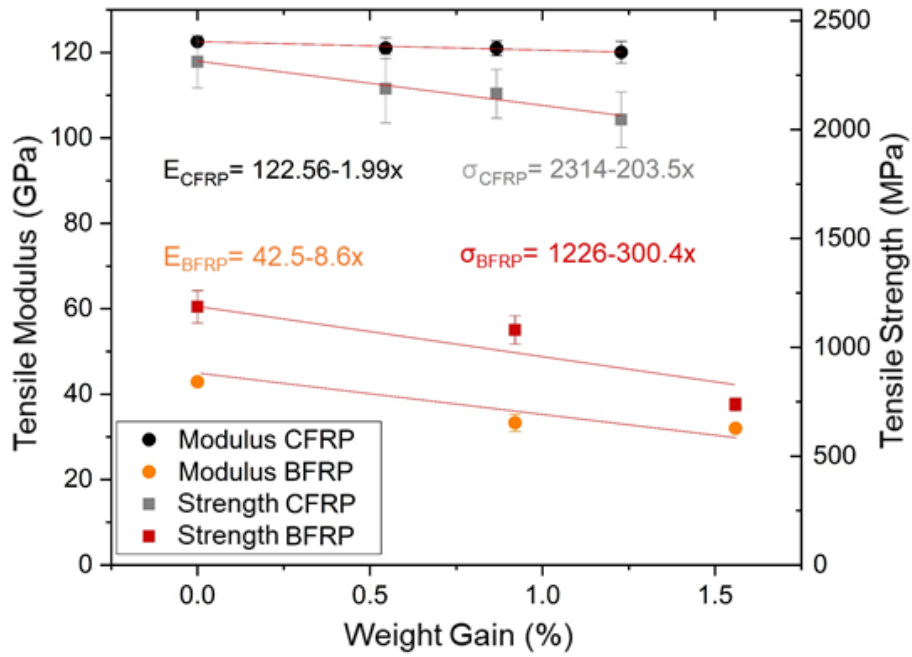
14 Although immersed ageing of the UD-BFRP was carried out at 55°C, it was considered that Equation 1  
15 could provide some indication of how the composite performs in comparison to the UD-CFRP. As such,  
16 after 60 days at 55°C and ambient pressure, the UD-BFRP specimens were calculated to have increased  
17 in weight by approx. 4.14% with respect to the resin mass fraction (31.4% for a fibre density of 2670  
18 kg/m<sup>3</sup> [58]). This was double the weight absorbed by the UD-CFRP in the same time span, but was  
19 consistent with the values reported by Davies and Verbouwe [15]. They performed water immersion  
20 studies (at 4°C, 25°C, 40°C, and 60°C) on quasi-UD GF/epoxy and BF/epoxy; Basaltex fibres were used  
21 in the latter case also. Interpolating between 40°C and 60°C at 60 days, their GFRP exhibited a weight  
22 gain of approx. 0.95%, while their BFRP exhibited a weight gain of approx. 1.15%. For their reported  
23 resin mass fractions of 36.2% (GFRP) and 33.5% (BFRP), this equates to weight gains, with respect to  
24 resin mass fraction, of 2.62% and 3.43%. Despite potential errors introduced by interpolation and  
25 approximation, these values are relatively consistent with both the values in this study (for BFRP) and  
26 the study by Grogan et al. [36] (for GFRP). For these studies, at least, this would suggest that water  
27 absorption in CFRP and GFRP was predominantly via the resin matrix, while there is some additional  
28 water absorption mechanism for the BFRP. As it is expected that the fibres would not absorb a  
29 significant amount of water (, this additional mechanism may be due to capillary action at the fibre-  
30 matrix interface, which can be damaged by swelling of the matrix [7]. Higher temperatures exacerbate  
31 this behaviour, which would allow more water ingress and increased saturation levels.

32 |The effect of the additional water absorption was clearly visible in the aged mechanical performance  
33 of the UD-CFRP and UD-BFRP, as shown in Figure 13, which shows the 0° tensile strength and modulus  
34 plotted in relation to their weight gain due to water absorption. Based on the linear fits presented in  
35 Figure 13, UD-CFRP showed a reduction of 1.62% in tensile modulus and 8.79% tensile strength per 1  
36 % weight gain, whereas basalt fibre samples displayed a greater dependency on water ingress, with  
37 reductions of 20.2% in tensile modulus and 24.5% in tensile strength per 1 % weight gain. Note that  
38 the individual data points are given in Table 7 and Table 8.

39 Davies and Verbouwe [15] tested their aged specimens in quasi-static flexure and cyclic flexure. The  
40 quasi-static tests, relevant to this study, showed comparatively less influence of water ageing on  
41 flexural modulus (approx. 5% reduction), but a very similar influence on flexural strength (approx.  
42 24.6% reduction). They also noted that their GFRP were less sensitive to water ageing.

43 The increased water ageing of the BFRP was most likely coupled to the increased water absorption,  
44 and was also likely a result of damage accumulating at the fibre-matrix interface. This suggests that  
45 improved fibre sizings must be developed for BFRP to better retain its mechanical properties in  
46 immersed conditions.





1  
2 **Figure 13: Mechanical properties of powder-epoxy based UD-CFRP and UD-BFRP in relation to their**  
3 **composite weight gain ( $W_c$ ) due to water absorption.**

4

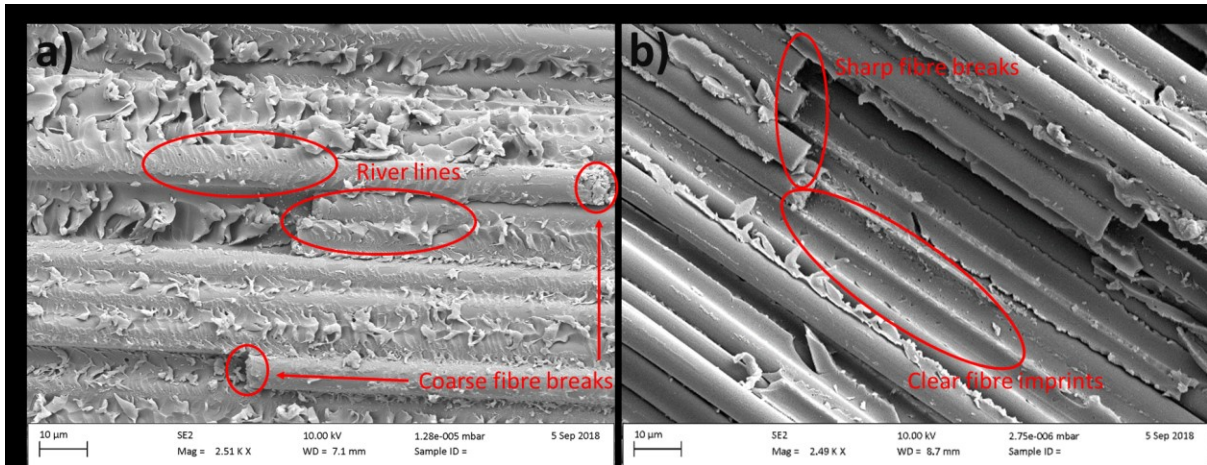
5 **Table 7: The influence of water ageing on the 0° tensile properties of UD-CFRP.**

| $W_c$ (%) | $\sigma_M$ (MPa) | SD (MPa) | $E$ (GPa) | SD (GPa) |
|-----------|------------------|----------|-----------|----------|
| 0.00      | 2311             | 120.2    | 122.6     | 0.5      |
| 0.55      | 2187             | 157.1    | 121.0     | 2.4      |
| 0.87      | 2164             | 112.1    | 121.0     | 1.7      |
| 1.23      | 2045             | 128.0    | 120.8     | 2.5      |

6

7 **Table 8: The influence of water ageing on the 0° tensile properties of UD-BFRP.**

| $W_c$ (%) | $\sigma_M$ (MPa) | SD (MPa) | $E$ (GPa) | SD (GPa) |
|-----------|------------------|----------|-----------|----------|
| 0.00      | 1186             | 74.3     | 42.9      | 0.9      |
| 0.92      | 1080             | 64.9     | 33.3      | 2.0      |
| 1.56      | 737              | 29.9     | 32.0      | 0.5      |



1  
2 **Figure 14: SEM fractographic photos of UD-CFRP epoxy samples a) dry and b) hygrothermally aged**  
3 **at 21°C for 1600 hours.**

4 The fracture comparison between dry and hygrothermally aged (21°C) UD-CFRP samples highlights  
5 two different failure mechanism. The dry sample fracture reveals coarse fibre fractures with multiple  
6 fragmentation. One can also observe the presence of river lines on both the fibres and the fibre  
7 imprints. This fracture behavior is typical of good fibre/matrix interface cohesion, leading to a brittle  
8 and explosive failure. The presence of water in Figure 14(b) discloses a very different fracture  
9 behavior. Sharp, clean fibre breaks and clear fibre imprints are witness to a poorer energy transfer at  
10 the interface during breakage, when compared to the dry case of Figure 14 (a). The presence of water,  
11 plasticizing and swelling the resin, likely caused a poorer interfacial cohesion which resulted in a more  
12 ductile and progressive failure.

13  
14 **6. Conclusions**

15 This study investigated a novel powder-epoxy towpregging line for manufacturing unidirectional  
16 carbon fibre reinforced polymers (UD-CFRP) and unidirectional basalt fibre reinforced polymers (UD-  
17 BFRP) as alternative material systems for ocean energy sub-structures such as wind and tidal turbine  
18 blade spars. One type of carbon fibre (CF) towpreg and two types of basalt fibre (BF) towpregs were  
19 produced; with powder-epoxy being used as the resin matrix in all cases.

20 Due to the processing characteristics of the powder-epoxy, it was possible to manufacture towpreg at  
21 120°C without inducing any significant curing. Furthermore, once cooled below approx. 40°C (i.e. its  
22 glass transition temperature), it was possible to store the towpreg under ambient conditions with no  
23 risk of out-time effects.

24 Electrical joule heating was used during towpregging to sinter/melt the powder-epoxy onto the carbon  
25 fibres, while IR heating was used for basalt fibres. Both heating methods were successful in producing  
26 towpreg, however, it was found that electrical heating of CF towpreg resulted in a more homogeneous  
27 heating profile.

28 Electrostatic spraying was found to be an efficient method of powder deposition, however, a  
29 consistent deposition rate and stable line tension were found to be important for maintaining a  
30 uniform towpreg fibre volume fraction (FVF). Tensile tests (0° direction) on UD-CFRP specimens  
31 showed that strength and modulus increased with FVF up to 57.5%, but then reduced due to  
32 insufficient laminate processing pressure (i.e. vacuum-bag-only), resulting in poor consolidation at  
33 higher FVFs. It was also shown that the FVF range of CF/powder-epoxy towpreg had a negligible effect

1 on the 0° tensile properties as long as the mean FVF was at the required level. This was attributed to  
2 the low viscosity of the powder-epoxy, which allowed it to flow from resin-rich regions to resin-poor  
3 regions.

4 In all cases, test laminates were manufactured with a fibre tensioning apparatus, which improved fibre  
5 alignment and straightness, thereby improving the mechanical properties of the unidirectional  
6 composite laminates. By comparing directly to the literature, it was possible to show that the  
7 towpregging process had no adverse effect on the 0° tensile properties of the composite. Moreover,  
8 comparison with commercially available UD-CFRP material systems showed that similar or better 0°  
9 tensile properties were achievable with the CF towpreg.

10 For the two types of basalt fibre investigated, UD-BFRP manufactured with Basaltex fibres generally  
11 outperformed UD-BFRP manufactured with Mafic fibres in a series of mechanical tests; 0° & 90°  
12 tension, 0° & 90° flexure, and 0° compression. This confirmed that there was some variation between  
13 basalt fibre suppliers, however, similar variations might be expected between glass fibre suppliers or  
14 carbon fibre suppliers (e.g. Toray T700S compared to Zoltek PX35). Comparing to UD-BFRP from the  
15 literature and commercially available UD-GFRP, the two sets of UD-BFRP performed well in tension  
16 and flexure, with notably high 90° properties similar to the properties of the neat powder-epoxy.  
17 Despite this, both sets underperformed in 0° compression tests. The high 90° properties and low 0°  
18 compression properties are thought to be related to the ductility and toughness of the powder-epoxy.

19 Finally, a hygrothermal ageing study of UD-CFRP (52.5% FVF) and UD-BFRP (Basaltex, 50% FVF) showed  
20 that both composites absorbed water and aged as a result. For UD-CFRP, the weight gain (w.r.t to  
21 resin mass fraction) due to water uptake was similar to the literature for neat powder-epoxy. For UD-  
22 BFRP, however, the weight gain almost doubled (with respect to resin mass fraction) for similar ageing  
23 conditions to the CFRP. Further analysis of values reported in the literature, confirmed this trend for  
24 UD-CFRP and UD-BFRP. This suggested that there was some additional mechanism for water uptake  
25 in the UD-BFRP specimens other than absorption into the resin matrix; potentially capillary action due  
26 to damage at the fibre-matrix interface. This hypothesis was supported by a greater reduction in 0°  
27 tensile properties for UD-BFRP than UD-CFRP after immersed ageing. Similar trends were noted in the  
28 literature for UD-GFRP and UD-BFRP.

29 Overall, this study showed that the pilot towpregging line has potential for upscaling to produce UD  
30 materials for the ocean energy industry. In particular, the UD-CFRP produced in this study performed  
31 well against commercially available material systems and in immersed conditions. The results for UD-  
32 BFRP show an increasing potential for commercial use, but further development is still required to  
33 compete with GFRP or CFRP, particularly in area of immersed performance.

34

## 35 7. Future work

36 This study covered a broad range of themes; from material development and production, to  
37 mechanical testing and hygrothermal ageing. As such, exploring many aspects in detail were outside  
38 the current scope of work. Nevertheless, the results of the study open numerous possibilities for  
39 future work, some of which are presented here.

40 Firstly, this technology requires further automation before reaching an acceptable industrial level of  
41 reliability. Better process control and a deeper understanding of how each process parameter  
42 influences towpreg production can be achieved via process monitoring and process modelling. In this  
43 regard, key processes are tow tensioning, electrostatic deposition, heating, and consolidation. With

1 further development, data from tension meters, IR sensors, solid particle concentration sensors, and  
2 optical sensors could be logged in real time, and combined with process modelling to form a closed  
3 loop system which can be optimised and self-regulate.

4 The ability to produce high quality powder-epoxy towpreg would open numerous possibilities in terms  
5 of application due to its latent curing characteristics. Future projects will consider application of the  
6 towpreg in subsequent manufacturing processes. Towpreg could be used 'as-is' for filament winding,  
7 subsequently reprocessed into tape for ATP, or pultruded into net sections which can later be  
8 reformed. This latter example is significant in the context of the current work, which is aimed at  
9 developing alternative, low-cost materials for wind and tidal energy.

10

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17

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