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ALIGNED SHORT FIBRE COMPOSITES WITH NONLINEAR BEHAVIOUR

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ABSTRACT

Continuous fibre reinforced composites have high properties for structural applications but tend to fail in a brittle manner, unlike metals. However, when the fibre aspect ratio is less than the critical value and a high level of fibre alignment is obtained, discontinuous fibre composites could potentially achieve a ductile or pseudo-ductile tensile response caused by deformation and slippage at the fibre ends. A lot of modelling work on aligned discontinuous fibre composites shows nonlinear behaviour on the stress-strain curve with a limited reduction of modulus and strength. Despite the interesting results from analytical studies, there have been limited experimental results to validate the models as the required fibre length leads to difficulties in producing high-quality specimens with consistent fibre length, good alignment, controlled volume fraction and uniformly distributed fibres.

In this paper, specimens with highly aligned discontinuous fibres and with fibre length close to the critical value, which can bring a brittle-ductile transition, are manufactured with the HiPerDiF method (High Performance Discontinuous Fibre method) and tested in tension. In particular, 1 and 3 mm carbon fibres are used to manufacture composite specimens with epoxy and polypropylene matrices. The analytical solutions are compared with experimental results.

1 INTRODUCTION

Discontinuous fibre composites can achieve high structural performance when the fibre aspect ratio is sufficiently high to enable load transfer and high levels of alignment are obtained [1-4]. Furthermore, they offer the scope to create a ductile or pseudo-ductile response by deformation and slip at the discontinuities when the fibre length is less than the critical value.

There have been a lot of interesting studies on the modelling of aligned short fibre composites. Pimenta and Robinson [5] modelled aligned discontinuous fibre composites with ductile and brittle matrices, taking into account a nonlinear matrix response, stochastic distribution of fibre strengths and end-locations, and investigated the effect of fibre length on the stress-strain curves. They demonstrated that, when the fibre length was around the critical fibre length (typically 0.5~1 mm), a pseudo-ductile behaviour can be achieved with a limited reduction of modulus and strength. Hishimoto and Okabe et al. [6-8] also developed a model for an aligned discontinuous carbon fibre/polypropylene composite with a Shear-Lag Model and investigated the effect of fibre length on the stress-strain curves, which showed ductility with reasonable modulus and strength [1, 2]. Despite numerous analytical studies, there have been limited experimental results to validate the models because of manufacturing difficulties in producing high-quality specimens with consistent fibre length, good alignment, controlled volume fraction and uniformly distributed fibres. This is because most short fibre composites are manufactured by an injection moulding, compound moulding, or extrusion process. Since significant fibre breakage occurs during these manufacturing processes and the fibre orientation varies depending on the rheology characteristic of resin and geometry of the mould for composite parts, it is difficult to control the fibre

orientation and length in composites. The issue is therefore how to manufacture highly aligned discontinuous preforms directly from short fibres close to the critical value in order to validate the modelling work and achieve ductility in composites. A few decades ago, aligned short carbon epoxy composites with 0.5-4 mm fibres were manufactured by Piggot et al. with a converging flow method (ERDE) and they reported the fibre length and orientation effect on the mechanical properties [9, 10]. Also, the same authors investigated the interfacial effects in carbon/epoxy composites on the critical fibre length. Among the test results, the composites with 2 mm long silicon-coated carbon fibres showed a stress plateau region on the stress-strain curve [10]. Bonderer et al. [11] made high stiffness but ductile plastic films, using 2D assembled thin platelets and ductile polymer inspired by nacre. At the macro scale, blocked-unidirectional discontinuous carbon fibre/epoxy prepreg composites have been also used to demonstrate the pull-out mechanism under tension resulting in nonlinear behaviour [12].

However, the manufacturing method should be applicable for different fibre types with a fast and continuous process. Work at the University of Bristol has recently shown a new method having a unique fibre orientation mechanism [13], the HiPerDiF (High Performance Discontinuous Fibre) method. It uses the momentum change of fibres suspended in a low-viscosity fluid to achieve a high level of fibre alignment. It was previously noted that the tensile modulus, strength and failure strain of aligned discontinuous fibre epoxy composites were close to those of continuous fibre composites. This is because the fibres were highly aligned and their length, 3 mm, was sufficiently long compared to the critical fibre length [14, 15].

This paper reports preliminary test results on specimens with highly aligned discontinuous fibres and with fibre lengths close to the critical value, manufactured with the HiPerDiF method. In particular, chopped 1 and 3 mm carbon fibres were used to manufacture composite specimens with epoxy and polypropylene matrices, to investigate the brittle-ductile transition during tensile tests. The analytical solutions developed by Pimenta and Robinson were also compared with experimental results.

2 EXPERIMENAL METHOD

2.1 Fibre orientation method

Previously, a tow type carbon preform was successfully produced with the first prototype HiPerDiF rig [14]. The fibre orientation mechanism is simply summarised as follows. Firstly fibres are dispersed in water and the fibre suspension is supplied into the fibre orientation head, which is composed of two parallel plates, by a peristaltic pump. Subsequently the fibre suspension jet is directed into a gap between two parallel plates at an oblique angle, this aligns the fibres by a sudden momentum change of the suspension provided that the fibre length is less than the gap distance as shown in Figure 1(a). The fibres fall onto a conveyor mesh belt where the alignment is finalised, creating a dry fibre preform. Meanwhile, the water is removed by a vacuum suction line underneath the mesh belt.
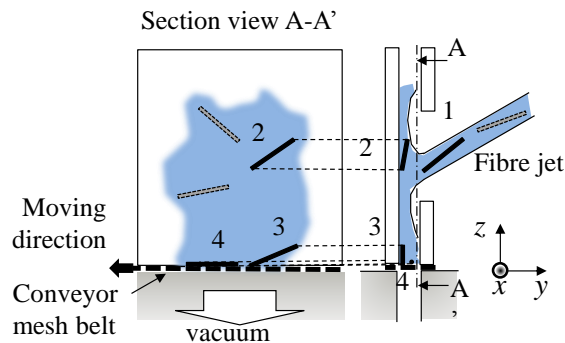

Figure 1: Short fibre orientation mechanism of HiPerDiF method.

In this paper, a new rig was built in order to produce a wider tape type carbon preform as well as to increase the production rate. As illustrated in Figure 2(a), the concept of the fibre orientation single unit, as described above, allows extending the fibre orientation head size along the direction perpendicular to

the channel (y-direction). The new HiPerDiF rig has a capacity of using 9 nozzles and the gap distance between each channel can be individually controlled by a spacer as shown in Figure 2(b). The fibres are aligned along the x-direction by falling on the conveyor belt as shown in Figure 2(c).
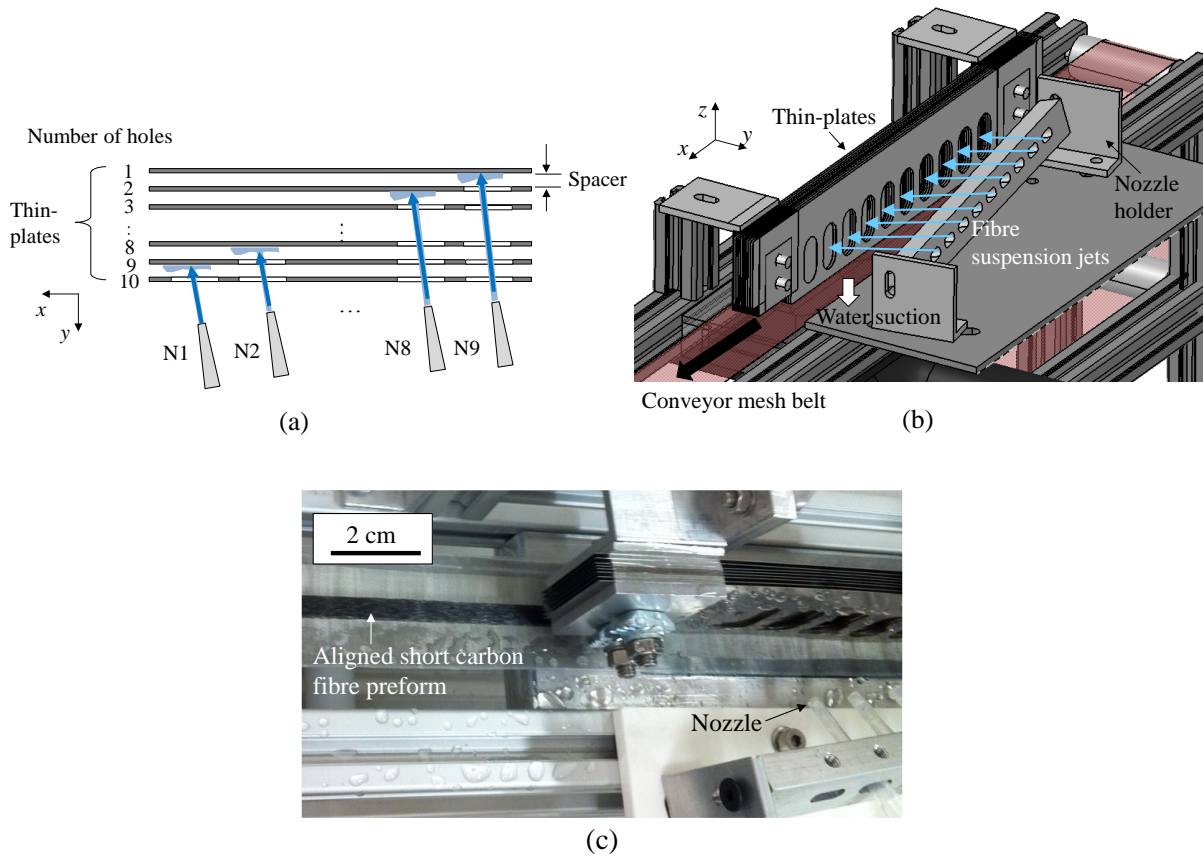

(a)

(b)

(c)

Figure 2: (a) Extended fibre orientation head, (b) Schematic diagram of fibre orientation head of scaled-up HiPerDiF rig, (c) Aligned short carbon preform from the HiPerDiF rig.

2.2 Materials

The 1 mm and 3 mm chopped carbon fibres were sourced from YF international and TohoTenax, respectively and their mean fibre diameter is 7 μm. The epoxy resin film was MTM49-3 from Cytec. The polypropylene (PP) film with 20 μm thickness was provided by Propex Fabrics GmbH (Germany). The melting point of the PP film is 163°C. The fibre and matrix properties from the manufacturers are listed in Table 1.

	Tensile modulus [GPa]	Tensile strength [MPa]	Density [kg/m³]	Sizing
1 mm carbon fibre	230	3500	1800	Unsized
3 mm carbon fibre	225	4344	1820	Water soluble polymer
	Tensile modulus [GPa]	Tensile strength [MPa]	IFSS** [MPa]	Density [kg/m³]
Epoxy	4*	80*	60	1220
PP	1.45	35	5	920

* Estimated values according to [16, 17], ** Estimated values according to [6, 18, 19]

Table 1: Carbon fibre and matrix properties.

2.3 Sample preparation

In this paper 1 mm chopped carbon fibre reinforced epoxy composites with different gap distances

(0.15 mm, 0.5 mm) and 3 mm chopped carbon fibre reinforced PP composites were manufactured. The carbon fibre length for each matrix system was selected depending on the matrix properties. The epoxy matrix has a good adhesion with carbon fibres even though they are unsized, therefore the value of 1 mm can be assumed to be close to the critical length of carbon/epoxy composites [5]. Meanwhile, it is well known that the interfacial shear strength (IFSS) between carbon fibres and polypropylene is relatively low compared to the IFSS between carbon fibres and epoxy resin. The typical IFSS of the carbon/PP composites is less than 10 MPa, which results in a few millimetres of critical length, as shown in Equation (1) below,

$$l_c = \frac{\sigma}{2\tau_i} d \qquad (1)$$

where σ is the strength of fibre and d is the fibre diameter, and τ_i is the IFSS. Therefore a chopped carbon fibre length of 3 mm was chosen for carbon/PP composites. The gap distance between the parallel plates in the HiPerDiF method, considered a key factor to affect the fibre alignment level, was chosen as a sixth of the fibre length. A gap distance of a half of the fibre length was also used to manufacture 1 mm carbon/epoxy samples. The manufacturing parameters and estimated areal density of the aligned short fibre preforms are summarised in Table 2.

	1CE/0.15Gap	1CE/0.5 Gap	3CP/0.5 Gap
Fibre length [mm] (± 1SD)	1 ± 0.1	1 ± 0.1	3 ± n.a.
Gap distance [mm]	0.15	0.5	0.5
Areal density of fibre preforms [gsm]	46.0	47.4	67.0

Table 2: Manufacturing parameters and estimated areal density of fibre preforms.

To manufacture the prepreg, the preform with 1 mm carbon fibres was placed on the epoxy resin film and pressure and heat (60°C) were applied. The preforms with 3 mm fibres were stacked together with the PP films on both sides and, in a similar way, compression and heat (170°C) were applied. Four plies of prepreg were used for each specimen. The stacked prepregs were placed in a semi-closed steel mould. The epoxy specimens were cured at 135°C for 135 minutes and the PP specimens were hot compacted at 185°C for 345 minutes in an autoclave using vacuum bag moulding both at a pressure of 6 bar. Since the carbon/PP prepreg is not a fully impregnated product, the duration time under the maximum temperature needed to be long to ensure that the PP penetrates into the carbon preforms sufficiently. After manufacturing, burrs at all edges along the fibre direction were gently removed using sand paper.

2.4 Tensile tests

Tensile tests were performed on an electro-mechanical testing machine with a cross-head displacement speed of 1 mm/min. The load was measured with a 10 kN load cell (Shimadzu, Japan) and the strain was measured with a video extensometer (IMETRUM, UK). White speckles were spray-painted on the black background of the specimens to allow the strain measurement with the video extensometer. The gauge length for the strain measurement was around 50 mm. Glass fibre/epoxy end tabs were attached using epoxy adhesives (Aladite2014, Huntsman) for all the specimens. The specimen dimensions are shown in Figure 3.
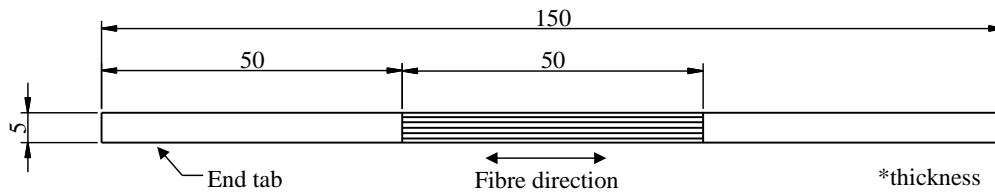
*0.27 mm for 1 mm carbon/epoxy specimen, 0.37 mm for carbon/PP specimen
Figure 3: Specimen dimension for tensile test [mm].

2.5 Resin burn off

Resin burn off tests were performed according to ASTM D3171 [20]. The samples were inserted into a furnace for 5 hours at 500°C under a fume cupboard. The samples were weighed before and after resin burn off with a resolution of 0.1 mg. The fibre volume fraction was calculated based on the sample weights and the density of carbon fibres, epoxy, and PP as listed in Table 1.

3 RESULTS AND DISCUSSION

3.1 Carbon/Epoxy composites

The tensile properties of the 1 mm carbon/epoxy specimens are summarised in Table 3. The tensile stress-strain curves for all composites were almost straight as shown in Figure 4(a). This is because the mean fibre length is just around the critical length for the carbon/epoxy composites. However, they gave a small nonlinearity just before the failure as shown in Figure 4(b). Since 1 mm is the mean chopped length and the fibre length happens to be shortened during the manufacturing process, the length of a few carbon fibres might be below the critical value. This is demonstrated by some pulled-out fibres on the fractured surface of the composite specimens, as shown in Figure 5(a).

	Tensile modulus [GPa] (0.4-0.7%) 1SD (CV)	Tensile strength [MPa] 1SD (CV)	Strain to failure [%] 1SD (CV)
1CE/0.15Gap	36.1 ± 0.591 (1.64%)	345 ± 15.1 (4.37%)	0.984 ± 0.0608 (6.18%)
1CE/0.5Gap	35.5 ± 0.591 (6.57%)	348 ± 42.9 (12.3%)	1.01 ± 0.0864 (8.56%)

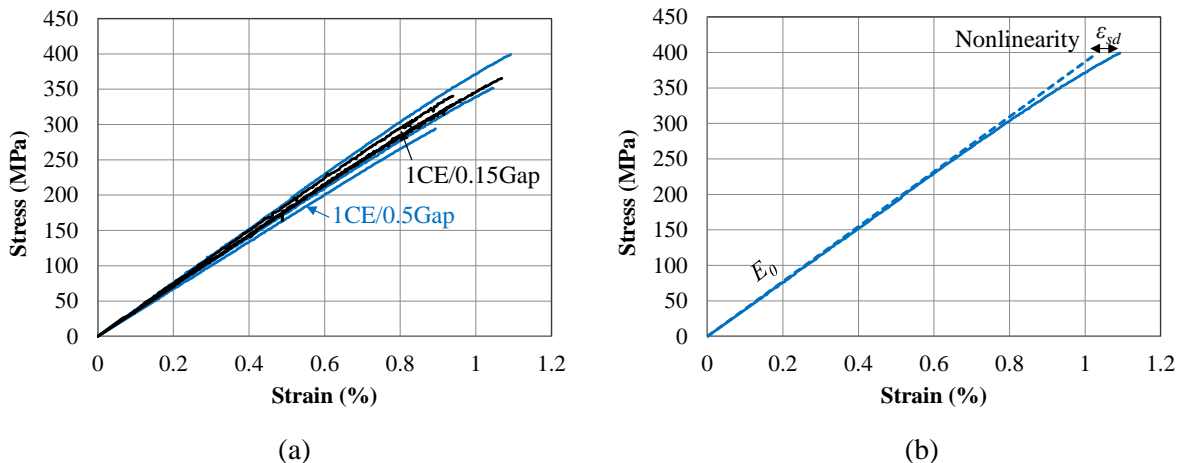Table 3: Tensile test results of carbon/epoxy composites with 1 mm fibres (v_f = 20-25%).

Figure 4: (a) Tensile stress-strain curves for aligned 1 mm carbon/epoxy composites, (b) Representative stress-strain curve showing nonlinearity before failure.

According to the test results, there is no significant difference between the carbon/epoxy composites made with 0.15 mm and 0.5 mm gap distance. This means that the gap distance would not affect the fibre alignment level significantly given that it is less than a half of the fibre length. However, more experimental data is needed to investigate the gap distance effect on the fibre alignment level in the future.
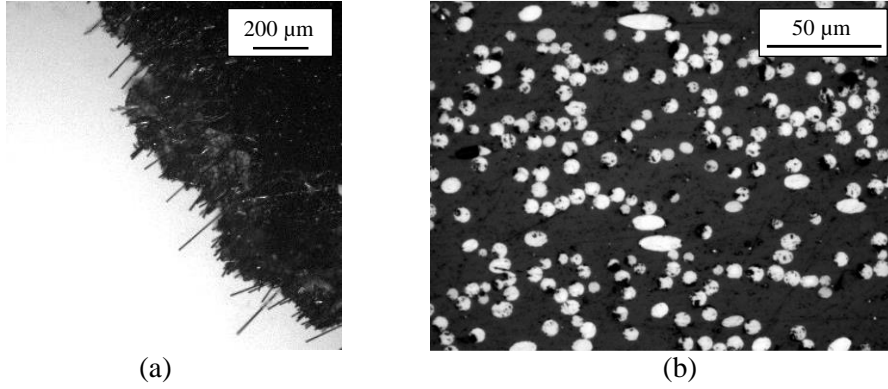
(a)   (b)

Figure 5: (a) A few pulled-out fibres on the fractured surface and (b) microscope cross-section image of aligned 1 mm carbon/epoxy composites.

The fibre volume fraction measured by the resin burn off method was around 20%. However, it seemed to be underestimated because these short fibres may be easily blown away by the forced air circulation in the furnace and fibre mass loss can occur under high temperature [21]. The fibre volume fraction may vary but it seemed to be around 20-25% from the cross sectional image as shown in Figure 5(b). In consideration of the fibre volume fraction, the tensile modulus, 36 GPa, and strength, 345 MPa, were reasonably high compared to the mechanical properties of short fibre composites manufactured by high fibre volume fraction injection moulding or the modified extrusion method [22-24]. Also, they are comparable with the aligned short fibre composites by the conventional fibre alignment methods, e.g. the converging flow method by ERDE [9] as summarized in Table 4.

Material	Tensile modulus [GPa]	Tensile strength [MPa]	Fibre v_f [%]	Fibre length [mm]	Manufacturing method
HTS/Epoxy	36	345	20-25	1	HiPerDiF method
Carbon/PEEK [23]	12	216	24	0.1	Injection moulding
Carbon/PEEK [24]	22	300	24	0.1	Injection moulding
AS4/PEEK [22]	38	113	60	0.1	Modified extrusion method
AS2/Epoxy [9]	25	220	15	1	Converging flow method
AS2/Epoxy [9]	42	380	28	1	Converging flow method

Table 4: Mechanical properties of short fibre composites.

3.2 Carbon/Polypropylene composites

The tensile stress-strain curves for the 3 mm carbon/PP composites show nonlinear behaviour as seen in Figure 6(a). The pseudo-ductile strain, ε_{sd}, was measured based on the definition suggested by [25] as the difference between ε_f and the elastic strain, ε_{E0}, at the same stress based on the initial modulus, E_0, as illustrated in Figure 6(b). The tensile test results are listed in Table 5. As expected, 3 mm is very close to or less than the critical fibre length of the carbon/PP composites due to the poor adhesion characteristics. Therefore, a nonlinear stress-strain curve was obtained although the pseudo-ductile strain value was not high. It was also observed that most of the fibres were pulled out on the fracture surface as shown in Figure 7(a).

	Tensile modulus, E_0 [GPa] (0.1-0.3%) 1SD (CV)	Tensile strength, σ_U [MPa] 1SD (CV)	Strain to fail, ε_f [%] 1SD (CV)	Pseudo-ductile strain, ε_{sd} [%] 1SD (CV)
3CE/0.5Gap	57.1 ± 2.43 (4.25%)	401 ± 18.5 (4.63%)	0.945 ± 0.0380 (4.02%)	0.243 ± 0.0423 (17.4%)

Table 5: Tensile test results of carbon/PP composites with 3 mm fibres ($v_f = 36\%$).

(a)   (b)
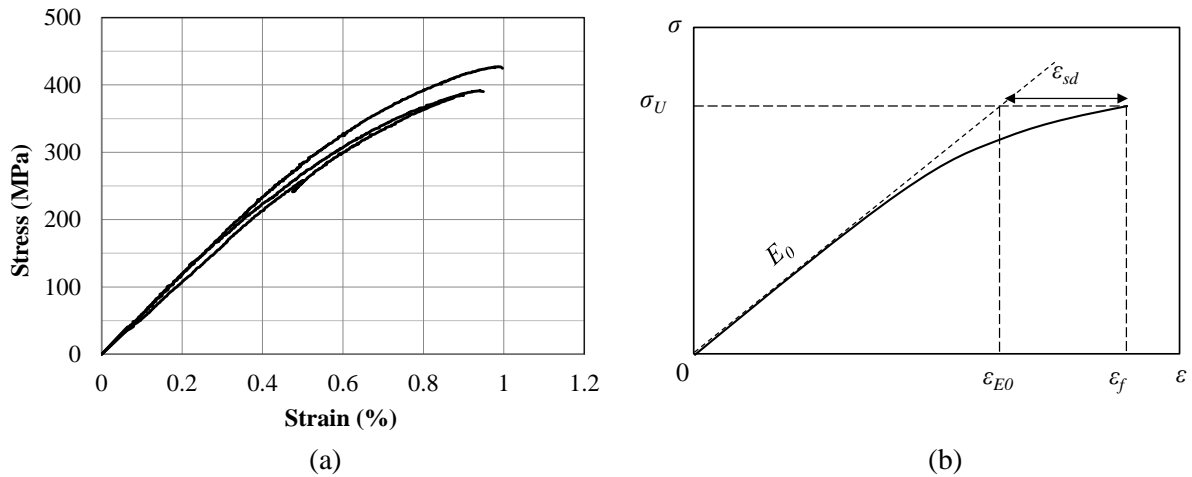
Figure 6: (a) Tensile stress-strain curves for aligned 3 mm carbon/PP composites, (b) Definition of pseudo-ductile strain for composites.

The fibre volume fraction measured by the resin burn off method was around 36%. Figure 7(b) shows an image of the composite cross-section, giving the evidence of the high fibre alignment level. The initial tensile modulus of carbon/PP with 3 mm fibres was 57.1 GPa which shows a 28% reduction compared to the modulus of continuous carbon/PP composites simply calculated by the Rule of Mixtures using continuous carbon fibre and matrix properties. The percentage modulus reduction of the aligned carbon/PP composites with 3 mm fibres is higher than that of the carbon/epoxy composites with the same fibre type and length [14]. This is probably because the PP matrix tends to shrink after the hot compaction process, causing misalignment of the short carbon fibres and a decrease in the composite tensile modulus. Despite the mechanical performance reduction, the aligned 3 mm carbon/PP composites definitely show much higher structural performance compared with other short carbon/PP composite materials [26].

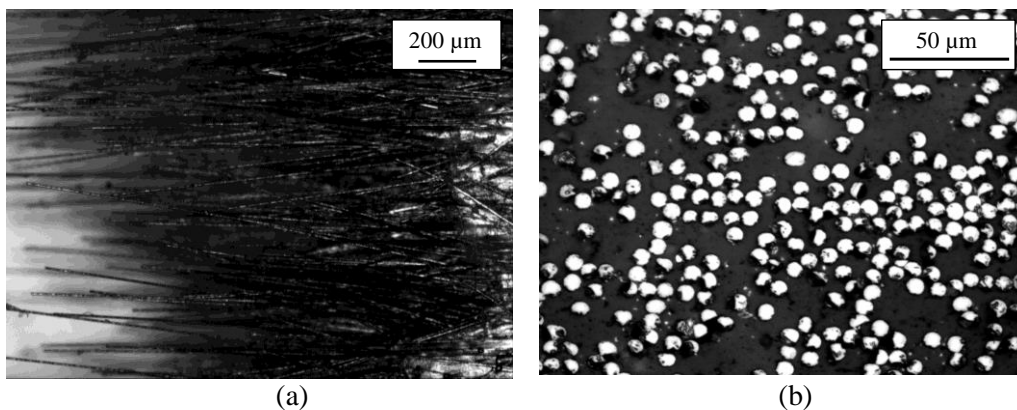(a)   (b)

Figure 7: (a) Pulled-out fibres on fractured surface and (b) microscope cross-section image of aligned 3 mm carbon/PP composites.

3.3 Comparison of experimental and modelling results

Figure 8 compares the experimental stress-strain curves of the aligned discontinuous composites with modelling curves obtained using Pimenta and Robinson's model [5]. For the 1 mm carbon/epoxy composites (Figure 8(a)), the model predicts higher strength and failure strain than those obtained experimentally; it is unclear whether this mismatch is due to fibre misalignments and premature failure in the experiments, or due to an overestimation of the strength in the model. Nevertheless, it is interesting to notice that the experiments and modelling agree up to the onset of nonlinearity (and, consequently, of significant damage accumulation) predicted by the model, which correlates extremely well with the moment of final failure observed experimentally.

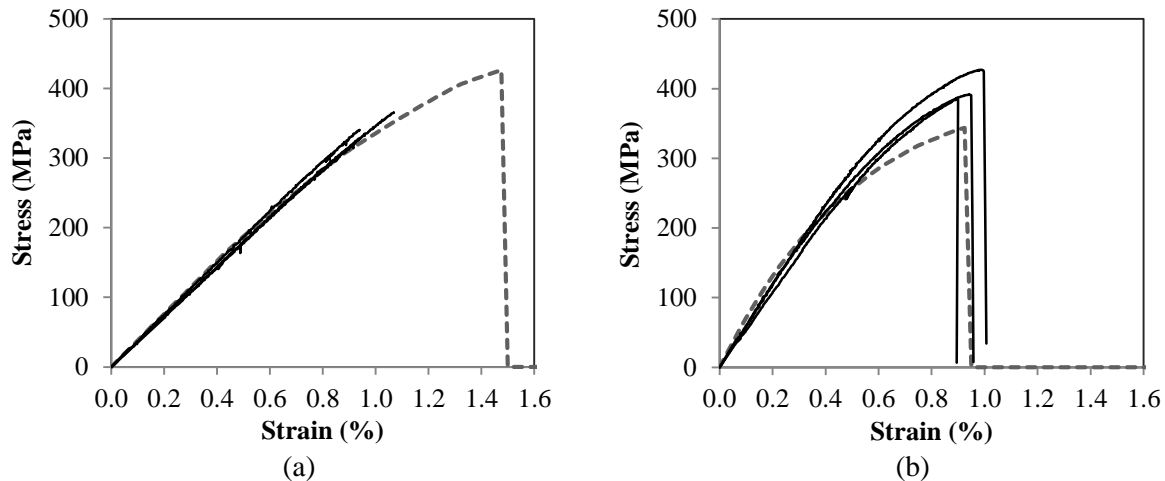(a)   (b)

Figure 8: Comparison between experimental (full lines) and modelling (dashed lines) stress-strain curves for aligned discontinuous composites; (a) 1 mm carbon/epoxy composites, (b) 3 mm carbon/PP composites.

In addition to predicting stress-strain curves, the model can also be used to explain the failure mechanisms of aligned short fibre composites. For the 1 mm carbon/epoxy composite, the model predicts that only 13% of the carbon fibres in the cross-section actually fail, which justifies the considerable amount of pull-out in Figure 5(a). This also reveals that deterministic estimations of the critical length (e.g. Equation (1)) are not reliable to predict the failure mode of aligned short fibre composites, due to the random location of fibre ends and size effects on fibre strength variability.

For the 3 mm carbon/PP composite (Figure 8(b)), the modelling and experimental curves show a similar nonlinear response. The model predicts a progressive loss of tangent stiffness with earlier onset and larger magnitude than observed experimentally; this could be due to an underestimation of the interfacial shear strength between the carbon fibres and the PP matrix, for which a large range of values exists in the literature. The model also predicts that, for the 3 mm carbon/PP composite, only 0.1% of the fibres actually break during the loading; this is corroborated by the large amount of pull-out observed on the experimental fracture surfaces, as shown in Figure 7(a).

4   CONCLUSIONS

Aligned 1 mm carbon/epoxy composites and 3 mm carbon/PP composites were successfully produced with the HiPerDiF method and tested in tension.

- The tensile stress-strain curves for the 1 mm carbon/epoxy composites were almost straight because 1 mm is still higher than the critical value. In consideration of the fibre volume fraction of 20-25%, the tensile modulus, 36 GPa, and strength, 345 MPa, were reasonably high compared to the mechanical properties of commercially available short fibre composites.

- Nonlinear stress-strain curves were obtained from the 3 mm carbon/PP composites with the fibre pull-out mechanism visible on the fracture surface. The measured fibre volume fraction was around 36%. The initial tensile modulus of carbon/PP with 3 mm fibres was 57.1 GPa which shows a 28% reduction from the continuous carbon/PP composites, probably because the high shrinkage of the PP matrix after the hot compaction process affects the fibre alignment level.

- The experimental stress-strain curves of the aligned discontinuous composites were compared with modelling curves obtained using Pimenta and Robinson's model. For the 1 mm carbon/epoxy composites, the experiments and modelling agree up to the onset of nonlinearity predicted by the model, which correlates extremely well with the moment of final failure observed experimentally. For the 3 mm carbon/PP composite, the modelling and experimental curves show a similar nonlinear response. The model predicts a progressive loss of tangent stiffness with earlier onset and larger magnitude than observed experimentally; this could be

due to an underestimation of the interfacial shear strength between the carbon fibres and the PP matrix. However, this is a good starting point to validate the models for aligned short fibre composites with nonlinear behaviour but intermediate structural performance.

ACKNOWLEDGEMENTS

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