

The application of composite through-thickness assessment to additively manufactured structures

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Abstract

This study looks into the applicability of through-thickness assessment to additive manufacturing (AM) carbon-fibre reinforced polymers (CFRPs). The study utilised a material extrusion printer that uses fused filament fabrication and composite filament fabrication technologies to manufacture functionally-graded polymer and composite polymer parts. The matrix material of choice was nylon 6. Samples were printed exploring a range of reinforcement volume content. In summary, this study presents an assessment of the applicability of through-thickness testing to AM CFRP specimens and provides a performance comparison between AM composite through-thickness properties and the properties of equivalent CM CFRP specimens.

Keywords

carbon fibre, through thickness, composite, mechanical behaviour, additive manufacture

1. Introduction

1.1. Composite materials

Composite materials are non-homogenous materials made from two or more constituent materials that differ in their mechanical and chemical properties and provide desirable tailored characteristics when combined. Fibre-reinforced polymers (FRPs) are composite materials made of a polymer matrix, which houses, supports and transfers the bearing load onto a fibre reinforcement that acts as the load-bearing structure in the composite. The use and manufacture of FRPs has been widespread since the 1930s, with the mass production of glass fibres discovered in 1933 [1] and they are today commonly used in industries that include aerospace, automotive, construction and defence [2].

1.2. Carbon fibre reinforced polymers

Carbon fibre reinforced polymers (CFRPs) use carbon-based (usually graphite) fibres of 5 μm to 10 μm in diameter, twisted into yarns that are woven or embedded into a polymer matrix [3]. These fibres provide good tensile properties with respect to their mass, making them useful for a large number of applications, including aerospace, marine and automotive parts, as well as end-user products, such as golf clubs and fishing rods.

1.3. Through-thickness testing of CFRPs

The nature of CFRPs and other matrix-based composites, where fibre reinforcement is applied in a plane-wise fashion, that is, in a layer-by-layer format, means that CFRP structures exhibit anisotropic properties [4]. This is primarily due to the relatively low structural strength of the matrix (polymer) material of the composite, as well as, in some cases, a relatively weak

fibre-matrix interface between layers, which may constitute a bottle-neck in the transverse (Z-direction) structural strength of a CFRP structure [2].

Through-thickness testing (also known as Z-direction tensile testing) is a mechanical property assessment method that applies a tensile force to a test specimen whose axis is perpendicular to the reinforcement plane of a composite material (Figure 1) [5]. The main purpose of through-thickness testing is to assess the resistance to lamellar tearing [6], a form of (initially microscopic) cracks that form parallel to the plate surface of a material and induce weaknesses in the material's through-thickness (Z) direction. The process of through-thickness testing is well-understood in the polymer composite world, and specification standards have been developed for testing matrix composite materials in the through-thickness direction [5]. Several studies have been conducted on through-thickness performance testing of matrix composites [7][8][9] and some introduced additional Z-direction reinforcements before testing [10]. However, these studies focus on the conventionally processed composites, but no literature is available on through-thickness testing of additively-manufactured (AM) composites.

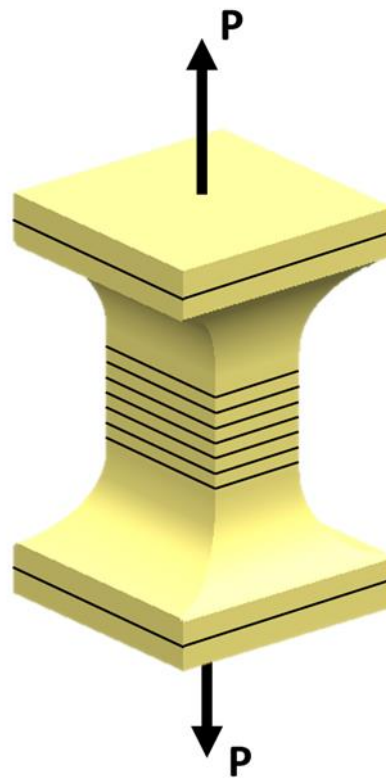


Figure 1: Through-thickness tensile test specimen

This study will aim to shed light on the applicability of through-thickness testing on AM parts as well as to evaluate their performance. This is of particular importance since AM is the latest process for manufacturing complex, non-homogeneous, micro-structured parts such as CFRPs. Current composite manufacturing processes, including thermoplastic compression moulding, filament winding, resin infusion, etc., offer various features, advantages and disadvantages, which provide variety in approaches and the potential to optimise production according to a balance of needs (e.g. size, quality, production rate, tooling costs). AM is the latest composite manufacturing process to be added to the list, which comes with its own design opportunities and challenges. Therefore, it is important to establish AM's

properties by applying relevant test methods to specimens produced by it, adding to the body of knowledge on the different properties that this new composite manufacturing process can bring forth.

1.4. CFRPs in AM

While conventional manufacturing techniques of CFRPs have been well-studied, the long cycle times and high production costs associated with their production have given AM a distinct potential advantage over other manufacturing processes, because AM carries the potential for both reducing time and cost of production as well as increasing design complexity of the end products [11].

The two main AM techniques that have been studied for the production of CFRPs are powder bed fusion, e.g. selective laser sintering (SLS) [12][13], and material extrusion, e.g. fused deposition modelling (FDM) [14][15] – also known as fused filament fabrication – with the majority of research into polymer composites falling into the latter method [16][17][18].

1.5. Material extrusion of CFRPs

Material extrusion (ME) (Figure 2) is an AM process that makes use of polymer filament feedstocks by extruding the material in its melted form, through a computer-controlled liquefier head, into a build platform in a layer by layer fashion. Advanced ME machines can support multiple extrusion nozzles with multiple materials. For example, in this case, a polymer matrix material and a separate fibre reinforcement material. Several studies have tested ME composites compared to composites manufactured using conventional methods and have shown promising results in tensile strength [15][16]. While several studies into the tensile properties of AM composite parts have been conducted [14][15][16], these studies focused on the longitudinal (in-plane) tensile properties, rather than the transverse (through-thickness) tensile strength.

2. Materials and methods

2.1. Manufacturing equipment and parameters

The experiments utilise an in-house Markforged Mark Two material extrusion machine for manufacturing the test specimens. The machine uses fused filament fabrication (FFF) and composite filament fabrication (CFF) technologies to manufacture functional polymer and composite polymer parts [19]. The machine is capable of printing four types of reinforcement material: carbon fibre, fibreglass, Kevlar and high strength, high temperature (HSHT) fibreglass, in addition to two choices of matrix material: nylon and the Markforged Onyx [20].

Test specimens were manufactured from nylon 6 as a matrix material and carbon fibre as reinforcement. All materials used were Markforged-certified materials and were obtained from CREAT3D Limited, a certified local distributor.

The print bed was levelled and print nozzle heights adjusted prior to the first print. The print bed was coated with a layer of Elmer's washable school glue in order to protect the bed, facilitate part removal and prevent the part from moving while the print is in progress. A 6 mm-wide nylon brim was printed under each specimen in order to increase the contact area with the print bed and prevent thermal curling. The nylon and carbon fibre nozzle temperatures were set to 275 °C and 245 °C, respectively.

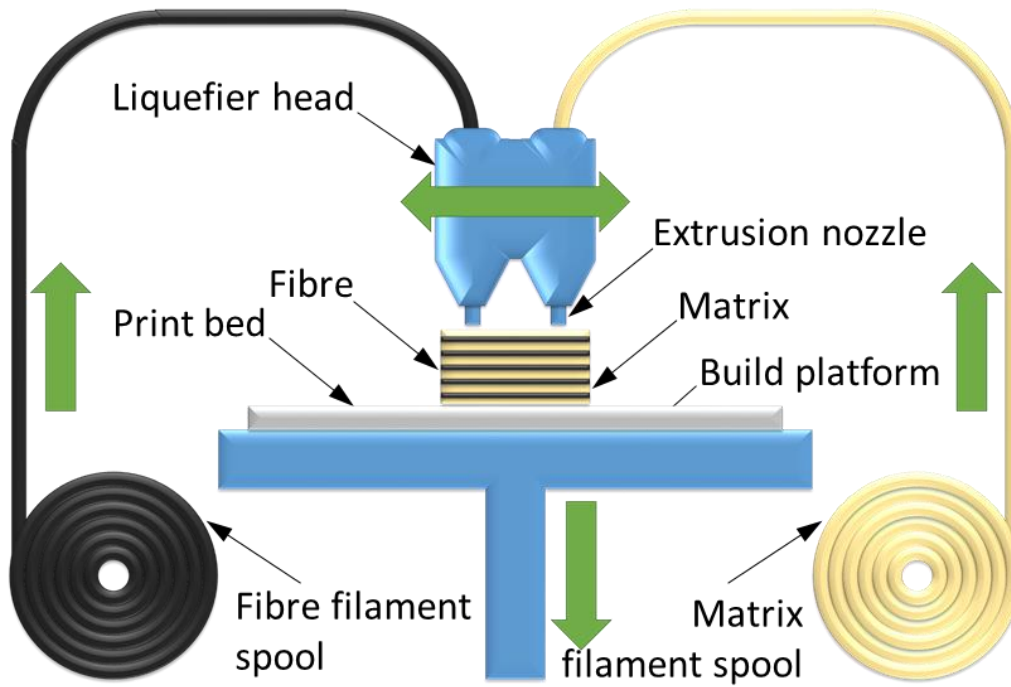


Figure 2: Schematic of the material extrusion process.

Nylon support structures were used during the printing process, which were later removed through manual prying with pliers. A purge line was printed prior to each print in order to purge the print head and extruder from any nylon that may have accumulated moisture from the atmosphere during idle times. The print head was also purged of nylon residue that may have accumulated on the nylon nozzle while carbon fibre was routed through the part. All specimens were printed with 100% fill density with a rectangular fill pattern to ensure the maximum fill density as per manufacturer recommendations. Layer height was fixed by the manufacturer at 0.125 mm. The specimens were built five at a time in the spacing shown in Figure 3.

2.2. Specimen design

All test specimens were manufactured to comply with ASTM D7291/D7291M-15 standards on through-thickness tensile testing [5]. The specimens were manufactured in accordance with recommendations from the National Physical Laboratory (NPL) [21], as shown in Figure 4. The test piece is a standard Royal Armament Research and Development Establishment (RARDE) specimen with 25 mm square ends and a 40 mm nominal height. The reduced gauge section is 12 mm long and has a rectangular cross-section of 10 mm \times 16 mm. The reinforcement was applied in the x - y plane (perpendicular to the height of the specimen) and was applied uniformly along the entire height of the specimen.

The reinforcement in each layer was laid out in a concentric fashion, where carbon-fibre rings ran continuously from the perimeter to the centre (Figure 5). This amounted to twelve rings in base layers and three rings in middle layers.

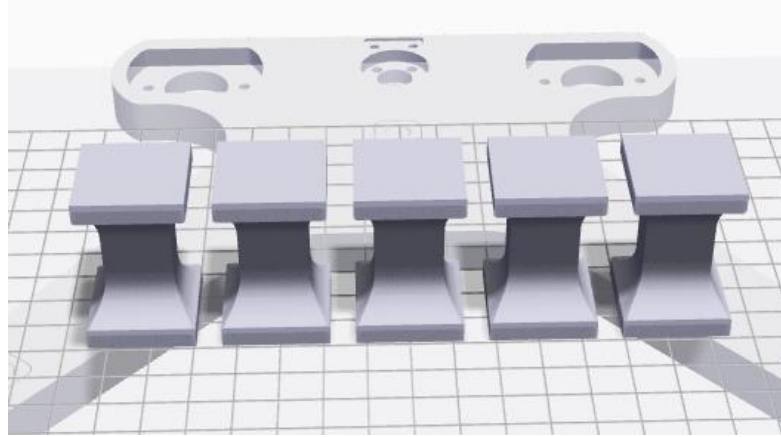


Figure 3: Specimen spacing in each print

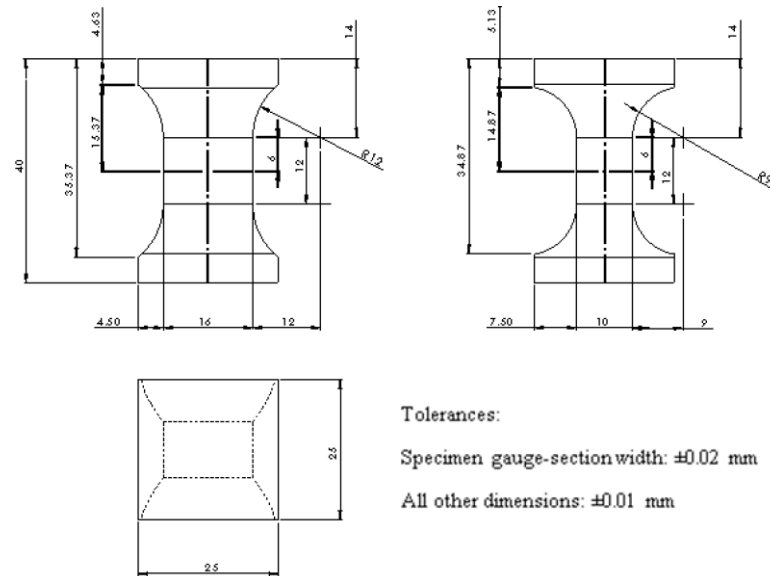


Figure 4: Schematic of through-thickness tensile CFRP specimen in accordance with ASTM D7291/D7291M-15 standards and NPL recommendations [21]

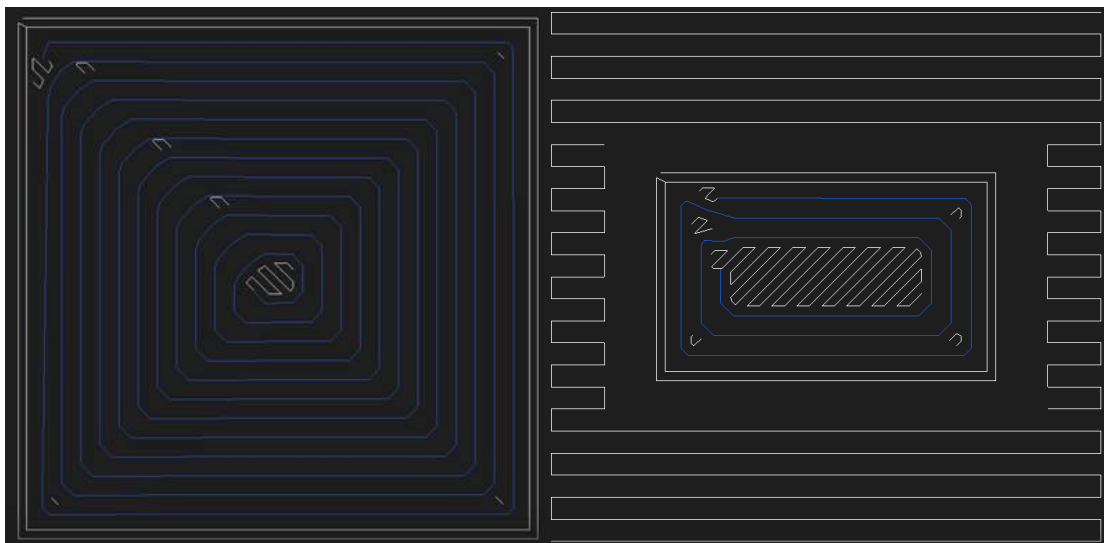


Figure 5: Per-layer layout of carbon fibre rings (blue) within nylon matrix and supports (white) at the base (right) and in the gauge section (left)

2.3. Specimen sets and experimental design

Four sample sets were produced for through-thickness tensile testing, each set consisting of five identical specimens to provide statistical significance and room for printing errors. The four sets were as follows:

1. Control samples: 100% nylon samples, no composite material deposited. These were used to benchmark the rest of the samples and provide a comparative baseline.
2. 25% fibre volume fraction (fvf) of carbon fibre (one layer of fibre in every three layers of nylon), evenly distributed along the through height of the specimen, i.e. homogeneously reinforced.
3. Half-density samples: approx. 37% fvf of carbon fibre to further test for correlation between fibre content and tensile strength.
4. Full-density samples: i.e. composite reinforcement present in print every layer, which amounts to approximately 78% fvf of carbon fibre for maximum possible fibre density.

2.4. Sample preparation

Support structures were removed from the printed samples using pliers. The test samples were then measured to check machine dimensional accuracy and prepared for tensile testing in accordance with ASTM D7291/D7291M-15 standards. As part of the preparation, a number of end tabs were machined from 6082 aluminium alloy bars using a XYZ Proturn SLX 425 lathe. End tabs and specimens were later grit-blasted using 150 μm to 300 μm glass beads in order to provide a suitable surface for adhesive bonding.

A number of suitable adhesives were tested to determine the best choice for bonding the samples to end tabs. The Loctite 410 instant adhesive was chosen. Specimens were bonded to the aluminium alloy end tabs using metal jigs to align the parts. Specimens were then left to cure for a minimum of twenty-four hours prior to testing.

Following curing, specimens were sprayed with a black and white texture in order to allow for the video strain gauge to record specimen strain under tensile loading while testing (Figure 6). A total of four specimens were bonded and prepared for the first round of testing (one of each set), followed by a second round for the remaining sixteen specimens.

2.5. Tensile testing and analysis

An Instron 5965 Mechanical Test Machine was used to determine the tensile properties of the specimens. This was then to be used to perform an evaluation on through-thickness properties and ultimately the applicability of through-thickness testing to AM parts. The strain under tensile loading was measured using an iMetrum video strain gauge that tracks the displacement of a set of texture dots painted on the specimens.



Figure 6: Through-thickness tensile specimen with fitted end-tabs and texture spray paint ready for testing.

The test was conducted at 18 °C and 50% relative humidity. Due to time and resource restrictions, a test speed of 1mm/min was used as per NPL testing protocols [22] instead of the ASTM D7291/D7291M-15 0.1 mm/min.

3. Results and discussion

3.1. Manufacturability

A total of five failed and twenty successful specimens were produced during the printing process in addition to a single-sample test print. The single pure nylon sample was printed in order to ensure all parameters are set correctly and that no complications during the print process occurred. Following the successful test print, five Set 1 (0% carbon fibre) specimens were added to the print job. However, significant distortion was present on all five samples (Figure 7). The distortion observed on all five samples is believed to be the result of thermal curling induced by the rapid cooling of nylon layers as they were laid onto the unheated substrate. Adding a brim to the parts seemed to reduce thermal distortion but failed to eliminate it. It was later observed that the addition of carbon fibre to the specimens significantly increased part stiffness and dimensional integrity. As a result, the pure nylon parts were re-printed with fibre-reinforced bases and a pure nylon gauge (Figure 8). Additionally, prior to each print, the print substrate was put under running hot tap water until hot in order to reduce the thermal gap between the printer nozzle and the substrate.



Figure 7: Thermal distortion in original Set 1 specimens.



Figure 8: Set 1 (0% carbon fibre) specimen with reinforced bases.

Thermal distortion was only observed during the five-sample print - but not in the initial single-sample print - despite both being printed with pure nylon. The reason behind this is believed to be the five-fold delay in laying each consecutive layer onto the previous ones as the machine continued to build the next layers on the remaining four specimens, which allowed more time for the plastic to cool down and deform. The option to programme the machine to print the samples in succession, rather than simultaneously, was considered. However, as of the time of writing, the printer firmware was incapable of incorporating such measures.

The production of Set 2 (25% fvf) and Set 3 (37% fvf) samples was successful. Set 4 (78% CF) prints showed carbon fibre splinters protruding through the matrix throughout the specimen's height (Figure 9). Repeat prints of Set 4 specimens conducted by the manufacturer confirmed the observation. The reason for this observation is thought to be excessive fibre content, where the density of fibre in the part is too great for the printer to print successfully. This may have caused some of the printed fibre to either tangle with previously-laid fibres or to dislodge the fibre, causing splinters and possible breaks in the printed fibre. In applications where maximum density is required, it is recommended that the parts be 'striped'. That is, to print several layers of fibre in succession (usually ten) followed by a few (usually two) pure nylon layers. This is thought to mitigate the effect of fibre splintering. This has not yet been done, but it is planned for future investigation.



Figure 9: Set 4 specimen with CF splinters

Dimensional measurements were recorded for the gauge sections of each specimen using a calibrated Clarke® Electronic Digital Calliper. A total of ten repeats were taken of each measurement and the tolerances were calculated with 95% confidence. As shown in Table 1, fibre-reinforced specimens consistently showed closer values to the target dimensions of 10 mm and 16 mm of the gauge section than the non-reinforced samples. This is primarily due to the fact that carbon fibre adds stiffness to the printed parts, thus greatly reducing the effects of the various factors that may influence the dimensional integrity, such as thermal and gravitational deformations. Conversely, fibre-reinforced specimens generally exhibit larger tolerances than non-reinforced samples. This can be attributed to the higher surface roughness of fibre-reinforced specimens caused by the presence of the fibre within the matrix, thus causing the matrix to take a rougher outline along the outer surfaces.

Table 1: Dimensional measurements of through-thickness specimens.

Sample		Wide Section/mm	Narrow Section/mm
Set 1	1	15.83 ± 0.03	9.71 ± 0.03
	2	15.85 ± 0.04	9.70 ± 0.05
	3	15.76 ± 0.05	9.70 ± 0.03
	4	15.73 ± 0.03	9.68 ± 0.02
	5	15.83 ± 0.01	9.68 ± 0.03
Set 2	1	15.98 ± 0.05	9.78 ± 0.01
	2	15.97 ± 0.05	9.86 ± 0.03
	3	15.93 ± 0.03	9.80 ± 0.02
	4	15.99 ± 0.06	9.80 ± 0.05
	5	15.96 ± 0.05	9.81 ± 0.03
Set 3	1	15.99 ± 0.04	9.81 ± 0.03
	2	16.01 ± 0.06	9.83 ± 0.05
	3	15.99 ± 0.05	9.80 ± 0.01
	4	15.94 ± 0.07	9.83 ± 0.05
	5	16.01 ± 0.05	9.81 ± 0.05
Set 4	1	16.02 ± 0.05	9.88 ± 0.07
	2	15.96 ± 0.06	9.91 ± 0.04
	3	15.99 ± 0.04	9.88 ± 0.05
	4	16.02 ± 0.06	9.88 ± 0.07
	5	16.04 ± 0.03	9.89 ± 0.06

3.2. Test results

Eight out of twenty specimens failed due to premature adhesive failure. The average stress value reached at the time the adhesive failed resulting in the tensile test being terminated was $7 \text{ MPa} \pm 1.5 \text{ MPa}$. Several factors could have played part in the failure of the adhesive. Firstly, since samples were left to cure for a total of twenty-four hours prior to testing, it is plausible that the adhesive had not reached its ultimate strength and that further time should have been allowed for sufficient curing. Secondly, it is possible that the specimens may have required a coarser surface texture in order to provide a larger surface area for bonding. A good practice guide by the Composites UK Trade Association on the adhesive bonding of composites shows that medium grit ($120 \text{ }\mu\text{m}$ to $200 \text{ }\mu\text{m}$) is ideal for composite surface pre-treatment. The guide further recommends that cryo-blast and soda-blast be used in treating composites, as opposed to shot-blasting, which is more suitable for metals [23]. Indeed, upon touch, little difference in surface roughness was observed following the grit-blasting of the specimens than the original 3D-printed surface roughness. A more suitable grit will be considered for future trials. Finally, nylon is notoriously difficult to bond due to the presence of slip additives within the material, which can migrate to the surface of the material and interfere with proper bonding. As a result, it may have been difficult for the adhesive to properly bond to the nylon surface, thus causing premature delamination in the glue and nylon interface.

Successful specimens were as follows:

1. Set 1 (0% fvf): Five out of five specimens successful.
2. Set 2 (25% fvf): Two out of five specimens successful.
3. Set 3 (37% fvf): One out of five specimens successful.
4. Set 4 (78% fvf): Five out of five specimens successful.

3.3. Fracture surfaces

Set 1 specimens

All five Set 1 specimens fractured at the interface between the fibre-reinforced base and the start of the gauged section, as opposed to the expected typical fracture within the gauge section itself. This suggests apparent weakness in the adhesion between carbon-fibre-reinforced layers and unreinforced layers. The cause of this systemic weakness is unknown, but it is thought that the sudden drop in fibre volume fraction may be inducing certain defects in the interface within the printed part. The fracture interface itself (Figure 10) exhibited ductile behaviour in all five specimens. This is due to the ductile nature of the nylon matrix.

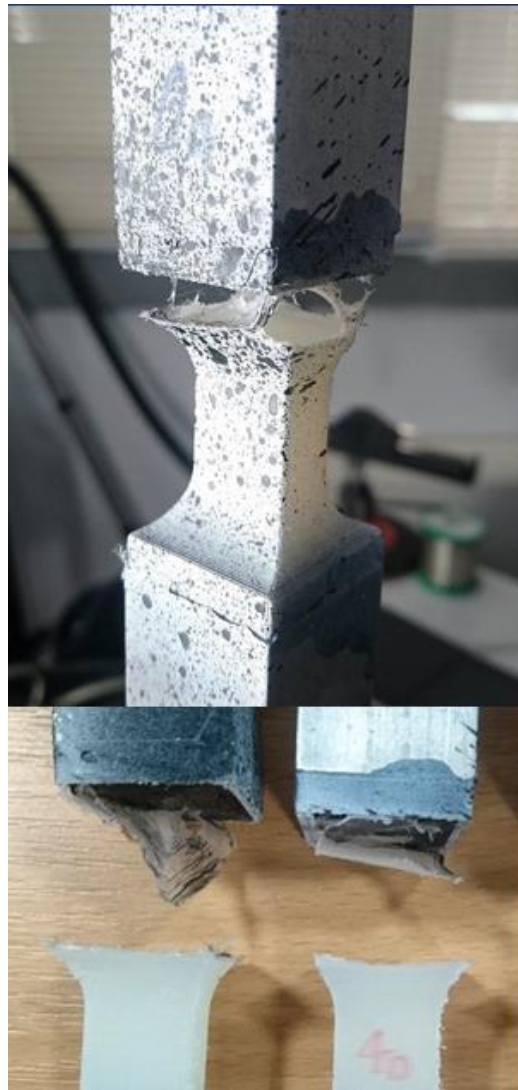


Figure 10: The ductile fracture interface of Set 1 specimens

Sets 2 and 3 specimens

The fracture surfaces of successful specimens from Sets 2 and 3 were similar. All successful specimens belonging to the two sets failed within the gauge section. The fracture interface was horizontal and suggested a final matrix failure (Figure 11). It was clear that the fibre had some function in resisting tension, as fibres were observed to stretch and break during testing.



Figure 11: The fracture interface of Sets 2 and 3. The top photo shows carbon fibre being stretched during testing, suggesting the fibre assisted in resisting tension

Set 4 specimens

Similarly to Sets 2 and 3, the failure occurred within the gauged section for all successful specimens. However, the role of carbon fibre providing tensile strength was much more apparent in Set 4 specimens as can be seen from the stretched and torn fibres embedded in the fracture surfaces (Figure 12).



Figure 12: The fracture interface of Set 4 specimens. Torn fibres suggest prominent role of CF in providing tensile strength

3.4. Tensile properties

Table 2 shows the ultimate tensile strength (UTS) values for each tested specimen.

Table 2: UTS values for tested specimens				
Sample		Max Load /N	UTS /MPa	Failure Mode
Set 1	1	1893.74	12.44	Specimen
	2	1069.83	7.01	Specimen
	3	1148.35	7.54	Specimen
	4	908.65	5.91	Specimen
	5	495.62	3.23	Glue
Set 2	1	1425.6	9.11	Specimen
	2	1337.76	8.54	Specimen
	3	1367.35	8.73	Glue
	4	1183.73	7.58	Glue
	5	1050.23	6.72	Glue
Set 3	1	1354.43	8.64	Specimen
	2	1195.11	7.69	Glue
	3	1309.49	8.36	Glue
	4	1167.52	7.43	Glue
	5	1159.33	7.39	Glue
Set 4	1	1101.54	7.01	Specimen
	2	1128.63	7.14	Specimen
	3	1137.17	7.17	Specimen
	4	1176.02	7.43	Specimen
	5	1134.31	7.17	Specimen

Figures 13 to 15 illustrate the tensile performance of each set relative to the other three. These graphs only illustrate specimens that have completed the test successfully.

As shown in Figure 13, the tensile behaviour of Set 1 specimens matched the ductile fracture patterns observed. While most samples had a fairly consistent elastic behaviour, the variation in UTS was high. This phenomenon can be explained by the possible presence of random print defects, which can unpredictably alter the tensile strength of the specimen. These defects are likely to be due to the sudden change in fvf. More research is required to study the effects of changes in fvf on part integrity. All specimens exhibited typical ductile behaviour, including the presence of high and low yield points. The ductile behaviour is consistent with the expected behaviour of nylon 6.

Figure 14 illustrates the tensile behaviour of the specimens from Sets 2 and 3. They exhibit consistent elastic behaviour and recorded the highest UTS values. This suggests the existence of an optimum fvf at which the combined strength of the nylon matrix and carbon fibre yields the highest tensile strength. Further research is required to determine this optimum point. The specimens were significantly less ductile than Set 1 specimens in their post-yield behaviour. This is expected as the carbon fibre provides additional stiffness to the specimens. However, further tests are required to provide better certainty of the results.

The tensile behaviour of Set 4 specimens is shown in Figure 15. As with other reinforced specimens, the elastic behaviour of all specimens tested was consistent, as were the UTS values. This is expected as the carbon fibre provides a more consistent build. However, the UTS values of Set 4 specimens were significantly lower than those of Sets 2 and 3. This suggests that the higher fvf came at the expense of matrix material, whose primary function is to provide interlayer adhesion, thus reducing overall strength.

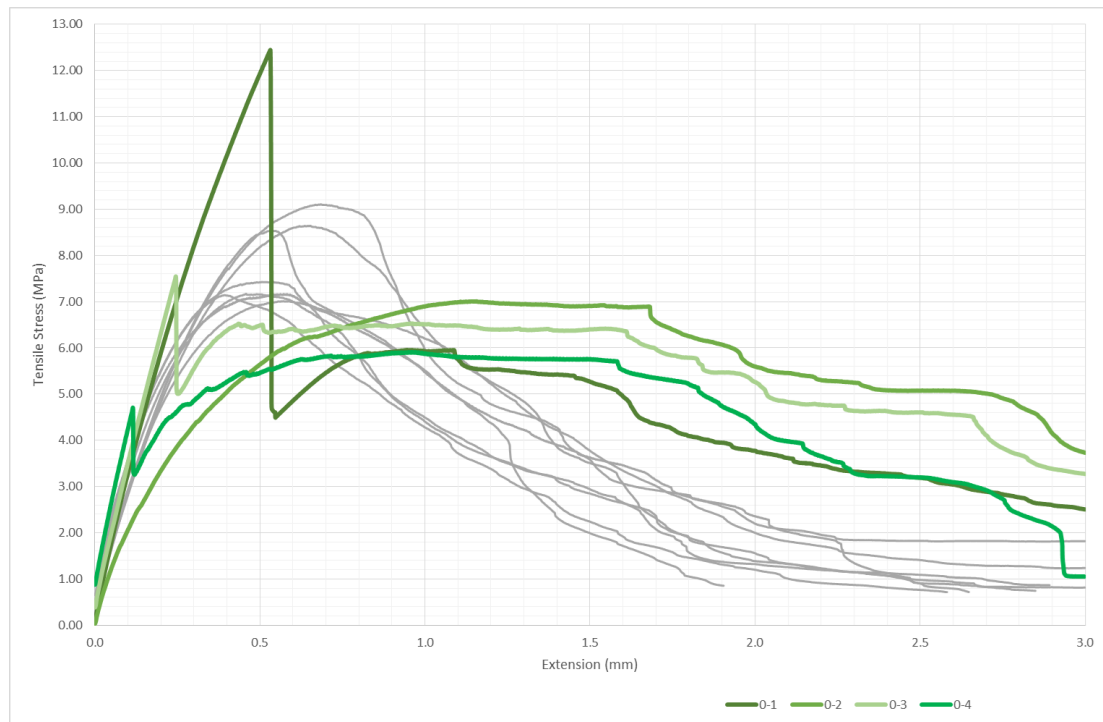


Figure 13: Tensile stress behaviour of Set 1 specimens

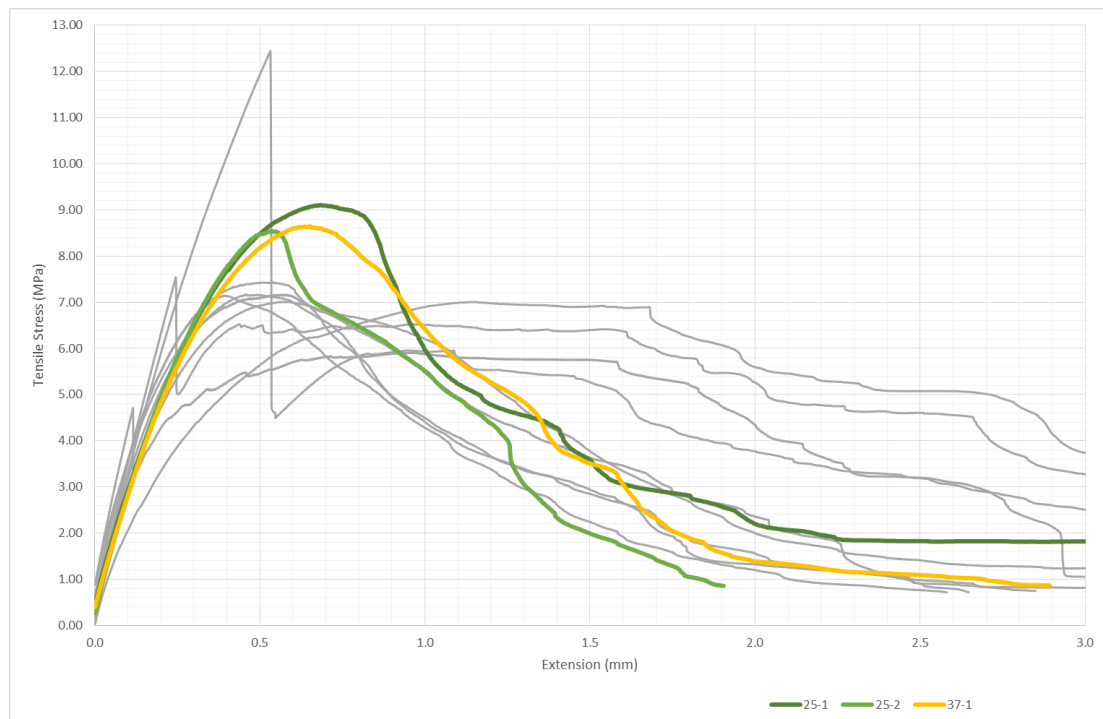


Figure 14: Tensile stress behaviour of Sets 2 and 3 specimens

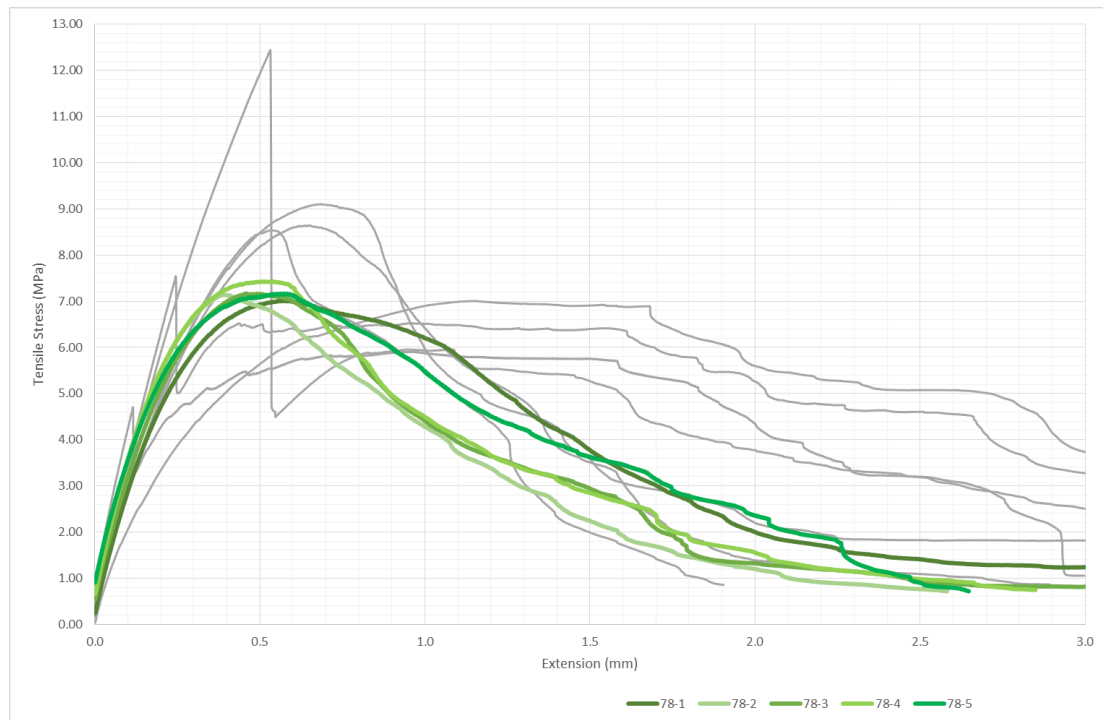


Figure 15: Tensile stress behaviour of Set 4 specimens

3.5. Future Work

Future work is planned to study the effects of print defects, fvf variations and conduct more thorough through-thickness tensile testing.

More specimens

More specimens will be manufactured in order to study the effect of the sudden change in fvf. This may be accompanied with SEM inspection of fracture interfaces to detect possible print defects in the interface between the last reinforcement layer and the first pure nylon layer. Different measures will be explored in order to prevent thermal deformation of unreinforced samples, including the introduction of a thick raft to be filed down in post-process.

Additional sets exploring a range of reinforcement ratios will be printed in order to determine the optimum fvf that provides the highest UTS values. All sets would have at least ten specimens in order to provide additional statistical significance as well as allow for failed parts.

Optimisation of the test

As demonstrated through the trials, despite following proper protocols, specimen bonding results were erratic, where glue failure occurred on a range of loads which seemed to follow no particular pattern. This is largely due to the difficult nature of nylon as a low surface energy material. As such, options will be explored in adjusting and altering the test method in order to account for inherent difficulties in both the material and the process, including exploring the options to bond specimens using a lap joint arrangement.

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