Ply and interlaminar behaviours of 3D-printed continuous carbon fibre-reinforced thermoplastic laminates; effects of processing conditions and microstructure

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Abstract

Taking advantage of an extended design and manufacturing space for composites, the technology of Fused Filament Fabrication (FFF) of continuous fibre-reinforced thermoplastics shows great potential for the production of the next generation of lightweight structural parts. This novel process is very much under development, and knowledge of the mechanical behaviour of the resulting 3D-printed materials is still limited. In this work, the intra- and inter-laminar behaviours of carbon fibre/polyamide printed laminates were extensively characterised to determine ply elastic and strength properties, as well as interface strength and fracture characteristics. Moreover, the effects of eventual production defects on these properties were analysed, putting in evidence some of the present shortcomings of the FFF process. Such defects include non-homogeneous fibre distribution, large amounts of intra- and interlaminar voids, and weak interlayer bonding, which are likely to be due to insufficient thermo-mechanical consolidation of the material during the FFF process, and have significant influence on the matrix-dominated mechanical properties. As a result, the transverse and interlaminar properties were found to be lower than those obtained through Hot Compression Moulding of an equivalent material. Besides highlighting possible process improvements, the mechanical characterisation carried out in this work promises a significant

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contribution to the abilities of designing and simulating general 3D-printed composite parts.

Keywords: Material Extrusion, continuous fibre-reinforced polymers, mechanical properties, fracture toughness, effects of defects

1. Introduction

The additive manufacturing of polymeric matrix composites has undergone considerable developments in recent years. This is mainly due to the ability of the process to produce final components using less steps than conventional manufacturing techniques, without the need for special tools and generating small amounts of waste material. The 3D-printing of composite materials, with the inclusion of fibres in the matrix, has substantially improved the mechanical properties, as well as the dimensional stability of the material during the process, allowing the production of larger parts [1, 2]. Due to the improved mechanical performance, in addition to prototyping, 3D-printing of composites is also suitable for manufacturing factory tools, spare parts and custom products. As a result, its use is increasing in sectors such as bio-medical, aerospace, automotive and industrial [3, 4]. To date, discontinuous carbon or glass fibres have mainly been used to reinforce the polymer, but in recent years continuous fibre composite printers have been developed, which have significantly improved the mechanical performance and greatly expanded the field of application of 3D-printed polymers [1, 2].

Fused Filament Fabrication (FFF) is the most widespread 3D-printing technology due to the simplicity of the process, the low cost of the printers and the ability to combine different materials [4]. The FFF printing of continuous fibre-reinforced thermoplastics is based on two different systems for the impregnation of the fibre in the matrix [5]: the use of a pre-impregnated filament and the impregnation inside the extrusion head of the printer. The second system does not require a pre-extrusion process of the material, and is therefore more economical. In addition, it is possible to combine different materials and respective proportions in the same printer. However, preliminary researches have indicated that the mechanical properties obtained are lower than those used by the pre-impregnated filaments, due to insufficient matrix-fibre impregnation [6, 7].

One of the most promising possibilities offered by the technology of 3D-printing of continuous fibre composites is its ability to steer fibres through

32 curved tracks making it suitable for the manufacture of non-conventional
33 laminates. An example of these types of laminates are variable stiffness pan-
34 els, where the load redistribution can be used for stiffness design, increased
35 resistance and improved stability against imperfections and geometric discon-
36 tinuities [8]. Advanced numerical models based on advanced failure criteria
37 and continuum damage mechanisms were developed for the design of these
38 laminates, and were successfully tested on components manufactured by Au-
39 tomated Fibre Placement (AFP) [9–11]. Although some preliminary studies
40 have been carried out [12–14], the applicability of 3D-printing technology for
41 the manufacture of non-conventional laminates is still to be developed. To
42 design and manufacture these types of structures with 3D-printing, the print-
43 ing process limitations and the mechanical behaviour of the printed materials
44 must be known in depth.

45 In recent years, different studies have addressed the physical and mechan-
46 ical characterisation of printed continuous carbon fibre-reinforced polymers
47 (CCFRP) obtained with pre-impregnated filament FFF technology. Sev-
48 eral publications analysed the microstructure of the printed composites as
49 well as the deposition and filling limits of the printing process [15–17]. The
50 mechanical characterisation of the printed CCFRP was also performed for
51 different load cases: tension [15, 16, 18–20], compression [19], in-plane shear
52 [19, 20] and interlaminar shear [21]. In other studies, the influence of the
53 laminate sequence, the type and density of reinforcement and fill patterns on
54 the tensile, compressive and bending behaviour of printed components were
55 analysed [22–25]. There are also studies that analysed other phenomena such
56 as creep [26], fatigue [27], quasi-static indentation [16], impact resistance [28]
57 and hygromechanical behaviour [17]. In these studies it was observed that
58 the mechanical behaviour of the printed materials was highly conditioned by
59 process-induced defects.

60 The aforementioned studies are an important contribution to the develop-
61 ment of this 3D-printing technology; however, there remains a lack of research
62 into the mechanical characterisation of printed continuous fibre-reinforced
63 thermoplastics. On the one hand, most of the published works have charac-
64 terised the properties of the laminate obtained directly from the print, which
65 includes contours and outer layers of non-reinforced material, and not the
66 lamina properties of the printed CCFRP. Lamina properties are necessary
67 for the general design and simulation of these laminated materials. To the
68 knowledge of the authors, only one study determined the elastic properties
69 at lamina level [20]. On the other hand, there are no published research

on characterisation of interlaminar fracture behaviour of printed continuous fibre-reinforced thermoplastic laminates. This characterisation is necessary in order to apply delamination failure criteria for composite damage tolerance and durability analyses. Thus, there is a need for a detailed study that collects a complete mechanical characterisation of the material necessary to drive the use of this new technology in the design of complex and optimised composite parts.

In this study, the ply and interlaminar behaviours of a 3D-printed continuous carbon fibre-reinforced polyamide laminate are extensively characterised to determine elastic, strength and fracture properties, and to identify the most relevant fracture mechanisms. At the ply level, different material orthotropic directions are addressed, i.e. longitudinal, transverse and in-plane shear, as well as load cases (tension and compression). At interface level, interlaminar resistance and critical energy release rates under mode I and mode II loadings are investigated. In the discussion of the results obtained, these properties are compared to those of an equivalent material manufactured by means of Hot Compression Moulding [29–32] to assess the performance of this novel FFF process. In addition, the microstructure of the printed material and the thermal history of the material during the process have been studied to identify process-induced defects and measure their impact on the resulting mechanical properties.

2. Printing process, material and test methods

2.1. Printing process and resulting material

The printer employed in this study was the MarkTwo® from Markforged®, which uses pre-impregnated filament FFF 3D-printing technology [33]. This printer has two extrusion systems, one for printing construction material and another one for printing reinforcement material (Figure 1(a)). The head moves in the xy plane during the printing of each layer, while the bed positions the part on the z -axis. The reinforced materials are polyamide (PA) based composites with continuous carbon, glass or aramid fibres. In this study, the layers were filled with continuous fibre-reinforced composite using a rectangular pattern with a 100% filling density and a bead orientation that depends on the test configuration (Figure 1(b)). The construction material can be either neat polyamide 6 (PA6) or PA6 reinforced with short carbon fibre. Both are used for printing the first and last layers of a laminate

105 (floor and roof layers, as shown on Figure 1(a)), and the contours of each
 106 layer (Figure 1(b)).

Figure 1: CCF/PA composite 3D-printing FFF system: (a) general drawing of the process and stacking sequence of the layers and (b) rectangular filling pattern of a layer.

107 The material characterised in this study was the continuous carbon fibre-
 108 reinforced polyamide (CCF/PA). Carbon fibre-reinforced composites offer
 109 high mechanical properties and low density, and are widely used in sectors
 110 such as aerospace and automotive. In addition, thermoplastic matrix com-
 111 posites are of special interest because they do not require a curing stage, are
 112 easier to recycle than thermoset resins, and can be mass-produced [34]. The
 113 composition and calorimetric properties of all the filaments available in the
 114 MarkTwo® printer were reported in [35] which states that the CCF/PA fil-
 115 ament has an average diameter of 363 μm and contains approximately 1006
 116 fibres of 7 μm in diameter, resulting in a fibre volume fraction of 34%. The
 117 results obtained in the calorimetry tests indicated that the PA matrix is
 118 amorphous and matches the profile of PA 6/3T.

119 The extrusion parameters are fixed for the MarkTwo® printer. The
 120 composite filament is extruded at a temperature of 252°C and at a speed
 121 of 15 mm/s. The layer thickness is 125 μm (Figure 1(a)), therefore the
 122 363 μm diameter filament undergoes a considerable flattening during the
 123 printing process. The width of the printed bead is 0.9 mm (Figure 1(b))
 124 which matches with the diameter of the printer nozzle.

125 The fibre and void contents of the printed CCF/PA composite were mea-
 126 sured in accordance with ASTM D3171 by matrix digestion in sulphuric

127 acid/hydrogen peroxide [36]. Three square samples with 25 mm sides and 2
 128 mm thickness with a $[0]_{16}$ unidirectional laminate were analysed. For these
 129 specific tests, the floor and roof PA layers and PA contours were removed in
 130 order to determine the amount of fibre and voids of the CCF/PA composite.

131 Matrix digestion of each sample was carried out in 50 ml of sulphuric acid,
 132 on a hot plate at 275°C for 5 hours. Then, 50 ml of 30% hydrogen peroxide
 133 was added to oxidise the PA matrix. In order to determine the volumetric
 134 fibre and void contents the density was determined by weighing the samples
 135 in air and water, in accordance with the procedure described in ASTM D792
 136 [37]. The density of the PA matrix and carbon fibre reinforcement were
 137 obtained from the literature [38, 39].

138 Table 1 compares the results obtained for the printed CCF/PA material
 139 and those for the pre-printed filaments [35]. Similar fibre contents were ob-
 140 tained using the matrix burn-off technique [18] and using microscope image
 141 processing technique [15]. The amount of voids is high compared to that of
 142 the pre-printed filament.

Table 1: Comparison of density, fibre content and voids content between the filament [35]
 and printed sample of CCF/PA (the standard deviation is indicated in parentheses).

Property	Filament	Printed sample
Density (kg/dm ³)	1.35	1.25 (0.01)
Fibre volume fraction %	33.9	31.4 (0.1)
Voids volume fraction %	0.2	7.5 (0.3)

143 2.2. Mechanical testing methods

144 In order to characterise longitudinal and transverse ply properties, ten-
 145 sile and compressive mechanical tests were carried out in accordance with
 146 the ASTM D3039 [40] and ASTM D3410 [41] standards respectively. Tensile
 147 tests were performed on a Zwick/Roell universal testing machine equipped
 148 with a 50 kN load cell and at a load-rate of 1.5 mm/min. Longitudinal and
 149 transverse strains were measured with a Gom-Aramis digital image correla-
 150 tion (DIC) system. In the case of compression tests, an Instron universal
 151 testing machine equipped with a 150 kN load cell was used at a load-rate of
 152 1.5 mm/min. The longitudinal strain of both sides was measured with strain
 153 gauges of a 3.18 mm sized active grid in order to determine the bending level
 154 in the test and the stiffness of the material. The characterisation of the in-
 155 plane shear behaviour was carried out in compliance with the ASTM D3518

156 standard by means of tensile testing of $[\pm 45]_{4S}$ laminates [42]. In this case,
157 the load-rate was 5 mm/min.

158 The characterisation of the interlaminar shear strength was carried out
159 by means of the short beam shear (SBS) test according to the ASTM D2344
160 standard [43]. The tests were performed in a Zwick/Roell universal testing
161 machine equipped with a 5 kN load cell at a 1 mm/min load rate and 24 mm
162 span length.

163 The characterisations of mode I and mode II interlaminar fracture tough-
164 ness were carried out using the double cantilever beam (DCB) and end-
165 notched flexure (ENF) tests respectively, in accordance with the ASTM
166 D5528 [44] and ASTM D7905 standards [45]. The DCB tests were per-
167 formed on a Instron universal testing machine equipped with a 500 N load
168 cell and at a load-rate of 1 mm/min. The pre-crack length was 50 mm and it
169 propagated to 80 mm under crack-opening load. The ENF 3-point bending
170 tests were performed on an Instron universal testing machine equipped with
171 a 10 kN load cell and at a 0.5 mm/min load-rate. The nominal span and the
172 average pre-crack length were 100 mm and 30 mm, respectively. The typical
173 fracture surfaces produced by the different test types were examined by scan-
174 ning electron microscopy (FEI Nova NanoSEM 450) and optical macroscopy
175 (Leica Z16 APO).

176 2.3. Samples preparation

177 The lay-up and dimensions of the samples for the different tests are given
178 in Table 2. Five repetitions of each experiment were performed. All samples
179 were obtained by cutting a multi-sample printed plate, thus also eliminating
180 the PA contour (Figure 1(b)). A single layer of floor and roof of PA6 was used
181 in the manufacture of the plates. In order to obtain the lamina properties
182 of the CCF/PA material exclusively, the results from the mechanical tests
183 were recalculated, based on a rule of mixtures applied to two homogeneous
184 materials, the PA6 of the outer layers and the CCF/PA of the inner layers.
185 The mechanical specifications for printed PA6 were taken from literature [35].

Table 2: Standards carried out, lay-up (bead/fibre orientation and number of layers) and dimensions (thickness $\times$ width $\times$ length) of the tested samples.

Test	ASTM	Lay-up	Dimensions (mm)
Longitudinal tension	D 3039	$[0]_{16}$	$2.25 \times 15 \times 250$
Longitudinal compression	D 3410	$[0]_{16}$	$2.25 \times 12.5 \times 135$
Transversal tension	D 3039	$[90]_{18}$	$2.5 \times 22 \times 200$
Transversal compression	D 3410	$[90]_{26}$	$3.5 \times 20 \times 135$
In-plane shear	D 3518	$[\pm 45]_{4S}$	$2.25 \times 22 \times 210$
Interlaminar shear	D 2344	$[0]_{46}$	$6 \times 12 \times 40$
Interlaminar fracture Mode I	D 5528	$[0]_{24}$	$9 \times 25 \times 180$
Interlaminar fracture Mode II	D 7905	$[0]_{24}$	$9 \times 25 \times 130$

Many of the tested samples required tabs to ensure proper load transfer. These tabs were manufactured separately by 3D-printing of continuous fibre-glass composite and bonded with an epoxy adhesive (Loctite® EA 9466™). Moreover, PA is a hygroscopic polymer and its mechanical properties depend on the quantity of absorbed moisture [17]. To ensure the same testing conditions in the different configurations, all the samples were conditioned in a climatic chamber for at least 4 days before testing at 23°C and 50% relative humidity.

The same samples were used for both mode I and mode II interlaminar fracture tests, i.e., the mode I crack was used as a pre-crack for mode II. Due to warping problems, it was not possible to print monolithic laminates thick enough to perform the test without fracture of the loading arms due to excessive bending deformation. For this reason, sandwich-type samples were manufactured, consisting of a core of printed CCF/PA material of 3 mm thickness, reinforced on the top and bottom with unprinted composite skins of epoxy resin and weave fabric Dyneema® (Figure 2). This solution was previously implemented in [46] with successful results. To create the delamination initiator, the print was stopped during the printing process and the required area was covered with paper adhesive tape.

Figure 2: Interlaminar mode I fracture test sample reinforced with Dyneema® composite skins.

205 3. Results

206 3.1. Ply longitudinal behaviour

207 The average measured mechanical values for longitudinal tensile and com-
 208 pressive tests are compiled in Table 3. For the longitudinal tensile tests,
 209 brittle fracture was observed at 1.27% strain (Figure 3(a)). The stress-strain
 210 behaviour is approximately linear-elastic, in spite of a slight stiffening effect
 211 which is characteristic of continuous carbon fibre-reinforced composites and
 212 is likely to be caused by the change of morphology of the fibres under increas-
 213 ing load, as discussed in [47]. The average values of initial tensile modulus
 214 (E_1^t in Figure 3(a)) and of tensile strength were 69.4 GPa and 905.3 MPa
 215 respectively.

216 With regards to the fracture behaviours, a longitudinal splitting mode
 217 is observed, mainly between material beads and between individual layers
 218 (Figure 4(a)). The mechanisms of fibre breakage, fibre pull-out and matrix
 219 fracture can be observed from the SEM images of the fracture surface (Figures
 220 4(b) and 4(c)). The pulled-out fibres have matrix residues on the surface
 221 (Figure 4(c)), which may indicate proper fibre/matrix interfacial bonding
 222 inside the beads.

Figure 3: Stress-strain curves in longitudinal direction and average linear fit: (a) tensile behaviour and (b) compressive behaviour. The linear fitting was made in a strain range of 0.1-0.3%.

Figure 4: Longitudinal tensile fracture: (a) longitudinal splitting fracture mode, (b) fibre breakage, fibre pull-out mechanisms and matrix fracture in the fracture surface and (c) matrix residues on the surface of the pulled-out fibres.

223 The longitudinal compressive behaviour can be considered linear-elastic
 224 up to failure, with a maximum deformation of 0.69% (Figure 3(b)). The av-

225 erage values of compressive modulus (E_1^c in Figure 3(b)) and of compressive
 226 strength were 63.9 GPa and 426 MPa respectively. Compared to tensile prop-
 227 erties, compressive stiffness was slightly lower while the compressive strength
 228 was 47% of the tensile strength in absolute value.

229 Macroscopic images of the side view of the fractured sample are shown in
 230 Figure 5 for two different magnification levels. The fibre kinking mechanism
 231 is observed near the tab (Figure 5(a)) due to micro-buckling of the composite
 232 layers in a narrow band. The average inclination of the kink band, β , was
 233 20° (3°), considering the average of 5 samples.

Figure 5: Macroscopic images of the side view of the longitudinal compression fracture: (a) fibre kinking mechanism near the tab and (b) fibre kinking band and inclination β .

234 The longitudinal tensile and compression properties are higher (about
 235 16%) than those reported by Markforged® [48]. This may be because Mark-
 236 forged® obtained the properties of the sandwich configuration (with internal
 237 CCF/PA layers and external non-reinforced PA layers) and not that of the
 238 lamina.

239 3.2. Ply transverse behaviour

240 The stress-strain curves resulting from the transverse tension and com-
 241 pression test are plotted in Figure 6. The corresponding fracture images are
 242 shown in Figures 7 and 8. With regards to the transverse tensile behaviour,
 243 very low plastic strain was observed and the fracture can be considered to
 244 be brittle (Figure 6(a)). The average values of tensile modulus and tensile
 245 strength were 3.5 GPa (E_2^t in Figure 6(a)) and 17.9 MPa, respectively (see
 246 Table 3).

247 As shown in Figure 7(a), the fracture is produced in a plane perpendicular
 248 to the load and parallel to the fibres, and coincides with an interface between
 249 beads. The fracture surface is very irregular and shows localised matrix
 250 fracture zones, fibre breakage and debonding zones, voids and stripped fibres
 251 with a low level of resin impregnation (Figures 7(b) and 7(c)).

Figure 6: Stress-strain curves in transverse direction and average linear fit: (a) tensile behaviour and (b) compressive behaviour. The linear fitting was made in a strain range of 0.1-0.3%.

Figure 7: Transverse tensile fracture: (a) fracture plane perpendicular to the load, (b) and (c) SEM images of the fracture surface with two different magnification level. Matrix fracture zones, fibre breakage and debonding zones, voids and stripped fibres with very little impregnated resin are observed.

Under transverse compression, the material showed a more ductile behaviour and exceeded 5% strain before failure (Figure 6(b)). This effect may be due to the elasto-plastic behaviour of the PA matrix. The mechanical properties for compression show a greater dispersion than for tension (Table 3 and Figure 6(b)). The compression modulus E_2^C was 3.7 GPa, slightly higher than the tensile modulus, and the compressive strength was 66 MPa, more than three times the tensile strength, in absolute value. The failure mode was by micro-buckling of the composite layers forming X- or V-shaped bands (Figure 8). Layer delamination and layer breakage zones were identified in these bands. The average inclination of the kink band β was 43° (8°) taking into account the 5 samples.

Figure 8: Macroscopic images of the side view of transverse compression fracture: (a) V-shaped kink bands and (b) X-shaped kink bands. Layer delamination and layer breakage zones are identified in these bands.

3.3. In-plane shear behaviour

Extremely high strains were achieved during the in-plane shear tests (Figures 9(a) and 9(b)). This may be due to the ductile nature of the PA matrix. For the highest applied strains, a stiffening effect can be appreciated. This is likely because of the alignment of the fibres to the load direction, as denoted by an inflexion point in the stress-strain behaviour. The in-plane shear strength of 61.5 MPa was considered to occur just before the initiation of the fibre alignment phenomenon (the inflection point) which corresponded to a 25% shear strain level. The shear strength for a 5% strain, as considered in the test standard, was also determined (Table 3). The shear modulus was 1.9 GPa (G_{12} in Figure 9(b)). From the obtained fracture surface, fibre breakage and pull-out, shearing of beads and inter-layer delamination can be observed (Figure 9(c)).

Figure 9: In-plane shear behaviour: (a) sample shape after and before the test, (b) shear stress-strain curves and average linear fit, and (c) macroscopic image of the fracture. Fibre breakage and pull-out, beads shearing and interlayer delamination fracture mechanisms are identified. The linear fitting was made between 0.2 and 0.6% shear strain.

3.4. Interlaminar behaviour

During the SBS tests small delaminations occurred between the load and support points in the area of maximum interlaminar shear stress (Figure 10(b)). Inelastic behaviour of the material was also observed, as indicated by the shape of the deformation (Figure 10(b)). The interlaminar shear strength was 37.9 MPa on average (Table 3).

Figure 10: Interlaminar shear behaviour: (a) stress-displacement curves of the short-beam shear test and (b) macroscopic image of the side view of a fractured sample. Interlayer delaminations and inelastic deformation shape are identified.

282 The load-deflection curves of the DCB tests are shown in Figure 11(a).
 283 The load-deflection ratio is linear, with a slight deviation before fracture
 284 propagation. This behaviour is characteristic of tough matrix composites.
 285 In the propagation phase a great dispersion in the results is observed. The
 286 crack growth was stable throughout the entire test. The mode I interlaminar
 287 fracture toughness was calculated using the modified beam theory method
 288 given in ASTM D5528 [44]. A resistance-type fracture behaviour was identi-
 289 fied (Figure 11(b)): the toughness increased in the fracture initiation phase
 290 and then tended to stabilise in the propagation phase. The cause of the in-
 291 creased delamination resistance could be the development of fibre bridging
 292 (Figure 12(a)). The mode I fracture toughness for a total crack length of
 293 80 mm was 2 (0.17) kJ/m². The fracture surface is very irregular (Figure
 294 12(b)). Debonded and fractured fibres are observed, which have probably
 295 formed bridges between the crack surfaces. These debonded fibres contain
 296 little matrix on the surface, and the imprints left in the matrix by the fibres
 297 are clearly visible. Matrix microcracking is also visible in matrix-rich areas
 298 and in areas where the fibres have been removed.

Figure 11: Mode I interlaminar fracture toughness test curves: (a) load-deflection curves of the crack opening tests and (b) resistance-type fracture behaviour (average and standard deviation values).

Figure 12: Mode I interlaminar fracture: (a) fibre bridging and (b) SEM image of the fractured surface. Debonded and fractured fibres, fibre imprints and matrix fracture are visible.

299 The load-deflection curves for the three validated ENF tests are shown
300 in Figure 13(a) for slightly different crack lengths (value in parentheses).
301 The curves are slightly non-linear and stiffness decreases with the load. The
302 fracture toughness obtained was 1.59 (0.33) kJ/m², and was calculated using
303 the compliance calibration method as described by ASTM D7905 [45]. As in
304 mode I, the fracture surface is very irregular, and debonded and fractured
305 fibres, fibre imprints and matrix deformation and microcracking are observed

(Figure 13(b)). Fracture toughness in mode II was lower than in mode I. This behaviour is not usual, and may be due to the type of structure of the material that does not favour the interlaminar shear microcracking mode of the matrix in mode II.

Figure 13: Mode II interlaminar fracture test: (a) load-deflection curves of the ENF test for slightly different crack lengths (value in parentheses) and (b) SEM image of the fractured surface. Debonded and fractured fibres, fibre imprints and matrix fracture are visible.

4. Discussion

As mentioned in the introduction, several studies have addressed the mechanical characterisation of the printed CCF/PA in recent years. Most of these works have characterised the properties of the laminate obtained directly from the print, which includes contours and outer layers of non-reinforced PA6 material, and not the lamina properties of the printed CCF/PA. With regards to the longitudinal tensile behaviour, the stiffening effect on the stress-strain curve and the longitudinal splitting fracture mode shown in the present paper were also reported in the literature [15–17]. For longitudinal compression, failure mechanisms similar to those obtained in the present work were reported in [19]. In the aforementioned works, the mechanical properties of the printed laminate were obtained rather than those of the CCF/PA lamina. This justifies that the reported stiffness and strength properties are lower than those presented in this work. The elastic properties of the lamina E_1^t , E_2^t and G_{12} were investigated in [20]. The reported values are higher than those measured in the present paper: 8% in E_1^t , 24% in G_{12} and 57% in E_2^t . Significantly thinner specimens were tested in that

327 work [20], resulting in a lower ratio of reinforced to non-reinforced layers.
328 Moreover, a lower modulus of elasticity for the PA6 of the outer layers was
329 considered for the determination of the CCF/PA lamina properties. These
330 reasons may be behind the differences in property values obtained in the two
331 studies. Finally, with respect to interlaminar behaviour, slightly lower (6%)
332 interlaminar shear strength values was reported in [21].

333 In order to assess the performance of the printed CCF/PA composite,
334 the mechanical properties obtained in the current work were compared with
335 those of an equivalent (reference) material manufactured by means of Hot
336 Compression Moulding (HCM) [29–32]. The reference properties are shown
337 in Table 3 along with those characterised in this study. The tensile mechan-
338 ical properties in longitudinal direction are similar to the values reported for
339 samples made by HCM [31]. However, for the remaining deformation modes,
340 especially for transverse loading, the properties are significantly lower. This
341 outcome is likely be due to the influence of defects induced by the 3D-printing
342 process which ought to have a larger impact on resin dominated properties.

Table 3: Mechanical properties of the 3D-printed CCF/PA and comparative with properties obtained by hot compression moulding reference process [30–32] (the standard deviation is indicated in parentheses).

Test	Property	Measurement	Reference
Longitudinal tension	Tensile modulus (GPa)	69.4 (0.2)	72.2 [31]
	Poisson’s ratio	0.41 (0.01)	-
	Tensile strength (MPa)	905.3 (4.3)	1047 [31]
	Ultimate tensile strain (%)	1.27 (0.04)	-
Longitudinal compression	Compressive modulus (GPa)	63.9 (1.3)	-
	Compressive strength (MPa)	426.0 (9.7)	-
	Ultimate compression strain (%)	0.69 (0.02)	-
Transversal tension	Tensile modulus (GPa)	3.5 (0.1)	7.4 [31]
	Tensile strength (MPa)	17.9 (1.7)	33.3 [31]
	Ultimate tensile strain (%)	0.58 (0.07)	-
Transversal compression	Compressive modulus (GPa)	3.7 (0.2)	-
	Compressive strength (MPa)	66.0 (6.6)	-
In-plane shear	Shear modulus (GPa)	1.9 (0.1)	-
	Shear strength at 5% (MPa)	43.4 (2.6)	-
	Shear strength at 25% (MPa)	61.5 (2.0)	-
Interlaminar shear	Interlaminar shear strength (MPa)	34.1 (0.1)	46.7 [30]
Interlaminar fracture	Mode I fracture toughness (kJ/m ²)	2.00 (0.17)	2.20 [32]
	Mode II fracture toughness (kJ/m ²)	1.59 (0.33)	2.68 [32]

The mechanical performance of the printed material depends largely on the quality of the bonding between the printed beads. Two phenomena occur in the bead bonding process: the creation and growth of the contact surface between beads, and the molecular diffusion of the polymer chains into the contact surface. Both phenomena depend on the thermal history of the material. The longer the material remains above the glass transition temperature T_g , the greater the molecular diffusion between the beads. Part design, process parameters and heat transfer conditions in the material and in the printing chamber directly influence the temperature history [4, 49].

In this study, the composite was extruded at a temperature of 252°C while the T_g of the amorphous PA 6/3T matrix is 143°C, as determined in [35]. In order to analyse the thermal history of the printed CCF/PA, the temperature of the material was measured during printing of a 40 mm long unidirectional sample. An infrared camera (FLIR Titanium 550 M) was used

for this purpose. The emissivity was adjusted to 0.75 taking into account the extrusion temperature of the material in the nozzle. It was observed that the recently printed bead cooled very quickly to a temperature below the T_g , in approximately one second (Figures 14(a) and 14(b)). In the region where the material was being deposited, at least the three layers underneath exceeded T_g . The temperature of the deposited material tended to stabilise at a temperature slightly above 50°C (Figure 14(b)) and the temperature in the printing chamber was stabilised at 28°C, for an outside room temperature of 19°C.

Figure 14: Thermal history of printed CCF/PA composite: (a) temperature distribution after printing a 40 mm bead and (b) temperature along the deposited bead for the same instant.

From the information above, it would seem that the deposited material heats the bottom layer sufficiently. However, cooling is rather fast and leads to the halting of molecular diffusion within a very short period of time, effectively not allowing for a proper consolidation of the bonding between layers. As a result, ply interfaces become weak regions in the laminates produced by FFF of continuously reinforced materials. Presumably the prefixed settings (temperature and printing speed) in the MarkTwo® printer seek a compromise between the mechanical performance of the printed material and the productive capacity of the printer. Similar conclusions were derived in [50], where the temperature and strain history of the printed CCF/PA were measured by sensors embedded in the material. The study also concluded that the fibre orientation strongly influences the process-generated temperature histories and residual strains. In contrast, in HCM, the creation of the interfaces and the diffusion of the material is ensured since the material is

380 kept under high pressure levels at temperatures above T_g for a long period
 381 of time (280°C for 3.5 min under a compression pressure of 4 kg/cm² for
 382 CF/PA6 pre-impregnated sheets [31]).

383 The microstructure of the printed parts was also analysed to evaluate
 384 the quality of the material and to identify process-induced defects. For this
 385 purpose, a unidirectional CCF/PA composite sample was printed, and the
 386 cross-section was examined by optical microscopy (Leica DM i8C). The mi-
 387 croscope images of the cross section of the unidirectional sample are shown
 388 in Figure 15 at different magnification levels. The interlayer limits are visi-
 389 ble (marked with horizontal dashed lines on Figures 15(a) and 15(b)). This
 390 may be an indication that the molecular diffusion of the matrix is in fact
 391 incomplete during the printing process. The interface between the adja-
 392 cent beads are also identifiable (marked with vertical dashed lines on Figure
 393 15(a)), due to the presence of large voids. This observation was also made
 394 in [16, 17], wherein the overlapping of the beads was identified. In addition,
 395 this overlap is not uniform, and limits of beads are alternated with more
 396 or less voids (Figure 15(a)). This large number of voids may indicate that
 397 the printing process is unable to adequately compact the extruded filament
 398 onto the printed part. It can also be observed that the fibre distribution is
 399 non-uniform, and both large matrix-dominated and high fibre density zones
 400 can be distinguished (Figure 15(b)). This phenomenon was also identified in
 401 the pre-printed filament [35].

Figure 15: Cross section of the unidirectional CCF/PA sample at different magnification levels: (a) bead limit regions (vertical dashed lines), interlayer limits (horizontal dashed lines) and voids; and (b) interlayer limits (horizontal dashed lines) and alternated matrix-rich and fibre-rich regions.

402 The fibre volume fraction obtained with 3D-printing of continuously re-
403 inforced laminates (31.4% reported in section 2.1) is lower than fibre volume
404 fractions obtained with traditional processes. Naturally, the amount of fi-
405 bre in the printed sample is limited by the amount of fibre in the filament
406 (33.9% [35]). The insufficient compaction of the material, mainly between
407 the beads, seems to be the origin of the large quantity of voids of the printed
408 parts (7.5% reported in section 2.1). These values are far from those that
409 can be obtained through the HCM process. A fibre volume fraction of up to
410 60% and a void content of less than 1% were obtained for a hot compressed
411 CCF/PA6 material in another study [29].

412 These process-induced defects have a great influence on the mechanical
413 behaviour of the laminated material. First, the high degree of in-homogeneity
414 of the material may be the cause of the early fracture under transverse ten-
415 sion due to the high stress concentrations that are produced in the matrix
416 near manufacturing defects, such as voids and regions with high fibre den-
417 sity. As shown in Table 3, the transverse tensile mechanical properties are
418 approximately half of those obtained through the HCM process [31]. Second,
419 the weak interlayer bonding makes the interlaminar strength of the printed
420 samples 27% lower (Table 3) than that obtained by HCM with a lower fibre
421 volume fraction [30]. In addition, the low interlaminar adhesion together
422 with the high compliance of the composite in transverse direction may fa-
423 cilitate the micro-buckling of the layers under compression loading. Finally,
424 the non-homogeneous structure of the material in the interlayer does not ex-
425 cessively penalise the fracture toughness in mode I (the toughness is 91% of
426 that obtained by HCM [32]). This may be due to the large number of fibre
427 bridges that are created between the fractured faces. In Mode II, however, it
428 seems that the most effective matrix shearing fracture mode is not activated
429 in the fractured surface, and the fracture toughness is significantly affected
430 (59% of that obtained by HCM [32]).

431 5. Concluding remarks

432 In this research, the mechanical behaviour of the printed CCF/PA com-
433 posite has been investigated, as well as the influence of defects and man-
434 ufacturing limitations on this behaviour. The mechanical performance in
435 longitudinal direction is equivalent to that obtained through the HCM ref-
436 erence process. However, under transverse and interlaminar loads (normal
437 and shear), the mechanical response is strongly influenced by manufacturing

438 defects, and the mechanical properties result to be significantly lower than
439 those obtained through HCM. Defects such as a large number of voids, non-
440 homogeneous distribution of fibres and poor bonding between beads and
441 layers were identified. These defects may be the result of the insufficient
442 thermo-mechanical consolidation of the material during the printing process
443 which occurs due to the fast cooling of the printed material bellow the glass
444 transition temperature.

445 It is clear from these results, that this process needs further develop-
446 ment to be considered viable for high-performance structural applications.
447 In this respect, different improvements are currently being implemented in
448 the process for the reduction of defects and limitations: material preheating
449 systems [51], layer post-compacting systems [52], multi-axis 3D-printing [53]
450 and simultaneous extrusion of different filaments [54].

451 Despite the limitations identified in the current process, the mechanical
452 characterisation carried out in this study will allow structural components to
453 be designed using advanced simulation techniques and explore the capacity of
454 this emerging technology for the design of lightweight structural components.

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