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Delamination observation occurred during the flexural bending in additively manufactured PLA-short carbon fiber filament reinforced with continuous carbon fiber composite

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ARTICLE INFO	A B S T R A C T	
Keywords: CCFRTC Flexural bending Delamination Microscope's micrograph	Additive manufacturing (AM) or 3D printing is process of fabricating polymer, ceramics, metallic and composite parts with complex geometries by means of optimized printing parameters. Fused deposition modeling (FDM) is an extrusion based 3D printing technology most commonly used for printing thermoplastic and fiber reinforced thermoplastic composite materials. This technique is extensively used due to its simplicity in usage, materials alteration and low cost and has the ability to fabricate parts with both short and continuous fibers. Continuous carbon fiber (CCF) are extremely lightweight, stiff and durable and when utilized as reinforcement material, they have a wide range of engineering applications. In this study, we are aiming to combine short carbon thermo- plastic material with CCF to form continuous carbon fiber reinforced thermoplastic composite (CCFRTC) material using FDM 3D printing technique. After the fabrication process, the additively manufactured composite specimen undertakes flexural bending test. During the flexural test, delamination occurred. This such caused was studied, discussed and explored by examining the fracture interface study using microscope's micrographs. At last, further study, recommendations and enhancement in the development were also presented.	

1. Introduction

Additive manufacturing (AM) or 3D printing, is process of fabricating parts and shapes of polymer, ceramics, metallic and composite materials with complex geometries shape followed by the CAD model and using optimized printing parameters [1-3]. The parts are fabricated layer by layer until the final product is obtained. AM technology is used for manufacturing light weight polymers and polymers matrix composite structures that have been extensively used in the engineering applications such as biomedical filed for tissue growth, architectural filed for structural models, aerospace, construction, textile, food processing industries, automobile, electronics, military and robots [3-7]. Various 3D printing techniques have been industrialized for the production of polymer and polymer composite parts. Mainly used technologies include; Selective Laser Sintering (SLS), Stereolithography (SLA) and Fused Deposition Modelling (FDM) [2,6]. Compared to conventional and traditional manufacturing, AM technology has the ability to shorten the design manufacturing cycle with more accuracy and perfection, thus it reduces the production cost and time [4].

Composite materials is a combination of two or more materials

which include a matrix and a reinforcement material that exhibits a higher strength-to-weight ratio and a substitute to metallic materials due to high-performance structures [8]. Previously, most of the composites were manufactured using traditional and conventional manufacturing techniques including pultrusion, vacuum bagging, filament winding and compression molding processes [9,10]. Polylactic acid (PLA), one the most widely used thermoplastics is a bio-based polymer extract from renewable resource environmental-friendly polymer widely used as plastic films, bottles, and biodegradable medical devices [11]. PLA is the most popular thermoplastic material broadly utilized to create object using FDM 3D printer [2].

FDM is an extrusion based 3D printing technology and most widely used AM technology for the production of thermoplastic polymer and thermoplastic polymer composite parts with complex geometries due to its simplicity, ease of use, low production cost and material filament adjustment property [4,12,13]. Most commonly used thermoplastic materials in the form of filament by the FDM process include acrylonitrile butadiene styrene (ABS), PLA, polypropylene (PP) and polyethylene (PE) [14,15]. The technology has the ability to print both the composite with either short or continuous fibers [16]. The quality of the

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Received 18 March 2021; Received in revised form 14 June 2021; Accepted 23 June 2021 Available online 4 July 2021 2590-1230/© 2021 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/). final 3D printed product depends on the printing parameters selected during the fabrication process. The pure thermoplastic polymers structures manufactured by FDM technique attains weak mechanical properties due to their less strength and stiffness and are not able to use as the functional components [6,17,18].

Number of researches have been performed to overcome the issue of poor mechanical performance of AM of pure thermoplastic materials. Now AM with improved technology has resolved the limitations of poor mechanical performance of pure thermoplastic materials. One of the best way is to introduce or add reinforcement in the form of fibers, particles or nanomaterials to form fiber-reinforced thermoplastic composites (FRTCs), which are considered as high performance materials and could be use in various engineering applications due to their remarkable functionality with much higher mechanical performance [1, 19]. Reinforcement fibers used in the manufacturing of composite may be either continuous or discontinuous, depending on the production process. However, the composites that are reinforced with short or discontinuous fibers possess fewer mechanical properties compared to continuous fibers [20]. Fig. 1 showed the types of orientation of fibers in a matrix, which could be either continuous carbon fiber reinforcement (CCFR) or short carbon fiber reinforcement (SCFR) elements. However, the composite reinforced with SCFR have less mechanical performance compared to the composites that are reinforced with CCFR, as the possibility of engaging continuous FRTC lead the functional part with much higher mechanical performance. AM with improved technology and process now has the ability to print FRPC structures and resolved the limitations of poor mechanical performance of pure thermoplastic materials [16,21,22]. Continuous carbon fiber (CCF), an ultra-high-strength material has the ability to use as reinforcement and to print with thermoplastic material forming continuous carbon fiber-reinforced thermoplastic composites (CCRFTCs). CCFRTCs are lightweight, stiff and strong and can be used in a wide range of engineering applications. Due to exceptional mechanical properties, recycling capability and potential to use as lightweight structures, CCFRTC are now becoming substitute materials to replace the conventional metals [23,24].

F. Ning et al. [25] fabricated SCFR reinforced ABS matrix composite and studied their mechanical and microstructural properties by achieving maximum tensile and flexural strengths of 42 MPa and 65 MPa. H.L. Tekinalp et al. [26] also fabricated SCFR reinforced ABS composite to study their processability, and mechanical performance by achieving the increase in the strength of composite part by 115% compared to pure ABS. The results also showed that by increasing the fiber content, voids between the printed layers decreased. Quasi-static indentation properties of the AM facesheet, cores, and sandwich structures were investigated [27]. L. Love et al. [28] evaluated the effects of SCFR contents on the strength, stiffness, and distortion of 3D printed parts. By the addition of SCFR composite parts, a limited enhancement in the mechanical performance has been achieved up to 20% due to the limitations in the reinforcement of SCFR [29]. R. Azzawi and N. Varughese [30] investigated the flexural behavior of encased steel composite beams within steel fiber reinforced concrete. Interlaminar fracture toughness of continuous carbon fiber-reinforced thermoplastic CFRTP [31] and tensile, compressive and shear strength of the AM CFRTP fabricated using extrusion-based technique were investigated and reported [32].



M. Rimašauskas et al. [16] developed impregnation process of CCF tow with a mixture solution of PLA thermoplastic pellets and dichloromethane with the ratio concentration of 90 g/10 g, respectively and prepared CCFRTP using FDM technique and achieved maximum tensile strength of 165 MPa. M. Heidari-Rarani et al. [33] prepared CCF reinforced PLA thermoplastic composite using FDM with the modification in the extruder design and achieved maximum tensile and flexural strength of 61.4 MPa and 152.1 MPa, respectively. N. Maqsood [21] prepared PLA- SCFR printed with CCF composite using FDM 3D printing technique and achieved the maximum tensile strength and Young's modulus of 227.56 MPa and 27.93 GPa, respectively. C. Yang [34] prepared CCF reinforced ABS thermoplastic composite and attained flexural strength and modulus almost six times higher than the conventional ABS material.

In the above reported researches, mostly studies have been performed either on the SCFR or CCFR composites and optimum strengths have been achieved and no research has been made on the combination of SCFR with CCFR composite and to study their fabrication, processability and mechanical performance. So, it would be of great interest to combine the SCFR with CCFR to form CCRFTC material and to study their fabrication, process parameters and mechanical performance. In this study, we have combined the SCFR with the CCFR to form a composite using FDM technique and discussed the complications faced during the fabrication process. Furthermore, the main delamination caused during the bending test has been reported and further work and improvement in the manufacturing process is also presented.

2. Materials, fabrication process and experimental set-up

2.1. Materials

Commercially available XT-CF20 (ColorFabb) 3D printing filament having diameter of 1.75 mm was used as a matrix material. XT-CF20 is a composite material made from the combination of PLA and short carbon fibers mainly 20% wt. carbon fiber content having flexural modulus and tensile strength of 6.2 GPa and 76 MPa, respectively (molded specimen) [35]. CCF tow T300B-3000 (3000 fibers in a tow, having diameter of one fiber equals to 7 μ m) made of polyacrylonitrile from Toray company having tensile strength, Young's modulus and density of 3530 MPa, 230 GPa and of 1.76 g/cm, respectively was selected as reinforcement material [36]. As, standard spool of CCF cannot be able to used directly for the printing. Therefore, it is essential to impregnate standard CCF tow before the printing process. Same procedure was followed as discussed previously by the author [16]. The standard non-impregnated CCF tow was impregnated in the solution of PLA pellets and di-methyl chloride (CH2Cl2) for better printing quality and performance.

2.2. 3D printing process and parameters

The specimen geometry was modelled using CAD software Pro-Engineer wildfire 5.0 and imported as STL file which was further exported to Simplify 3D software for printing using optimized printing parameters. MeCreator 2 3D printer from Geeetech was used for the fabrication of CCFRTP specimens. The printer was used due to its simplicity and ease of use. Furthermore, the extrusion device of the FDM 3D printer was modified using custom made extrusion process. Two inputs and one output were designed in the extrusion system. XT-CF20 filament having diameter of 1.75 mm and impregnated CCF were injected through two separate inputs using material driver, where they combined each other in the heating unit through guide pipe. The two different materials matrix and reinforcement fused together in the extruder and made a bond with each other where they pushed towards the printing nozzle of the finally extruded through the nozzle and printed on the building platform made of borosilicate glass which was mounted on aluminum plate. The schematic view of 3D printing of CCFRTC process is shown in Fig. 2. 3D printing parameters used for the



Fig. 2. Schematic view of 3D printing of CCFRTC process.

CCFRTC fabrication are presented in Table 1.

2.3. Flexural bending test measurement

Flexural test was performed to study and analyze the performance of 3D printed CCFRTC specimen fabricated using FDM technique. In this study, ASTM D790 [37] standard was used to perform flexural test. According to mentioned flexural testing standard, 5 specimens were prepared for the test. Three-point bending set-up (including one midway loading nose and two supports) on Tinius Olsen H25KT (capacity 25 kN) universal testing machine was used to perform the flexural test. According to standard for the flexural test, the specimens with dimensions of $123 \times 12.7 \times 3.2$ mm were fabricated. The test was performed using the crosshead motion rate and span support length of 1.35 mm/min and of 51.2 mm, respectively. The flexural strength and modulus was calculated using the equation (1) and equation (2), respectively.

$$\sigma_f = \frac{3PL}{2bd^2} \tag{1}$$

where, σ_f = flexural stress, P = load at a given point on the loaddeflection curve (N), L = support span (mm), b = width of tested beam (mm) and d = depth of tested beam (mm)

$$E_f = \frac{(\sigma_{f2} - \sigma_{f1})}{(\varepsilon_{f2} - \varepsilon_{f1})}$$
(2)

where, E_f = flexural modulus, σ_{f2} and σ_{f1} are the flexural stresses at the predefined points on the load deflection curve and ε_{f2} and ε_{f1} are the flexural strain values at the predetermined points on the load deflection curve.

 Table 1

 3D printing parameters used for CCFRTC specimens.

Nozzle diameter	1.5 mm
Extrusion multiplier	0.5
Extrusion width	1.5 mm
Layer height	0.5 mm
Printing speed	3.0 mm/s
Extruder temperature	250 ⁰ C
Bed temperature	90 ⁰ C
Fan speed	60%

2.4. Fracture interface study of the specimen

Fracture interface study was performed on the specimens using microscope (Delta Optical Smart) micrographs after performing flexural test. This study was performed to examine the behavior of deposited 3D printed layers and failure mode of fibers caused in a composite after performing the test.

3. Result and discussion

3.1. Observation of 3D printed composite specimen

The CCFRTC specimens were fabricated using FDM 3D printing technique. The matrix filament was extruded at a temperature of 250° C. The extruding temperature was selected on the basis of recommended temperature mentioned by the producer. Extrusion width, layer height and printing speed were kept constant throughout the printing process. Matrix filament was melted in the extruder at a temperature of 250° C where they directly fused with the impregnated CCF that is passing directly through the printing nozzle and made a bond, extruded and printed on the build platform.

After the fabrication process, the 3D printed CCFRTC specimen was examined through microscope's micrograph to observe the morphology of printed layers. Fig. 3 shows the microscope's micrograph of deposited layers and lines. Total number of layers and lines counted were to 8 and 7, respectively. From the figure, some void gaps can be clearly seen after the printing in the layers of composite part. Irregularity can also be seen in the printed layers. This may be due to the non-uniform printing and extrusion process. During the extrusion process, sometimes the matrix during its semi-melted state extruded non-uniformly through the printing nozzle that creates such voids. The layers are connected and bonded together through the matrix material. While, the lines printed of the composite part showed more uniform distribution compared to the layers. The lines of the composite part and bonded together through the extruded SCFR matrix material.

The approximation of content of carbon fiber in the matrix was calculated using the length of tool path of the specimen and content measured considered as the weight ratio of carbon fiber to composite specimen [21,29]. Thus, from the scheming, approximately 22% carbon fiber content was calculated for the 3D printed CCFRTC part and it is worth to mention that carbon fiber content from SCFR matrix filament was not included in the calculations.

3.2. Flexural response of composite specimen

Flexural bending test was carried out to study the flexural properties of prepared 3D printed CCFRTC specimen. According to the ASTM D790 [37] standard, flexural test would be only valid, if the specimen breakage in the outer region occurred within the 5% strain limit. In this experiment, the specimen breakage occurred within 5% strain limit. Three point flexural bending setup and typical flexural stress-strain curve are presented in Fig. 4 (a & b). From the stress-strain curve, it can be seen that CCFRTC specimen achieved maximum flexural strength of 134.58 MPa and flexural modulus of 12.26 GPa. In comparison to pure PLA and SCFR filaments [21], the flexural strength is increased by 67.79% and 73.31%, respectively. Table 2 presents the flexural properties measured of composite specimen.

During the test, when the CCFRTC specimen undergoes bending, upon the applied load, delamination occurred towards the sides of applied load between the layers instead of breakage or fracture in the composite part. The sudden breakage and rapid decrease in the stress value was also noticed during the test with a high standard deviation values. Such fluctuation may be caused due to poor interfacial bonding between the 3D printed composite layers.



Fig. 3. 3D printed specimen (a) fabricated for flexural test and microscope's micrograph of (b) deposited layers and (c) deposited lines.



Fig. 4. 3D printed CCFRTC specimen (a) 3 point flexural bending test (b) typical average stress-strain curve and (c) fracture interface microscope's micrograph after performing flexural test.

 Table 2

 Result of flexural properties measured.

Specimen	Flexural strength (MPa)	Flexural Modulus (GPa)	
CCFRTC	134.58 ± 5.93	12.26 ± 0.86	

3.3. Fracture interface observation

Based on experimental result flexural bending test and delamination caused during the test, it was decided to study the fracture interface of CCFRTC part to observe the deformation, delamination and fracture occurrence during the test. To examine such existence, fracture interface of was observed using microscope's micrograph to explore the specimen interfacial adhesion between the matrix and reinforcement. Fig. 4c

presents the microscope's micrograph of the composite specimen after performing the flexural test. From the figure, it can be seen that delamination occurred instead of rupture. This usually happen due to poor interfacial bonding between the printed layers. During the mechanical test, the lines of the composite part getting separated upon the applied load creating gaps between the layers toward the sides. The impregnated CCF displaced from the layers but didn't breaks. The delamination occurrence started from the upper layer lines and shifted towards the bottom lines. In each case, the same behavior has been seen by forming the slits in each layer line. This is due to insufficient bonding between the matrix and the fiber that indicate poor adhesion.

4. Future suggested work and recommendations

CCF reinforced with SCFR composite reported better flexural strength and modulus levels, but the main problem caused was the poor interfacial bonding between the matrix material and reinforcement. High tensile strength has been also achieved up to 227 MPa previously with the same composite material [21]. The composite part with the same material with improved adhesion and interfacial bonding could be made using more optimized and altering the printing parameters. The extrusion temperature may be varying with the decrease in extrusion width to make better bonding between them. The settling time after the fabrication process should be increase and the rapid cooling of the printed parts must be avoid in order to get sufficient time to made a bond that could result in the better adhesion and performance and to evade delamination during bending. 3D printed CCFRTC with improved structure still has great potential to be used in high engineering applications.

5. Conclusion

CCFRTC structure was prepared by the combination of SCFR with CCFR using FDM technique. After the fabrication process, the distribution of layers and lines in a 3D printed specimen was analyzed using microscope's micrographs. The layers distributions showed some void areas due to non-uniform extrusion of matrix material. During the flexural bending test, the composite specimen undergoes delamination instead of breakage. Microscope's micrographs of the specimen after performing the flexural test exposed that this happened due to weak and insufficient interfacial bonding between the matrix material and reinforcement which created gaps and separated from each other. This bonding of the CCFRTC could be improved using optimized printing parameters and could has potential to be use in structural applications.

Author contributions

Nabeel Maqsood: Conceptualization, Methodology, Software. Data curation, Writing – original draft preparation. Writing- Reviewing and Editing, Software, Validation: Marius Rimašauskas: Conceptualization, Methodology, Software. Visualization, Investigation. Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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