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# Characterization of continuous carbon fibre reinforced 3D printed polymer composites with varying fibre volume fractions

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# ABSTRACT

Fused deposition modelling (FDM) is one of the most popular additive manufacturing (AM) technique which is used to investigate the elastic properties of 3D printed polyamide-based polymer composites structures. The aim of this work is to study the mechanical properties of continuous carbon fibre reinforced polyamide polymer composite samples using tensile and flexural testing by varying the fibre volume contents with applying pressure, temperature and holding the samples for 60 minutes in the platen press. The results showed that the strength and stiffness increased with the increase in fibre volume content (fraction). Hot pressed samples exhibited the increase in tensile strength by about 27 % and elastic modulus by 11 % because of increasing the fibre volume fraction from 29 % to 35%. Synergetic effect of both short and continuous carbon fibre was also studied, and it was observed that the tensile properties were higher for the samples reinforced with short and continuous fibre than only continuous fibre polymer composites. Effects of voids on 3D printed continuous carbon fibre-reinforced polymer composites were quantified. A microstructure study of the 3D printed polymer composites was carried out using scanning electron microscope (SEM). Following SEM analysis on the tested specimens, it was observed that there was a strong correlation between the mechanical properties and the microstructure. Fibre volume fraction was measured using acid digestion method to determine the amount of fibre contents before and after hot pressing (compaction). From Micro- Computed Tomography (uCT) it was confirmed that hot pressing reduced the void content which in return increased the strength and modulus.

#### 1. Introduction

3D printing technology first emerged in 1986 and is rapidly growing in many industries because of its flexibility to fabricate complex geometries with no expensive tooling required. Since then, a lot has changed in the last few decades ranging from prototyping to the final functional product. 3D printing has some advantages as it is fast from designing to prototype, complex geometries can be manufactured with minimum wastage of materials as compared to traditional manufacturing techniques [1,2]. Continuous carbon fibre reinforced (CCFR) polymer composites are light weight and strong materials which have a wide range of applications in automotive, aerospace and medical industries. Reinforcements may be continuous or discontinuous which include carbon, glass and aramid fibre. Continuous carbon fibre reinforced polymer composites are replacing metals as a substitute due to its high strength and stiffness, light weight, excellent fatigue and wear resistance [3,4]. Due to these exceptional properties, CCFR polymer composites are widely used by aerospace, automobile and sports industries. CCFR composites are highly anisotropic because of this it allows the recognition of various design [5].

AM technology is expanding day by day in many industries because of manufacturing geometries with minimum production cost and time. One of the major concerns (limitation) of the FDM is the lower mechanical properties of 3D printed polymer materials which include

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Acrylonitrile Butadiene Styrene (ABS), polyether ether ketone (PEEK), Polylactic Acid (PLA) and Nylon. Nylon along with continuous carbon fibre is used to fabricate polymer composite. Strength and stiffness can be increased by adding reinforcement which can be either short and/or continuous fibre providing bridging effect to achieve desire mechanical properties. Faisal et al. [7] studied the effect of material formulation on mechanical performance and bioactive potential of PEEK and hydroxyapatite composites. It was found that in terms of mechanical performance no significant difference was noticed, although it showed better bioactive performance in comparison to pure PEEK. Yong et al. [6] studied the synergetic effect of both short carbon fibre (SCF) and continuous carbon fibre reinforcement on the mechanical properties for 3D printed polymer composites using FDM technique. It was concluded that the tensile strength was increased but there was detrimental effect on the elastic modulus of both SCF and CCFR.

Process parameters like layer thickness, printing (extruder) temperature, build orientation, infill angle, infill density, printing speed, fibre volume contents affect different performance of the samples manufactured through FDM technique. Chacon et al. [8] studied the effect of process parameters on mechanical performance of 3D printed tensile and flexural polymer composites samples reinforced with continuous carbon fibre. It was observed that the effect of layer thickness on the mechanical properties was marginally significant. Strength and stiffness were increased with the increase in fibre volume content with build orientation in the longitudinal direction with maximum area on the print bed. Hao et al. [9] observed that the tensile strength of composites samples reinforced with continuous carbon fibre decrease with an increase of layer height from 0.2 mm to 0.4 mm and extrusion width from 0.86 mm to 1.5 mm. It was also observed that the failure occurs due to the fibre pull out as SEM confirms the weak interface between the fibre and the matrix. Additionally, increasing printing temperature from 190 °C to 230 °C and printing speed from 50 mm/min to 400 mm/min decrease the tensile strength of the PLA base composite samples.

Voids also referred as porosity can appear as induced defect at multiple length in 3D printed parts (a) micro voids within fibre and matrix filament, (b) macro voids between the layers (c) meso voids between fibre bundle after deposited with in a layer. Voids are formed primarily due to entrapment of air and due to moisture absorption during the material storage and processing. Porosity and bond quality between the layers are the main concern areas for researchers in fibre reinforced composite parts fabricated through AM. This issue has been addressed by researchers through the addition of fillers, flake, particle reinforcement. Some of these procedures have been effective in reduction of the porosity by 10 % or less than 10%. Tekinalp et al. [10] revealed from his microstructure-mechanical property relationship that a relative high porosity (20%) is observed in 3D printed composites as compared to the parts fabricated through compression moulding, yet both exhibits comparable strength and modulus. The difference in strength of the two types of fibre reinforced samples was marginal, with fibre placement at 0-90° in samples developed through FDM versus the samples in which fibre orientation was random prepared through compression moulding, compensating for some of the strength loss from porosity. Hou et al. [11] studied that porosity had a great influence on the mechanical properties of 3D printed parts and observed pore distribution under different fibre contents from the experimental data. It was observed that the porosity increased with the increase of fibre contents which led to the creation of larger pores which affect the mechanical properties.

Hui et al. [12] investigated the influence of hot press and mixed fibre angles on the mechanical properties of 3D printed composites with varying pressure, temperature and time. It was concluded that hot pressing considerably increased the mechanical properties of 3D printed carbon fibre composites. The hot-pressed composite samples at 200 °C exhibits higher tensile strength and elastic modulus than non-heated samples. Also, at a pressure of 200 kPa and 30 minutes withholding time showed the highest strength and modulus due to strong interface bonding by removing the air gaps induced during printing of the hotpressed composite. In another study by Masahito et al. [13] reported the 3D hot compaction with a roller of continuous carbon fibre reinforced thermoplastic composite against the printer build platform immediately after the printing to reduce voids and improve adhesion between the layers. The hot compacted tensile and bending specimens showed superior properties than non-compacted samples. The void content fractions for compact samples were reduced to 3% from 10 % for non-compact samples, indicating that the voids were discharged by hot compaction during 3D printing.

In comparison to conventional methods, FDM technique is simple in design, and operation but lack the ability to pressurize the samples during 3D printing. In this work, novel technique of compacting (pressurizing) the samples using hot press machine after printing was used to increase the fibre contents, reduce the void contents which in return increase the mechanical properties. The fibre volume fraction was



Fig. 1. Flat plate with estimation of plastic and fibre estimation calculated by Eiger software.



Fig. 2. 3D printed polymer composite plate after pressing and extracting the rectangular tensile samples using water jetting.

calculated by acid digestion method for each specimen type before and after compaction and was compared to the Eiger software which also gave the estimation of the amount of fibre and polymer (Nylon) used. However, the Eiger did not consider the amount of binding agent present in each fibre filament or the volume of short fibre present in the matrix (Onyx) but from the literature it is clear that it contains 40% of fibre [14].

Previously research has been conducted to improve the strength and modulus of 3D printed polymer composites. However, in this study fibre volume fractions were controlled (increased) by using two methods. One method was by varying the number of fibre layers in the composite specimens with controlled fibre orientation having maximum number of fibre layers in the specimen. In second technique, fibre volume fraction was increased using hot platen pressed machine by applying pressure at 130 °C to investigate the mechanical properties by improving the interface bonding between fibre and matrix and reducing the voids that were induced during printing. Subsequently, tensile strength and elastic modulus of hot press samples were compared to the non-press samples and there was significant improvement in the mechanical properties which were almost equivalent to the samples manufactured using conventional manufacturing techniques. This technique will help researchers and designers to enrich the mechanical properties of 3D printed polymer composite for application required in high strength structures. Furthermore, SEM was employed to study the microstructure behaviour as well as voids in the specimen.

## 2. Materials and methods (Experimental procedures)

## 2.1. Specimens fabrication

In this work, different types of polymer composite specimens were fabricated using a Mark forged Two 3D printer, and mechanical testing was conducted using a tensile machine. The first type of specimens were reinforced at different orientations with continuous carbon fibre; this is a proprietary material of the Mark forged. Fibre volume fraction was varied for these specimens by changing the number of fibre layer in the polymer composite. The second type of specimens were printing using both Onyx (chopped carbon fibre) with continuous carbon fibre to study the synergetic effect on the mechanical properties. Also, a large plate was printed and subsequently compressed to achieve high fibre volume content before test samples were extracted according to the ASTM standard.

3D printed non-press polymer specimens were fabricated using zerodegree, Quasi-isotropic (0,  $\pm$  45,90) and  $\pm$  45-degree fibre orientation with each set consisted of five samples according to the ASTM standard. All the samples were printed with 100 % fill density and solid fill structure with 0.125 mm layer thickness. For the press samples, a large plate having dimension of  $190 \times 130$  mm (Fig. 1) was printed with four Nylon layers on the top and four layers on the bottom. The top and bottom Nylon layers was removed with a sharp knife to achieve maximum fibre volume fraction before placing the plate in the platen press. A metal plate was cut having a thickness of 3.5 mm in which the 3D printed part was placed and was press with a hydraulic head pressure of 50 bar and maintaining temperature of 130 °C with 1 h holding time in the press machine. Before these conditions, trials were carried out to achieve the best possible temperature and pressure at which the desire thickness was achieved with better surface finish. After pressing the large plate was kept for two hours in the press machine, the plate was then removed after cooling naturally as it might distort the shape if removed straight away and residual stresses might also appear. The tensile samples were extracted from the large plate using water jet cutting having dimension of  $165 \times 19$  mm as shown in Fig. 2. End tabs were bonded to these samples using Araldite adhesive. Tabs prevented damage to fibres from jaw faces which needed to be gripped in a range of manual or hydraulic grips with serrated jaw faces. Bonding of tabs is generally time consuming and expensive and difficult with thermoplastic composites because of low adhesion. To overcome adhesion problem, the outer layers of nylon was removed so the tabs was bonded directly the rough surface of the specimens with minimum glue thickness.

# 2.2. The Markforged two 3D printer

The Markforged Two 3D printer is the first commercial FDM based composite printer was developed by Markforged in 2014, which offers printing head with two separate extrusion nozzles for polymer and continuous reinforcing fibre. Both the nozzles do not work simultaneously, rather one stops while other works. The printing process K. Saeed et al.

#### Table 1

Testing parameter for tensile and flexural specimen.

Categories	Tensile test	Flexural test
Testing speed	2 mm/min	2 mm/min
Load cell capacity	100 kN	50 kN
Sampling frequency	20 Hz	20 Hz
Distance between grips	115 mm	-
Distance between two points	-	102 mm

consists of two stages. The first step is to print Nylon (matrix) with the plastic dispensing nozzle with a temperature of 270 °C, secondly the fibre reinforcement in the matrix is applied with a hot end temperature of 250 °C on a non-heated print bed with a maximum printable size of 302 x132 x154 mm. The Mark forged Two 3D printer allows continuous fibre reinforcement to be positioned in different patterns (orientation) on layer to layer basis in the slicing software.

Mark forged Two 3D printer has few limitation outline below:

- Printing speed are predetermined and can not be change along with the nozzle temperature for both the nozzles. The nylon filament temperature is 270 °C and 250 °C for fibre filament to melt the fibre filament resin.
- First and final layers can not be printed of fibre, which increases the amount of resin volume in the printed part.
- Build plateform can not be heated and must be kept at room temperature.
- A layer thickness of 0.125 mm is used for printing carbon fibre while 0.10 mm thick layer is printed with glass fibre and Kevlar.

## 3. Results and discussion

Tensile and flexural tests were performed using an Instron machine with 100 kN capacity. Extensometers were used to measure the strain in longitudinal and transverse direction to measure Poison's ratio for tensile samples. Test conditions for tensile and flexural test are summarized in Table 1. Density and fibre volume fraction were measured before and after pressing in accordance with the ASTM D-792 and ASTM D-3171 respectively [15,16]. Matrix digestion method was used using 60% w/w Nitric acid at 70 °C with magnetic stirring for 3 h. After this, the samples were rinsed with deionized (DI) water and a final wash with acetone. The fibre was then dried in the oven and the mass of the fibre was calculated. The density of the carbon fibre calculated was 1.766 g/

cm<sup>3</sup> as calculated the same by Fuji et al. [17].

#### 3.1. Tensile test results

The performance of 3D printed specimens was evaluated by performing tensile tests according to ASTM standard [18]. Samples were tested at a rate of 2 mm/min and data from the load cell and extensometer was collected at a frequency rate of 20 Hz. Mechanical properties of the 3D printed polymer composites were compared with the supplier data sheet and compared to other values available in the literature. Van der Klift et al. [19] reported the discontinuities, and from the experimental results it was obvious that these discontinuities of the fibres leads to premature failure in the areas where fibre were absent or couldnot be printed. Dickson et al. [20] addressed that problem by fabricating test samples in which the fibre ends were laid down beyond the end tabs. That approach resulted in the increase of tensile strength without modifying the samples between printing and testing. In this study to overcome this problem, a large plate was printed, and samples were extracted using water jet machine before testing using the tensile machine.

In 3D printing, no pressure is applied during the layers deposition upon each other which play a fundamental role on the manufacturing of laminated parts. The absence of pressure leads to porosity and results into the presence of defects which affect the strength and stiffness of the structure. Justo et al. [21] investigated the plane strength and stiffness properties of nylon based composites using tensile and compression test. From the results, it was observed that the AM specimens are not comparable in strength to the specimen fabricated using conventional methods, but there was significant improvement in strength and stiffness as compared to unreinforced specimen. Caminero et al. [22] studied the interlaminar bonding performance using short beam shear test on 3D printed polymer composites reinforced with carbon, glass and Kevlar. The results indicate that it is still a challenge to increase interlaminar shear performance of 3D printed specimen in comparison to autoclave technique. In another research by Caminero et al. [23], impact damage resistance of 3D printed thermoplastic composite are carried out to determine impact strength. Glass fibre reinforced samples were found to exhibit the best impact performance followed by carbon fibre. Furthermore, it was also observed that impact strength increases with the increase in fibre volume content. In this work, tensile tests were carried out on several different samples including reinforcing short carbon fibre along with continuous fibre, dog bone and rectangular samples which were pressed after 3D printing:



Fig. 3. Tensile strength comparison of continous carbon fibre with synergetic effect of short and continous carbon fibre.

#### Table 2

Tensile strength and elastic modulus comparison of press 3D printed polymer composites using platen machine.

Fibre Angle	Hot press temp (°C)	Time (minutes)	Pressure (bar)	Tensile strength (MPa)	Elastic Modulus (GPa)
Unidirectional (0°)	130 °C	60	50	$\begin{array}{c} \textbf{768.35} \pm \\ \textbf{39} \end{array}$	80.39
$\pm$ 45 orientation	130 °C	60	50	$\begin{array}{c} 597.45 \pm \\ 25 \end{array}$	71.68
Quasi Isotropic $(0^{\circ}, \pm 45, 90^{\circ})$	130 °C	60	50	$\begin{array}{c} \textbf{655.94} \pm \\ \textbf{31} \end{array}$	77.91

#### 3.2. Synergetic effect of short and continuous carbon fibre

Composite characterization is a challenging task as it is anisotropic in nature, meaning that bulk properties vary strongly with direction. The properties of polymer matrix composites are also influenced by temperature and humidity. The mechanical properties of continuous carbon tows and short carbon fibre reinforced with nylon was evaluated by conducting tensile tests. From the results in Fig. 3, it is obvious that the synergetic effect of short and continuous carbon fibre properties is superior to the individual continuous carbon fibre. The maximum tensile strength and elastic modulus achieved was 482 MPa and 61 GPa as compared to the continuous carbon fibre specimen which was 416 MPa and 56 GPa, respectively. The increase in mechanical properties was due to the increase in the fibre volume fraction from 28% to 31 % which was calculated using acid digestion method.

# 3.3. Hot pressed dog bone shape samples

Table 2 shows the mechanical properties of the 3D printed composite pressed samples for different fibre orientation and was compared to unpress sample that are shown in Fig. 5 and Fig. 6. It is observed that the pressed samples showed the best performance in terms of tensile strength and elastic modulus. The maximum value of unpressed samples obtained for tensile strength and elastic modulus was 588.27 MPa and 73.43 GPa respectively, for dog bone shape samples with fibre at

unidirectional (0-degree) orientation. Dog bone shaped test samples were extracted from the large press plate using water jet machine as printed samples resulted in failure at the radial section due to premature failure. To resolve this issue water jet was used and the samples extracted are shown in Fig. 4. Melenka et al. [24] predicted the elastic modulus of 3D printed parts reinforced with continuous carbon fibre using volume average stiffness (VAS) method. Samples manufactured by 3D printing using Mark forged Two have voids which is constant for all types of specimen configuration in this study. Voids contents was approximately 4.2 % for non presses samples and reduced to only 0.26 % for pressed samples which was measured from microsectionong analysis discussed later in this paper.

# 3.4. Hot pressed rectangular samples

A large plate with dimension of 190 mm x 125 mm was printed with Mark forged Two 3D printer. After that, 3D printed plate was placed in an aluminium mould (3.5 mm thick) that matches the dimension of the plate. Next, it was heated and compressed by a hot-press machine. During hot-pressing, the temperature was maintained at 130 °C, pressure in the hydraulic press circuit was maintained at 50 kPa for 60 min and then left until it cooled naturally to room temperature. It must be noted that excessive pressure for more time may destroy the path of the continuous carbon fibre which may lead to premature failure. Samples were extracted from the plate as shown in Fig. 2.

It was observed that the tensile strength and elastic modulus of 3D printed pressed samples increased which resulted in a brittle fracture as the composite becomes stiffer. Tensile strength and elastic modulus of unidirectional pressed 3D printed polymer composites were 768.35  $\pm$  39 MPa and 80.39  $\pm$  1.2 GPa respectively. Tensile strength and elastic modulus were approximately 27% higher than that of unpressed 3D printed samples due to the lower void fraction and increased adhesion between the layers as reported by the author [25]. Tensile strength and tensile modulus of 800 MPa and 60 GPa has been reported by the manufacturer [26]. Fracture rupture of the pressed samples occur near the gripping area and the main reason was the delamination of the layers due to the brittle nature of the composite samples. In most samples, the fracture occurs near the gripping region which means that the samples



Fig. 4. Dog bone shaped tensile samples extracted from large plate using water jet machine.



Fig. 5. Tensile strength comparison between press and unpress samples for different fibre orientation.



Fig. 6. Elastic Modulus comparison between press and unpress samples for different fibre orientation.

were strong enough to bear the load but fail due to the stress concentration factor.

The pressed specimen using platen press exhibit higher mechanical property values than those of the 3D printed unpressed samples because of the reduction in porosity and increasing the interlaminar strength. Hot pressing was carried out using platen press to the 3D printed plate as a post process. Fig. 5 and Fig. 6 shows the tensile strength and Elastic modulus comparison for pressed and unpressed samples for different fibre orientation respectively while Fig. 7 shows the tensile strength and elastic modulus comparison with the variation in fibre volume fraction.

# 3.5. Flexural test

Flexural specimens were fabricated according to ASTM D790-15 standard, and the fibre volume fraction was varied by varying the number of fibre layers. The test specimen geometry was design in Solid Edge which is a computer aided design (CAD) software for 3D modelling. Effect of fibre content on the flexural properties was studied by modifying the number of fibre layers using isotropic configuration to vary fibre volume fraction. Five specimens of each type configuration were tested according to the standard. All the specimens were printed with 5 top/bottom and 1 wall layers. In Eiger, it is possible to change the fill density but for these test samples 100% fill density was kept the same. A universal tensile machine was used to apply the load to the specimens. Fig. 8 shows the stress–strain curve for flexural test with 24 layers of carbon fibre having good consistency. Flexural tests results were analysed and the factors that affect the flexural modulus and proportional limit responses were identified. Flexural strength and flexural modulus of 478.92 MPa and 45.78 GPa was recorded respectively from the three-point bending test which are summarized in Table 3.

Tianyu et al. [27] studied the flexural properties of AM specimen and the experimental results shows that the concentric infill pattern show high flexural strength and energy absorption capacity with 43.5 % weight carbon fibre. Miguel et al. [28] evaluated the compressive and flexural properties of additively manufactured specimen. There was significant improvement in flexural strength as compared to compressive strength. Premature failure was observed due to the delamination of layers caused during manufacturing process.

Impact testing was performed by subjecting the 3D printed samples to a swinging pendulum. This impact test measures the energy absorbed by the pendulum and is used an indicator of material toughness.



Fig. 7. Comparison of tensile strength and elastic modulus with increasing (varying) fibre volume fraction.



Fig. 8. Stress strain curve for flexural test of 3D printed composite specimens with 24 layers of carbon fibre.

Table 3		
Flexural properties of 3D printed polyme	r composites with	different fibre vol-
ume fraction.		

Specimen type	Flexural modulus	St deviation	Proportional limit (flexural strength)	St deviation
24 fibre layers	45.78 GPa	2.56	478.92 MPa	4.95
16 fibre layers	15.81 GPa	4.09	295.51 MPa	5.82

Specimens were fabricated according to ASTM D256 standard having a V-notch in the middle of the specimen. A notched Izod impact test was also conducted at room temperature using an impact pendulum (Zwick)

according to ASTM D256 standard. In this work only samples printed with zero-degree orientation were studied with average impact energy absorption rate of 49 J. Impact testing is normally done to determine the amount of energy absorbed by the specimen because of change in potential energy related to the difference in the height of the swinging pendulum before it is released and the maximum height it reaches after the impact.

# 4. Fibre volume fraction

The fibre volume content can be increased or decreased by varying the number fibre layers in 3D printed samples. This also increase or decrease the thickness of the sample accordingly. Beside this, individual layers of fibre can be changed to any desired orientation within the same

## Table 4

Fibre volume fraction contents calculated by acid digestion method for 3D printed polymer composites having 24 layers of fibre (dog bone shape samples).

Samples (FVF)	Density (kg/m <sup>3</sup> )	Fibre content Volume (V <sub>f</sub> %)	Resin content Volume (V <sub>r</sub> %)	Void content (%)
zero-degree orientation	1194.07	34.75	65.25	8.35
Quasi orientation (0, $\pm$ 45,90)	1250.79	34.91	65.09	4.55
±45-degree orientation	1261.44	34.84	65.16	6.92

geometry. The fibre volume ratio for each sample was calculated from the fibre and nylon volumes stated by the Eiger software. Acid digestion method was used to find the fibre volume fraction (FVF) of three samples of each type on a hot plate at 280  $^\circ$ C for 3 hours in accordance to

procedure described by ASTM D 3171 [16]. The fibre volume fraction of each pressed plate was calculated according to ASTM standard and is summarized in Table 4. Before calculating fibre volume fraction density of resin, fibre and the composite samples was calculated. The fibre volume fraction calculated for zero degree, quasi-isotropic and  $\pm$  45-degree orientation samples were the estimated 34.75 %, 34.91 % and 34.84 % respectively and the same is reported by Blok et al. [29].

## 5. Micro sectioning analysis

Void content analysis was performed in accordance with AITM 4–0005 and were evaluated after the specimen is polished. Micro sectioning analysis was carried out for both unpressed and pressed samples. The microsections were cut from the samples using a diamond saw (wet) and the specimens were marked-up prior to machining. After machining the specimens were potted in resin to aid with specimen polishing. The microsections were ground and polished through four stages. The first two stages consist of grinding the specimen surface



Fig. 9. potted 3D printed polymer composites specimen for micro sectioning.



Fig. 10. Thickness measurement and void content analysis for un pressed specimen [Magnification 50X].



Fig. 11. Thickness measurement and void content analysis for press specimen [Magnification 50X].



Fig. 12. Fiber orintation at 0/90° with magification of 50X.



Fig. 13. Micro graph of 3D printed polymer composite specimen showing continuous strands of carbon fibres b) voids between the layers c) Polyamide observed in CF filament.

using SiC paper of 600 grit and 1200 grit with water. The final two stages aimed to improve specimen surface to a mirror polish using polishing cloths and self-lubricating polycrystalline diamond suspensions. The completed microsections as shown in Fig. 9 were then examined microscopically and the appropriate images recorded. Stream basic (Image processing software) was used to capture the images of the samples which were extracted from tested tensile specimens.

Fig. 12 shows the distribution of fibre and resin in the composite samples with fibre orientation in longitudinal and transverse direction.

Also, it should be noted that the void content of 0.26 % measured for press samples (Fig. 11) as compared to non-pressed samples which has void contents of 3.96 % as shown in Fig. 10 at 50X magnification. Voids or cluster of voids are present throughout the specimen for un pressed samples along with resin rich areas.

# 6. Scanning electron microscopy (SEM)

Scanning electron microscopy was carried out to analyse the fracture



Fig. 14. Fibre orientation in longitudinal,  $\pm$  45° and transverse direction.



Fig. 15. SEM image showing the fibre in the printed specimen after fracture.

behaviour of the fracture samples. SEM was performed to identify and verify the internal structure/print quality of the polymer composite and examine the fracture mechanism. The specimens were coated with Gold-Palladium target and was mounted with a conducting carbon tape. Carbon tape was also applied before SEM to avoid charging.

Fig. 13 shows that the failure occurs due to the fibre breakage along with some resin rich areas, matrix pull out can also be observed. The strands of broken fibres are observed in the fracture samples. Some of the fibres are broken and some of them are pulled out from the matrix. Fibre breakage was the main cause of the sample failure. From SEM examination, it clearly indicates porosity in the low pressure manufactured samples, which is the main cause for the reduction of strength in

3D printed polymer composites. It was noticed that the voids (porosity) are present between the fibre itself as well as between the printed layers. Porosity can be either unintentionally, which cannot be controlled in the well-prepared structure, or it can be engineered (controlled) for a particular function by changing the dimensions, size, or orientation. Fig. 14 shows the fibre orientation in longitudinal,  $\pm$  45 and transverse direction with fibre breakage and pull-out, while Fig. 15 shows the fibre breakage, resin rich region and distribution of fibre within the composite sample.



Fig. 16. µCT images showing dark regions as voids distribution of 3D printed polymer composite specimen.



Fig. 17. µCT images of 3D printed polymer composite after pressing using platen press with lower voids content.

# 7. Micro computed tomography (µCT)

Micro-computed tomography was carried out to observe the fibre orientation and level of porosity inside the specimen using SkyScan 1275 Bruker machine. The images were taken keeping X-ray detector at 3 MP (1944  $\times$  1536) with a 75  $\mu$ m pixel size. 20 – 100 kV range. A 10 W X-ray source with a less than 5  $\mu$ m spot size at 4 W target power. Data Viewer an imaging software of Bruker was used to process the images.

Samples were scanned before mechanical testing to calculate the porosity of 3D printed polymer composites. The largest derease in porosity was from 20 % to 4.5 % for pressed samples at 0-degree orientation using the platen press, which also exhibits high tensile strength and modulus. Fig. 16 shows the 2D  $\mu$ CT images recorded for contionous carbon fibre reinforced unpress samples. The side shows the nylon at 45° orientation while the fibres are in longitudnal direction. From the literature it is clear, that there is also porosity in the filament as resin are not accumulated fine around the fibre as it is impossible to achieve uniform fibre distribution throughout the spool. This leads to the fact that the porosity in 3D printed samples appear due to the non-adequate bonding between the layers as well as due to the non-uniform distribution of fibre in the individual fibre.

Voids in the 3D printed samples were extracted from the  $\mu$ CT images with dispersed voids observed in the specimen. A large number of voids

were distributed along the fibre direction which mainly depends on the path of the print. Less number of voids were observed (Fig. 17) in the pressed samples as most of the voids discharged during the press machine using platen press by applying pressure and maintaining a constant temperature. The void fractions calculated using micro sectioning was 4.2 % and 0.26 % for unpressed and press samples respectively.

The applied pressure after 3D printing resulted in the loss of porosity between the layer specially at the outer surface which was directly in contact with the hot plate during pressing. Fabienne et al. [30] characterize the filament and matrix interface by performing fragmentation tests on mono filament carbon fibre/resin composites along with micro CT observations on tested samples. From the cross-sectional observations, it was clear that triangular voids were seen as well the fibre breakage of the individual layer.

# 8. Conclusion

The main goal of this work was to study the effect of varying the carbon fibre contents in 3D printed specimens with polyamide-based composites, along with the application of pressure on 3D printed parts using platen press at a constant temperature for 1 hour. Conclusion drawn based on results obtained as below:

- 1. Highest strength and modulus obtained were for the hot-pressed samples with unidirectional fibre orientation compared to any other orientation. Reason is that the fibre in longitudinal orientation resists the stretching and bear the maximum load.
- 2. The hot-pressed samples at 130 °C, pressure of 50 bar with 1 hour holding time in the press machine shows highest tensile strength and elastic modulus as the layer's adhesion being improved which was verified through micro-CT analysis before and after the pressing. Also, the interface between the layers was improved by the escape of air gaps that were induced during printing.
- 3. Scanning electron microscope was used to see the fracture type and the fibre distribution. It was found from the mechanical testing that the tensile strength and elastic modulus was increased significantly (768.35 MPa, 80.39 GPa) for the hot-pressed samples as compared to non-pressed samples (604.34 MPa, 73.1 GPa). Flexural strength was also increased by increasing the number of fibre layers and shows linear relation with the fibre content.
- 4. From Micro CT analysis it was observed that the porosity decreased for the pressed sample; this was the assumed reason for the improvement of the mechanical properties.
- 5. Micro sectioning analysis shows that all the samples contain cracks with an average void contents of 3.96 % for the unidirectional samples produced without the application of any pressure.

In conclusion, mechanical properties of 3D printed polymer composites have been improved considerably due to increased fibre content and reduction (discharged) of void content due to hot pressing using platen press. This finding could increase the practical applications of 3D printed polymer composites using continuous carbon fibre.

# **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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