ARTICLE IN PRESS

Materials Today: Proceedings xxx (xxxx) xxx



Contents lists available at ScienceDirect

Materials Today: Proceedings

journal homepage: www.elsevier.com/locate/matpr



Strength and fracture behaviour of polymer matrix composite layered structures made by additive manufacturing

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ARTICLE INFO

Article history: Received 21 October 2019 Received in revised form 16 November 2019 Accepted 26 December 2019 Available online xxxx

Keywords:
Below-knee prosthesis
Additive manufacturing
Filament
Fracture
Strength
Direct deposition method

ABSTRACT

In this paper, studies on the tensile strength, fracture initiation and propagation behaviour of composite material layered shell structures manufactured by extrusion based additive manufacturing (AM) is reported. To understand the basic mechanical behaviour, simple tension tests have been conducted on ASTM-D638 specimens of carbon fiber and natural fiber reinforced polymer matrix composites made by AM in different modes as well as directly in filament form using a filament extruder. The tensile strengths reveal that the internal defects unique to additive manufacturing, such as lack of fusion (LOF) sites and voids, play crucial role in the macroscopic fracture. Based on the tensile test results, finite element analysis of shell structures typical of below-knee prosthetic sockets has been conducted to predict the fracture initiation sites and propagation paths. The analysis results have shown close correlation with the experimental observation of the fracture phenomenon in the prosthetic sockets given to pilot test amputees.

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Selection and peer-review under responsibility of the scientific committee of the 2nd International Conference on Recent Advances in Materials & Manufacturing Technologies.

1. Introduction

Additive Manufacturing (AM) is a technology that builds threedimensional (3D) complex geometry objects layer-by-layer from a variety of materials including thermoplastics, thermosetting polymer liquid resins, ceramics, metals and alloys using a variety of processes that fall into the categories of vat photopolymerization, powder bed fusion, material extrusion, material jetting, binder jetting and direct energy deposition processes. Among them, fused deposition modelling (FDM) is one of the most widely used and affordable AM technology due to simplicity of process involving extrusion of fusible thermoplastic polymers. FDM gives reasonable strength at low cost, acceptable surface finish and durable parts. The raw material to FDM is normally in the form of wire or filament of diameter 2.85 mm or 1.75 mm, based on the machine used. The thermoplastic polymer filament is fed into a melt-head, in which the filament is inductively heated to a suitable temperature above the glass transition temperature of the polymer. The polymer in semi-liquid state is extruded out of the melt-head through a nozzle with opening diameter ranging between 0.4 mm and 1.2 mm and is deposited on the build platform as per the path program supplied and to the control system that drives the dc motors responsible for x-y motion of the extrusion head and z-motion of the build platform.

2. Additive manufacturing of composite material structures

Most of the FDM technologies commercially available till date can manufacture parts in only polymer matrix material without any fiber reinforcement. The most popular polymers for FDM include acrylonitrile butadiene styrene (ABS), poly-lactic acid (PLA), nylon, polycarbonate (PC), linear low density polyethylene (LLDPE) and polycarbonate. Parts made solely of these materials perform well in a variety of applications yet they are not suitable for applications involving high mechanical loads, giving rise to the need to produce composite material parts through additive manufacturing route. The superiority of composite materials in terms of strength to weight ratio in comparison to unreinforced polymers or metals is well-known and is already in widespread industrial practice, wherein the fibers owing to their small cross section have fewer flaws as compared to bulk materials and exhibit

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https://doi.org/10.1016/j.matpr.2019.12.347

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Please cite this article as: S. P. Regalla, S. S. Karwa, S. Rajesh et al., Strength and fracture behaviour of polymer matrix composite layered structures made by additive manufacturing, Materials Today: Proceedings, https://doi.org/10.1016/j.matpr.2019.12.347

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higher strength along their lengths [1]. Manufacturing composite material parts through AM instead of the well-established conventional methods such as compression mould technique has several advantages including the ability to manufacture complex geometry parts with thin extended portions and faster processing. The efforts to make composite material parts through AM began only in recent times and researchers attempted making fiber reinforced plastic (FRP) composite through FDM with a variety of well-known fiber materials including carbon fiber and glass fiber. Tekinalp et al. [2] investigated processibility of short fiber (0.2–0.4 mm) carbon fiber reinforced ABS composites and studied the microstructure and mechanical performance in comparison to traditional compression moulded composites. They found that despite higher porosity, the tensile strength and modulus of AM samples were higher by 115% and 700% respectively and attributed it to better and consistent fiber orientation in AM. Similar results were observed on AM made specimens with carbon-fiber reinforcement in ABS matrix [3] and in other polymer matrices [4–8].

In the history of composite materials use over the several decades, the use of natural fibers has been seen as a welcome replacement to man-made fibers like carbon fibers and glass fibers due to the advantages of natural fibers including less weight, low cost, being inexpensive, having well-established supply-chains, easy recyclability and biodegradability. Researchers working with the traditional techniques of composite materials manufacturing have repeatedly reported that the chemical modifications of natural fibers can better improvise their interlocking and surface adhesion with polymer matrix thereby making them more suitable to automobile and plastics industries [2].

The research work to manufacture natural fiber reinforced polymer composites using AM is not fully yet investigated in the literature. Thus the present work reported in this paper is aimed to address this gap. Unlike the synthetic fibers such as carbon and glass fibers, which can be manufactured to the required diameter and length, the natural fibers occur innately in nature as short to medium length fibers and in diameters ranging upward of 50 um. Therefore, continuous fiber can be obtained only by weaving or stranding, which results in the net diameter to be well above the typical FDM nozzle diameters. Due to this reason, even though continuous fiber reinforcement is preferable for high mechanical properties, practically only short fiber reinforced plastic (SFRP) composites are viable. The SFRP composites have been made by the conventional techniques such as compression moulding previously and the parts made by this route provided moderately improved mechanical properties. They are commonly used for low-cost composite part fabrication. Hence it was felt worthwhile to investigate their manufacture through AM. The fiber distribution, fiber orientation and the length of the fibers decide the mechanical properties of the SFRP composites. In the present work, additive manufacturing through FDM process of ASTM-D368 tensile test specimens made with carbon as well as two different natural fibers, namely jute and coir (coconut) with polylactic acid (PLA) as matrix material was conducted and tensile tests were conducted on them. Tensile tests were also conducted directly on the filaments of composite materials.

3. Factors to be considered while manufacturing SFRP filaments

Extensive experimentation was done by the authors in the present work to manufacture raw material filaments for FDM using a commercial filament extruder (Noztek-Pro) with a variety of new natural or synthetic fibers and different combinations of matrix materials (Fig. 1). In order to be compatible with the commercial FDM machines, the innovative new composite material filament must be made with specified diameter of 1.75 mm or 2.85 mm



Fig. 1. Experimental raw material filament extrusion.

based on the FDM machine used and with only a small tolerance zone permissible on this diameter. Extruding consistent diameter filaments that are later to be used in an extrusion-based additive manufacturing machine (such as FDM) at the small-scale or laboratory level is a major difficulty. The first challenge here is to maintain uniform diameter in the filament material extruded from the filament extruder. The gravitational forces of the just extruded semi-solid filament results in elongation, with consequent reduction in diameter due to volume constancy and results in variable diameters. Variable diameter filament poses problems when it is inserted into the FDM machine for part manufacturing. The rollers, which are the only means to push the filament into the melt-head (or hot-end) of the FDM machine, contain peripheral grooves to grip and generate frictional pushing force. The roller pushing mechanism works as long as the filament has uniform diameter but it fails by slipping on the filament when diameter is smaller and gets stuck by indenting into the filament eventually leading to gouging of filament surface when the diameter of the filament is much larger. Several solution methods to overcome this problem were experimented in the present work to obtain a uniform and smooth extruded filament. One of the solutions implemented was to use cooling fans at the exit of the filament extrusion die so that the semi-solid extruded filament solidifies faster and thereby minimizes the chance of elongation due to gravitational pull. Second solution used was a room temperature water bath instead of cooling fans at the exit of the die, with which the cooling rate was even faster resulting in even more consistent diameters over sufficiently long lengths of filament extruded. In the case of the water bath, due to rapid cooling rates for solidification in case of some filament matrix materials such as nylon and PLA, shrinkage in diameter and breaking due to brittleness were observed. This may be due to a complex combination of amorphous and limited crystallinity resulting from rapid solidification rates (Fig. 2). More investigation in this aspect is in progress.

It was observed that the size consistency of extruded filament depends on the temperature at which it is extruded, force of ejection from the nozzle including the gravitational force, feeding rate in the hopper and glass transition temperature of the thermoplastics. Especially with composite material filaments, apart from getting a consistent filament it becomes important to keep a check on the fiber distribution in the matrix to get better mechanical properties. In mechanical mixing mode, where the beads of matrix material and the chopped fiber are pre-mixed in the required proportion and poured into the filament extruder, the buoyancy and shear strain rate differences during the melting phase make short fibers to be displaced resulting in uneven distribution of short

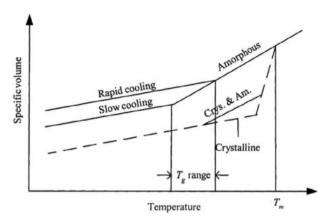


Fig. 2. Cooling curves for thermoplastics [9]

fibers in the filament. Introducing short fibers into the molten polymer matrix material at a downstream position in the extruder can be an alternative solution. Compatibilizers are used not only to promote interfacial properties between matrix and reinforcement but also to improve flexibility and handling of filaments. They improve physical bonding between polymer-polymer or fiber-polymer composites by concentrating at the interfaces [3]. Many researchers have studied effects of various compatibilizers for various fiber and polymer blends, one among them is the synthesized interfacial modifier agent, acrylic acid grafted polypropylene (AAGPP) in glass fiber reinforced nylon 6/polypropylene blends [4]. In SFRP, the critical fiber length (l_c) given by [10]:

$$l_c = \frac{\sigma_f \mathbf{d}}{2\tau_c} \tag{1}$$

which is important for optimum strengthening. Here $\sigma_{\rm f}$ is fiber ultimate strength, τ_c is either the fiber-matrix bond strength or shear yield strength of the matrix (whichever is lower) and d is fiber diameter. When the fiber length $l < l_c$, which is the characteristic of SFRP, the composite failure occurs at the interface whereas when $l > l_c$ the composite is called continuous fiber composite and in this case the fiber has to fail for the composite to fail [5]. Experiments conducted in the present work using a range of sizes of fiber on both sides of the critical length showed that although it is ideal to have continuous fiber for better strength, it is difficult embed long fibers in the FDM filaments during their manufacturing in the extruder. Even if such filaments are made with some success, unless the fiber is exactly centred in the filament, the buoyancy and hydrodynamics in the melt-head or extrusion head of the commercial FDM machine results in displacement of the fiber and eventual clogging of the FDM nozzle. More experimentation in this aspect can nevertheless help open up new possibilities. In the work reported in this paper, the fiber length has been maintained to be just above the l_c .

Fiber breakage instances have been reported in the literature [6] during processing and it was observed to arise from the interaction of fibers with polymers and surfaces of the processing equipment. It increased with the quantity of fiber and depended on the matrix material, the process conditions, and the fiber loading. For effective stress transfer the fiber breakage must be minimized through maintaining high aspect ratio (length to diameter ratio).

It is crucial to maintain the substrate or the previously completed layer at around the glass transition temperature to avoid its early solidification and to achieve full inter-layer bonding to avoid defects like delamination and warpage. This necessitates accurately choosing melt head temperature and built plate temperature. Optimization of these process parameters is possible using Taguchi design of experiments strategy combined with numerical modelling as simple one-dimensional transient thermal model to measure the thermal evolution using infrared imaging

[3]. It is well known that incorrect setting of other FDM process parameters including part orientation, layer thickness and road gap also can compromise mechanical properties. Supporting the feasibility of making carbon fiber reinforced plastic (CFRP) parts through FDM process Matsuzaki [8] successfully made high strength carbon fiber reinforced with infill speed of 25 mm/s, nozzle temperature of 220 °C and layer thickness of 0.15 mm.

4. Experimental work

In this section experimental work of fabrication of composite material filaments using the laboratory level filament extruder shown in Fig. 1 for subsequent use on a commercial FDM machine is explained first. Then the combinations of composites investigated along with material properties of their constituents and their processing are presented. This is followed by the presentation of a new approach of direct deposition to produce ASTM-D638 composite specimens through FDM is discussed. Tensile loading performance of both kinds of specimens was investigated. Later, the outcomes of this work in the form of statistical inferences, achieved strength of composites, an alternative to composite filament based 3D printing, etc. are outlined in results and discussion section. And at the end, the conclusions drawn from the results obtained are furnished. This paper combines the use of natural SFRPs and direct deposition technique to make better quality 3D printed products.

Composite material parts may be manufactured through FDM in many possible ways, each method still being at the research level and no method having clearly proven better than the others yet. In the present work, two methods have been investigated. The first method is to manufacture extruded filaments of the intended composite material and then use it in a commercial FDM machine to obtain the parts for testing. The second method, christened here as 'direct deposition method', is to directly sprinkle chopped fibers on the partially completed specimen at calculated intervals of layers completed, by pausing the FDM process at each of those intervals. In addition to investigating these two methods to make ASTM-D638 specimens and testing them, the composite material filaments themselves were also directly tested on universal testing machine (UTM) for determining strength properties. These experiments are explained in the following sub-sections.

4.1. Composite material tensile testing specimens made using filament manufacturing

Fig. 3 shows the pellets of poly-lactic acid (PLA) matrix material of average diagonal length of 3 mm and Fig. 4 shows the chopped



Fig. 3. Pellets of PLA.



Fig. 4. Chopped carbon fibers.

carbon fibers conforming to the critical length criterion of equation (1). These pellets and fiber shown below are mixed well in different proportions as per the experimental strategy in a container.

The temperature of the extruder is set approximately 250% of the glass transition temperature of the polymer matrix even though theoretically only the glass transition temperature needs to be crossed throughout the mass of each pellet to get extruded. The higher temperature accounts for the heat losses from the filament despite the thermal sealing and the need to ensure that the temperature everywhere in the mass of each bead reaches at least the glass transition temperature. The extruder used in the present work is a single screw extruder. Thermal analysis of FDM extrusion head has been investigated in the literature extensively (for example, [11]) but no rigorous analysis of filament extruders is reported in the literature, though it is very important.

The pellets of PLA of size 3-4 mm were mixed with chopped fibers of different materials and fed into the hopper of the filament extruder and extruded at 140 °C to obtain consistent filament of diameter 2.85 mm. For making composite filaments, artificial and natural fibers were used to study the variation in results among them. Among many artificial fibers available commercially, carbon fiber (CF) was chosen for this experimentation. Coconut or coir fiber (CoF) and jute fiber (JF) were selected among the natural fibers because they already have an established supply chain down to village markets to support their extensive use in many age-old applications such as rope and basket manufacturing. The mechanical properties of these materials obtained from literature [2,10] are given in Table 1. After many preliminary experiments with different weight proportions of fibers in matrix, the proportions of the matrix (PLA) and reinforcements (carbon/coconut/jute fibers) were decided as 9:1 by weight %. Thus three different composites viz. PLA(90%) + CoF(10%), PLA(90%) + JF(10%), PLA(90%) + CF(10%) were experimented. For this, the continuous fibers were cut manually to a fiber length of 3-4 mm based on the interpretations and calculations discussed in the previous section. The feedstock of manually mixed PLA and chopped fibers prepared as per weight proportions was then fed into the extrusion machine and several trials were done before arriving at the temperature of the nozzle to be fixed for each composite combination to achieve good quality composite filament.

The three types of composite material filaments explained in the previous sub-section were used for additive manufacturing of dumb-bell shaped ASTM-D638-02A Type-I standard composite material test specimens using CreatBot commercial FDM machine. The machine is shown in Fig. 5 and the test specimen dimensions are shown in Fig. 6.

Previous to additive manufacturing, the specimen standard geometries as shown in Fig. 6 were prepared in a CAD system and converted to STL file format before importing the same into the FDM machine software for mathematical slicing and part programming along with other setup parameters. These AM parameters included layer thickness of 0.2 mm, orientation along x-direction of the machine flat on the FDM machine table and 1 mm nozzle. Fig. 7 shows the tensile test specimens manufactured.

4.2. Composite material tensile testing specimens using 'direct deposition method'

In this method, an alternative way of direct sprinkling or depositing fibers during FDM building of the plain polymer was experimented. The same proportion of chopped fibers by weight % as that used in composite filament extrusion of previous subsection was used to sprinkle them manually after deposition of every 4 layers of PLA in FDM machine. This was done by pausing the FDM machine, dispersing the fibers on the whole substrate of PLA and



Fig. 5. 3D Printer.

Table 1 Mechanical properties of materials [2,10].

Sr. No.	Name of material	Density (g/cc)	Fiber diameter (microns)	Tensile strength (MPa)	Young's Modulus (GPa)
1	PLA (pellets)	1.24	_	40-45	3.45
2	Carbon fiber	1.75	10	4000-4200	230-240
3	Coconut fiber/Coir	1.2	200-400	175-220	4-6
4	Jute fiber	1.3-1.46	100	393-800	10-30

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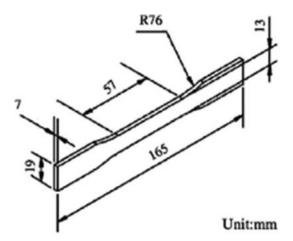


Fig. 6. 3D Printer.



Fig. 7. Composite filament based specimens (a) PLA (90%) + CoF (10%), (b) PLA (90%) + JF (10%), (c) PLA (90%) + CF (10%) and (d) Plain PLA.

then resuming the printing. Due to this, the fibers were randomly oriented and some could even go off the contour of specimens. Manual sprinkling of chopped fibers was observed to result in 5–10% of fibers not getting used up in the specimen and fall off, which was accounted for by starting with equal amount of extra fibers. The tensile testing specimens made by direct deposition method are shown in Fig. 8.



Fig. 9. Brittle Fracture of PLA(90%) + CF(10%) specimen.



Fig. 10. Samples of PLA(90%) + JF(10%) filament.

5. Tensile testing on UTM

Simple tension tests on a 100 kN computerized universal testing machine were conducted on the specimens made by the two methods explained in the previous section at room temperature.



Fig. 8. Sprinkled direct deposition method based specimens (a) PLA (90%) + CoF (10%), b) PLA (90%) + JF (10%), c) PLA (90%) + CF (10%).

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5.1. Tensile testing of ASTM-D638 specimens

Fig. 9 shows a snapshot of a specimen at fracture during the tensile testing on the UTM at the nominal speeds meant for static testing. As can be seen in this figure, the specimen undergoes brittle fracture true to the nature of the matrix material. Similarly, all other specimens were tested. The stress-strain diagrams and the various mechanical properties were recorded.

Table 2Tensile testing results of ASTM specimens.

Material	Ultimate Tensile Strength (MPa)			
composition	Direct Testing of Filaments	FDM specimens with extruded composite material filament	FDM composite material specimens with Direct Deposition Method	
PLA(90%) +CoF(10%)	43.3	43.7	47.9	
PLA(90%) +JF(10%)	45.5	45.2	44.6	
PLA(90%) +CF(10%)	55.1	51.6	51.7	
PLA	41#	40\$		

^{\$} Average value of broad range of reported values [12] as well as estimated values in the present experiments.

5.2. Tensile testing of the composite material filaments

Filaments of the composites extruded were cut to a length of 165 mm (same as the length of ASTM specimen). Four such samples (as shown in Fig. 10) of each composite and plain PLA were made ready for tensile test and the average of their UTS values was considered for reference.

The objective of testing of the filaments of the composite materials directly was to understand the mechanical behaviour of the bonding between the matrix and the fiber devoid of the lack of fusion (LOF) defects that are innate to the AM process through FDM. These test results are expected to set a benchmark for strength values of the AM made ASTM-D638 specimens.

6. Discussion of experimental results

The purpose of this research was to understand the comparative mechanical behaviour of carbon fiber reinforced and natural fiber reinforced composite material parts made by additive manufacturing namely the FDM. Two different methods were investigated for making the ASTM-D638 specimens namely, the first method involving the making of composite material extruded filaments and manufacturing the test specimens from those filaments on an FDM machine and the second method using a 'direct deposition method' wherein the chopped fiber was sprinkled on the FDM specimen in the making by pausing for every four layers. In addi-

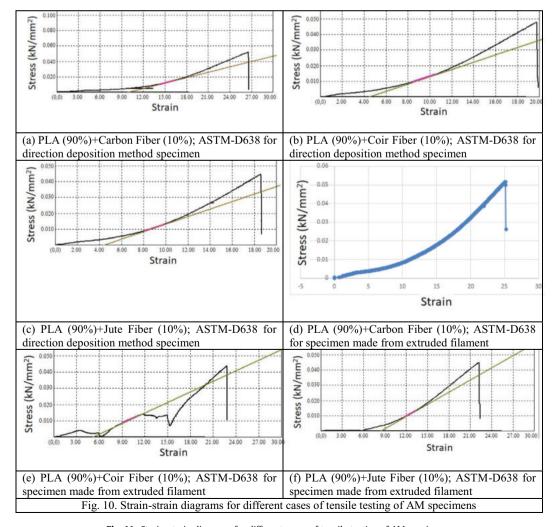


Fig. 11. Strain-strain diagrams for different cases of tensile testing of AM specimens.

[#] Average value of the tests conducted.

tion, the extruded filaments of the three composite materials and the plain PLA were also directly subjected to tensile testing. Table 2 gives the results of mechanical properties measured in all these simple tension tests.

Fig. 11 shows gives the stress –strain diagrams obtained from the UTM.

It can be observed from the results of Table 2 and Fig. 11 that the carbon fiber reinforced composite ASTM-D638 gives the highest tensile strength in comparison to the ASTM-D638 specimens of all other cases. The strength of all composite material specimens is higher than the plain PLA though the net difference is not significantly large probably due to insufficient control of many parameters of both the filament extrusion and additive manufacturing through FDM. This result is in agreement with that in the literature [2] who found a rise of 30% in ultimate tensile strength of ABScarbon fiber composite at 10% fiber loading. The natural coconut and jute fiber composites have given impressive strength results and hence appear promising for further detailed investigation. The tensile strength of the filaments themselves gave slightly higher strength as compared to their ASTM-D638 counterparts because the plain filaments possess lesser lack of fusion defects compared to FDM specimens. The 'direct deposition method' investigated in this work also gave impressive results almost as good as the extruded filament based specimens. With sufficient automation, this method may have a lot of promise for future study.

7. Finite element simulation of prosthetic socket with composite material properties

This section presents the results finite element simulation of FDM made PLA below-knee prosthetic socket and their comparison to the experimental results obtained by PSPR-3D-Tech [13] in their on-field experimental studies conducted on amputees. The PSPR-3D-Tech made their experimental below-knee prosthetic sockets through digital reverse engineering of the residual limb of the amputee, three-dimensional solid model reconstruction from the digital reverse engineering data, addition of lobes and bottom blending and then additive manufacturing through FDM. The methodology followed involved C¹ continuity blend geometries of finite length. The lobe surface and the bottom blended pyramidal extension was generated using tangent control constraint in CAD systems after replication of the customized pyramidal geometries of the pylon system of the below knee prosthesis. In the present finite element analysis, the 3-dimensional solid geometry of the socket for analysis was directly obtained from PSPR-3D-Tech.

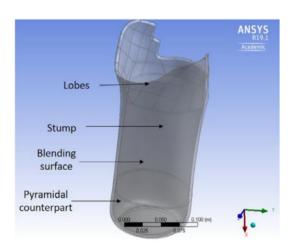


Fig. 12. Engineering of Below-knee prosthetic.

Fig. 12 shows these engineering surface reconstructions and Fig. 13 shows the completed surface model of a typical amputee's prosthetic socket. Stump length was 0.088 m at the lobe's topmost point and 0.045 m at the lobe base. The thickness of socket is a critical customized parameter deciding the weight and at the same time the strength of the prosthetic socket. The optimum socket thickness varies from patient to patient and is required to be arrived at using finite element analysis for each patient separately based on his stump geometry and body weight. Therefore, the finite element analysis conducted for one of the pilot amputee's prosthetic socket is explained here. The Fig. 14 and Fig. 15 show the boundary conditions and loading on the socket.

Static finite element analysis under these boundary conditions gave deformation and von-Mises stress results as shown in Fig. 16 and Fig. 17, respectively. A maximum deformation of approximately 2.4 mm observed in the lobes in Fig. 16. The maximum von-Mises stress was observed at the root of the lobes with a magnitude of 13.9 MPa as shown in Fig. 16. Comparing with the tensile strength of the socket material this corresponds to a factor of safety of 2.62 against static failure by fracture.

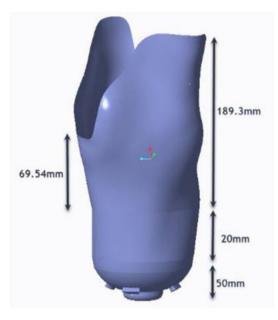


Fig. 13. Final CAD model of below-knee socket of a sample amputee.

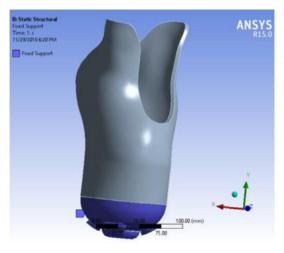


Fig. 14. Displacement boundary conditions.



Fig. 15. Load equivalent to patient's weight: 550 N load normal to the surface.

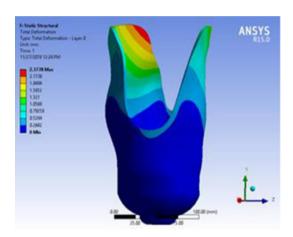


Fig. 16. Deformation distribution.

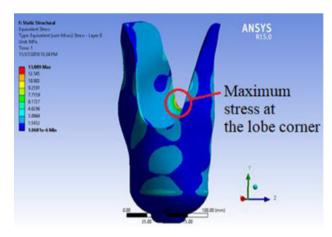


Fig. 17. Von-Mises stress distribution.

Fig. 19 shows the physical pilot prosthetic socket of the same design and material given to the amputee (Fig. 18) for wearing. The location of the fracture in the actual prosthetic socket was very closely at the same location (root of the lobe) as predicted by the finite element analysis in Fig. 17.



Fig. 18. The pilot prosthetic socket was given to an amputee.



Fracture observed in the actual prosthetic socket given to patient also at the lobe corner

Fig. 19. Location of fracture on the used socket.

8. Conclusions

Experimental research conducted on synthetic and natural fiber reinforced PLA composite material FDM specimens conforming to ASTM-D638 standard through two different methods namely, own laboratory extruded filaments and a novel 'direct deposition method' showed higher tensile strength, though not significantly, indicating viability of producing composite material parts through FDM. The favourable agreement of the strength values of these

specimens with strength results of direct tensile testing on corresponding composite material filaments supports this validity, despite the fact that the FDM specimens may potentially possess the lack of fusion (LOF) defects. Finite element static loading simulation on below-knee prosthetic sockets of these composite materials manufactured by a novel combination of reverse engineering and additive manufacturing successfully predicted the location of the fracture in the actual physical socket given to amputees. Since the actual use of the prosthesis and corresponding accumulation of damage leading to fracture occurs due to fatigue loading during the use by the amputee, the static factor of safety of von-Mises stress equal to about 2.6 is low proving that the 4 mm of thickness of socket used is insufficient. Further investigations are necessary with fatigue loading to predict the correct thickness.

- (1) Srinivasa Prakash Regalla = Corresponding author; principal researcher; subject matter expert; team co-lead; ideation and methodology; paper articulation.
- (2) Sagar S. Karwa = graduate student; experimental work.
- (3) Sreeram Rajesh thesis student; simulation work.
- (4) P. V. Shyam = research manager; experimental work; CAD modelling work; interaction with patients; integration of experimental and simulation work.
- (5) Prakash N. Shrivastava = senior member of the research team; ideation; delineation of experimental work.

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.

Acknowledgment

The experimental work reported in this paper was carried out with the help of the BIG-5 research financial grant of the BIRAC, Department of Biotechnology, Government of India.

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