Comparative study of the influences of international standards on structural performance and fracture behaviour of 3D printed long fiber composite structures

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Abstract. The material properties of the 3D printed composite structures are commonly done following international standards, which show some dimensional differences. This study focuses on the influences of the standards applied for sample preparation on the tensile properties of the 3D printed continuous carbon fiber reinforced composite parts. The specimens were fabricated by Markforged® Mark two 3D printing machine using carbon fiber as reinforcement and Onyx® as matrix material based on ASTM D638 and ASTM D3039-D3039M standards. The experimental results revealed that the specimens fabricated based on ASTM D638 showed a premature failure at the location where the straight gauge section of the specimen ends, and the curved transition regions begin due to stress concentration. The tests based on ASTM D3039-3039M standard showed better tensile strength and less stress concentration compared to ASTM D638. Fracture analysis with SEM reveals fiber breakage, debonding, and fiber pullout, which created cavities and voids between layers as the reasons for the tensile failure.

1. Introduction

Recent advancements in additive manufacturing (AM) have made popular manufacturing methods for, their low material waste, ease of manufacture, advantage of design freedom, and environmental friendliness. AM is defined by ASTM F2792−12a as the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing fabrication methodologies [1,2]. According to ISO 17296-2 standard AM is divided into seven groups based on fundamental parts of machines' functionalities; vat photopolymerization, material jetting, binder jetting, powder bed fusion, Material extrusion, Directed energy deposition, and Sheet lamination. Furthermore, AM technique can be categorized based on the types of materials used and the deposition technique [3].

Additively manufactured parts have weaker out-of-plane material properties than in-plane material properties, as a result, AM parts are mostly used as a prototype than functional parts [4]. In the aircraft industry, the application of additive-manufactured parts especially composite parts is increasing because of low maintenance costs, weight, and better reliability of the parts printed from additive-manufactured composite [5]. The development of using composite fibers in the production of polymer parts using Fused Deposition Modeling (FDM) increases the mechanical properties and thereby, functionality of the parts [6]. Therefore, to have high-performance additive manufactured polymers product with high Young's modulus and strength, it is important to reinforce with high-performance reinforcement composite polymers [7]. Reinforcement of 3D-printed polymers can be done Using both short and continuous fiber reinforcement. The short fiber-reinforced 3D printed parts have poor mechanical properties compared to continuous reinforced parts. The development of long fiber reinforcement 3D printed composites like, Markforged Mark Two changed 3D printed composite parts into a commercial product [8].

Though the start of AM technology lasted several decades, there is still a lack of understanding of the fundamental character of parts produced by AM technology. The study on the improvement of the mechanical properties continued to be the core of research on AM composite parts. In tensile testing of AM composite parts, the cause of fiber fracture that occurs is induced by stress concentration due to the geometry of the specimen. Fiber fracture that occurs at an angle is coordinated fracture and propagates the matrix cracks from the fiber break points. The stress concentration causes the failure of many fibers at similar locations [9]. Another study done by Garrell M G et al. on stress concentration in tensile specimens produced based on ASTM D638 standard using finite element analysis concluded that the specimen failed at the location where the straight gauge section of the specimen ends and the curved regions begin, this is due to high-stress concentration [10].

Mechanical and structural efficiency of 3D printed continuous fiber reinforced composite parts are highly influenced by microstructural features, including micro-void content, fiber geometry, and coherence of fiber/matrix interface. Crack tip strain was evaluated by digital image correlation technique revealing that the cross-layer fracture resulted ahead of the crack tip compared to the inter-layer fracture, fiber pull-out failure at the micro-scale, and adhesion due to short fiber-bridging. Different studies have been conducted to understand the mechanical properties of continuous fiber-reinforced polymers. The fracture behavior of 3D printed continuous fiber reinforced polymer composites and stress concentration due to the geometry of the specimen have not been well understood either experimentally or numerically. To design and produce 3D-printed CFRP composites it is important to understand the fracture mechanics of 3D-printed CFRP composites [11].

ASTM D638-14 and ASTM D3039-3039M are standard test methods used for the determination of the tensile properties of plastics and polymer matrix composite materials [12]. Originally, ASTM D638-14 was developed for conventionally manufactured plastics, typically demonstrating isotropic properties. In contrast, 3D-printed reinforced plastic parts have shown anisotropic properties or transverse isotropic properties. Because of the anisotropic nature of the 3D material and tensile test specimen geometry, crack initiation at fillets results in premature failure. So, it is important to choose a different standard to reduce stress concentration [13]. ASTM D 3039/D 3039M is the standard method used with continuous or short fiber, to determine in-plane tensile properties of polymer matrix composite materials reinforced by high-modulus fiber. The standard method used straight tab and has less stress concentration compared to ASTM D638-14 standard [14].

The Markforged Mark Two 3D printing machine uses a dual nozzle system. The machine can print different materials like carbon fiber, Kevlar, glass fiber as reinforcement, and onyx and nylon as matrix material. Markforged Mark two 3D printing works based on material extrusion AM technology, where a filament of polymer is melted and extruded through a nozzle onto a planar platform, with the addition of multiple layers forming the component [8]. However, since the machine is a newly developed technology (Markforged Inc., USA), its products are not well studied [13].

The composites nature and process variation make 3D-printed composite parts not easy to predict mechanical and microstructural properties, because of the composite nature and process variation. In view of these factors, further study is needed to understand and predict the performance of continuous fiber-reinforced 3D-printed composite parts [15]. The objective of this work is thus to study the influence of international standards on the tensile properties of additively manufactured carbon fiber-reinforced polymer.

2. Experimental methods

2.1. The material

Continuous carbon fiber and Onyx® materials supplied by PLM Group Norway AS were used as raw materials for the current study. Continuous carbon fiber has high strength and stiffness when printed with Onyx and it can easily be printed in different shapes [16,17]. Onyx is a combination of nylon-based thermoplastic and chopped carbon fiber made into filament. It was used as a matrix. The carbon as shown in Figure 1, is made from many micro-size continuous carbon filaments bundled together by sizing agent to form carbon filament as a reinforcement material. [18]. Both carbon and onyx are supplied in filament form and have densities of 1.2 gm/cm^3 and 1.4 gm/cm^3 respectively and have thickness of 1.75 mm onyx and 0.35 mm of carbon fiber in diameter.

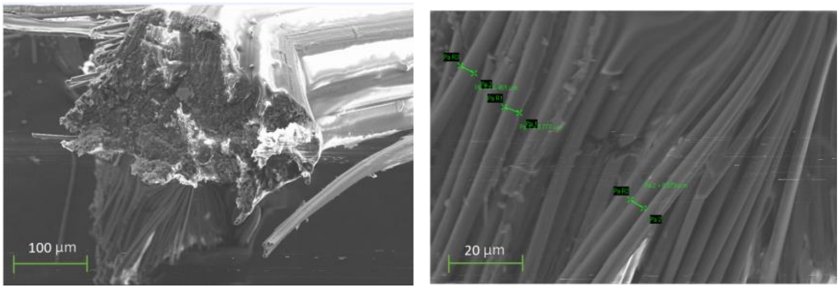


Figure 1. SEM image of carbon fiber filament.

2.2. Model preparation and printing

The geometry of the tensile specimen was designed and converted to Stereolithography (STL) using Autodesk inventor 2023. The STL file is imported into the Markforged Eiger software for slicing, adjustment of printing parameters, material selection, etc. [19,20]. The specimens were manufactured by Markforged Mark Two desktop type 3D printing machine. The machine has a printing volume of 0.584 m × 0.330 m × 0.355 m and two nozzles, one for material and the other for fiber feeding having temperatures of 252 $°C$ and 277 $°C$ for carbon and Onyx respectively to heat the filament as shown on Table 1. As Onyx filament passes through the nozzle during printing it melts, and the carbon fiber heated to provide adhesion to the matrix.

The printing parameters applied are shown in Table 1 for both matrix and fiber. Test specimens for tensile test were printed with orientations of 0° based on ASTM D638 and D3039-3039M standard method. For ease of reading, we refer ASTMD638-14 as S1 and ASMT D3039-3039M as S2, hereafter. Schematics that illustrate the geometry of the two standards are shown in Figure 2. The geometry of the test specimen manufactured by S1 is rectangular while that of S2 is closer to the dog-bone type. The dimensions of the test specimens were 160 mm x 13 mm x 3.2 mm. To reduce experimental error, five samples were fabricated by employing the same parameters for each standard as given in Table 1.

Table 1. Printing parameter used to create samples.

Parameters	Carbon fiber	Onyx
Filament orientation	0°	±45°
Layer thickness (mm)	0.125	0.125
Number of layers	12	14
Infill density (%)	100	100
Roof and floor		4
Number of walls	2	
Fiber nozzle temp ($°C$)	252	
Plastic nozzle temp ($°C$)	277	

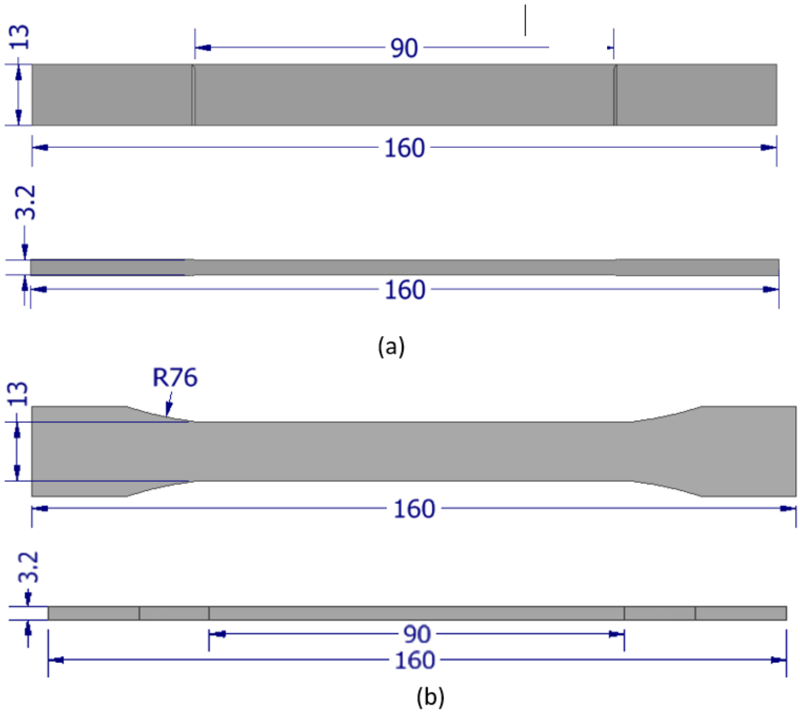


Figure 2. Tensile specimens manufactured by (a) S2 and (b) S1 standards.

Five samples were printed for each standard group (S1, S2) with the same amounts of carbon fiber onyx ratio, and stacking sequences as listed in Table 1. They were printed in unidirectional (0°) orientation of carbon fibers for both standard groups as shown in Figure 3.

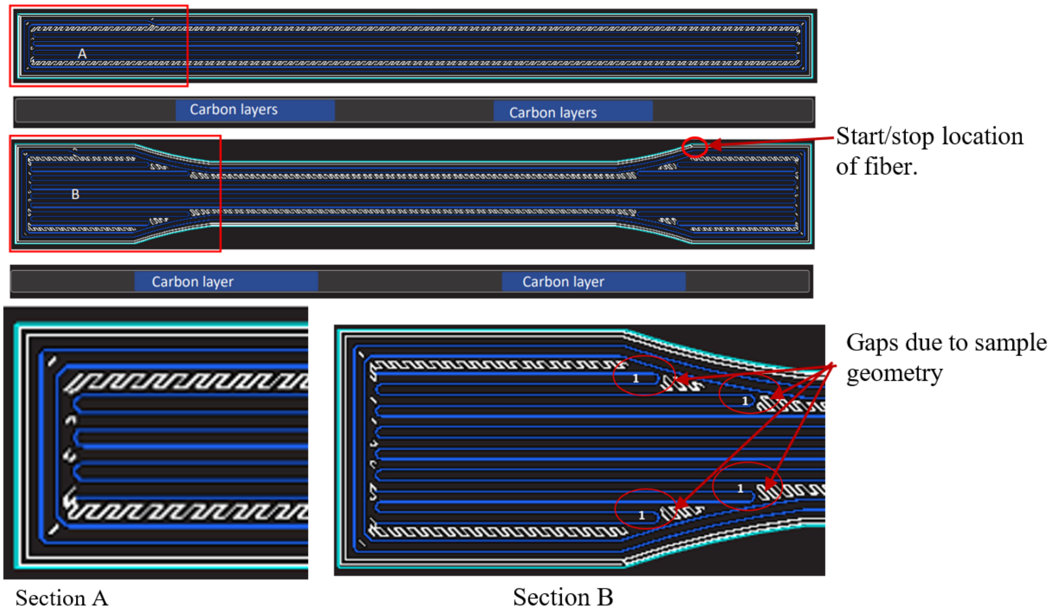


Figure 3. Fiber arrangement of S2 (A) and S1 (B) Specimen.

2.3Tensile test
The tensile test was done using an Instron 5985 universal testing machine which has a maximum loading capacity of 250 kN. The specimens were at a uniform strain rate of 1mm/min at room temperature. The schematic diagram of the specimens is shown in Figure 2.

2.4. Fracture properties
The fracture surface morphology of the tensile specimen was analyzed using scanning electron microscopy (SEM) (Gemini SUPRA 35VP ZEISS) equipped with EDAX energy dispersive X-ray spectroscopy (EDS).

3. Results and discussions

3.1. Tensile test
The effect of fiber stress concentration due to standard method selection on mechanical properties was studied under tensile load. All tensile tests were performed until complete specimen failure. The tensile properties of the tested specimens are shown in Table 3 and the stress versus strain curve was also shown in Figure 5 (a) and (b).

Table 3. Tensile test result.

Standard type	Load (kN)	Strain (%)	Stress (MPa)	Modulus (MPa)
S1	10,51	4,79	245,51	6591,81
S1	10,61	4,26	247,96	7451,93
S1	11,8	4,76	275,75	7726,67
S1	10,17	4,12	237,61	7617,56
S1	11,24	4,55	262,67	7111,25
S1	11,5	4,7	268,75	7472,61
Average. *	*10,97*	*4,53*	*256,38*	*7328,64*
StDev. *	*0,64*	*0,28*	*14,89*	*416,66*
S2	12,06	5,58	281,7	8926,95
S2	12,04	5,87	281,22	8674,66
S2	13,93	4,52	325,49	8937,28
S2	12,2	4,82	285	9729,34
S2	13,09	5,22	305,78	9752,32
Average *	*12,66*	*5,20*	*295,84*	*9204,11*
StDev. *	*0,830*	*0,548*	*19,424*	*501,182*

The test results show brittle behavior as can be observed from Figure 5 (a) and (b). This shows that the specimens were influenced by the carbon fiber properties.

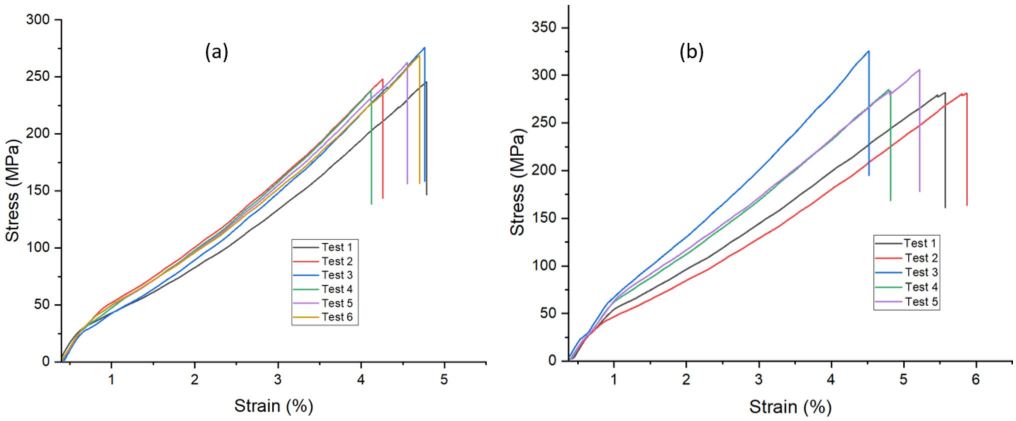


Figure 4. Tensile test stress vs. strain (a) S1 method, (b) S2 method

Both samples were printed with the same orientation, layer stacking, infill density, and percentage of carbon fiber filaments versus Onyx but with different standard methods as shown in Table 1. The samples tasted according to S2 Standard showed an average tensile strength of 295.84 MPa ±19 and elongation of 5.20 % ± 0.55, whereas the specimens produced according to S1 showed an average tensile strength of 256.38 MPa ± 14 and elongation of 4.28 % ± 0.28. The samples manufactured based on S2 standard have better strength and ductility compared to the samples produced based on the S1 standard. Using S1 standard for continuous reinforced composite 3D printed polymer has lower tensile strength and elongation values by 7.1% and 10.64% respectively, than that of S2. The S1 test result showed less deviation compared to S2.

S1 standard is a test method used to determine the tensile properties of plastic and composite parts that have isotropic properties. However, 3D-printed reinforced composite polymers have orthotropic/ transversely isotropic properties. Therefore, it is not easy to determine the properties of additively produced composite parts [13]. The tensile test result from the standard shows lower tensile strength, strain to failure, and modulus of elasticity.

As shown in Figure 3 section B the fiber arrangement shows irregularity and gaps at the transition area from gauge to tab regions which were numbered as 1 with a red circle, those gaps between adjacent layers increase the probability of failure due to the stress concentration. The start/end of fiber shown at the start of the tab on the same part B also contributed to the premature failure of the samples manufactured based on S1 standard.

3.2. Fracture properties

Tensile tests after the failure of S1 and S2 were shown in Figure 4. In cases of the specimens fabricated based on S1, the material fractures occurred outside the gauge section. On the other hand, the tensile fracture specimens prepared based on the S2 standard method occurred in the gauge section of the samples as shown in Figure 4.

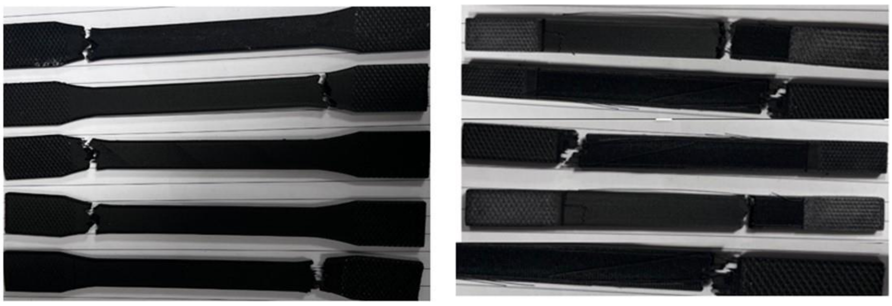
(a)  (b)

Figure 5. Fracture profile of materials failure during tensile test (a) S1, (b) S2

The fracture analysis of the tensile specimens was carried out after the tensile test to understand the fracture behavior connected with damage to continuous fiber-reinforced 3D parts. It provides information which affects strength and mechanical performance of 3D printed continuous carbon fiber reinforced parts. Figure 6. Shows cross-sectional SEM images of the examined tensile fracture parts at different magnification. The fracture surface morphology Figure 6 (a & b) reveals matrix-dominated areas with voids and weak inter-bead adhesion as well as fiber debonding from matrix areas by poor bonding interfaces. In Figure 6 (c.) pulled-out carbon fibers can be seen which create voids in the matrix. While Figure 6 (d) shows inter bead voids induced by a pattern of manufacturing. Figure 6 (e & f) also shows weak bonding between the onyx matrix and carbon fiber as well as pull out of carbon filament from the matrix. While in Figure (g) There are matrix-dominated areas, with voids, dry fibers, and fibers deboned from the surrounding matrix generated by a poor bonding interface, with damaged carbon bundles and creates a cavity in the matrix parts [18].

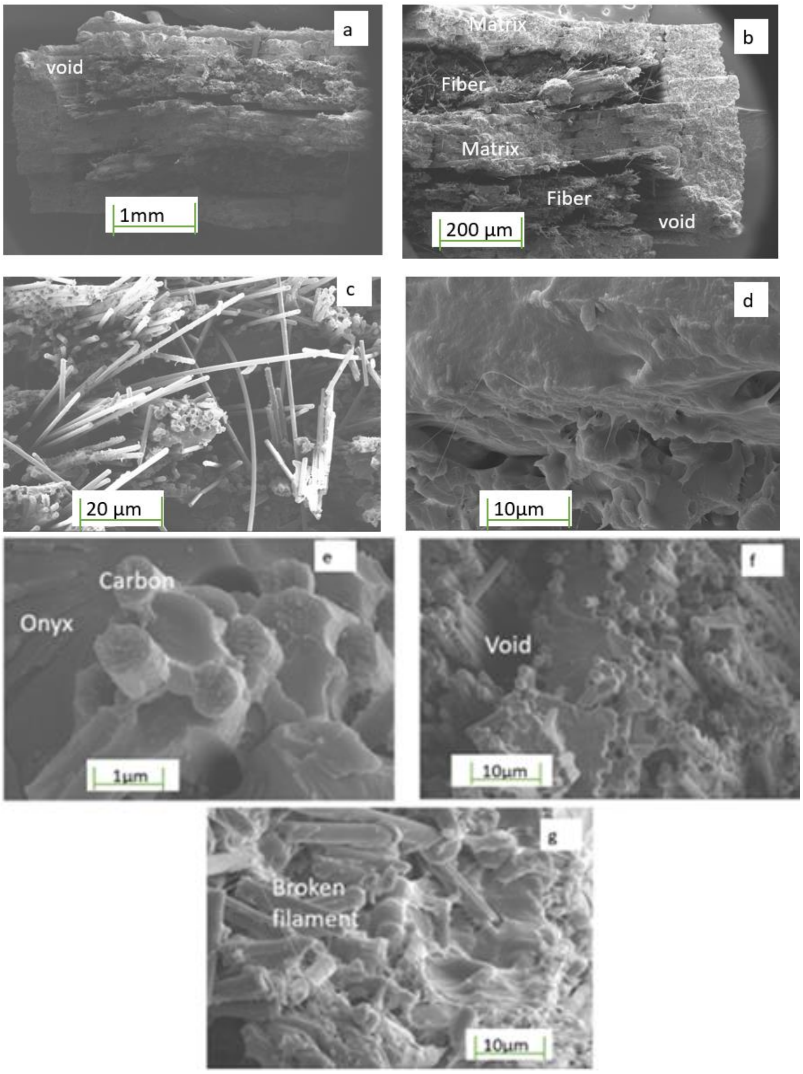


Figure 6. SEM images of the fracture surface

The mechanical performance of 3D printed continuous fiber reinforced composite fiber is affected by manufacturing induced defects such as inter layer and inter bead voids, lack of fusion between matrix and reinforcement. As shown in Figure 3 section B the geometry of samples has high contribution to the failure of 3D produced parts.

In Figure 7 the horizontal axis of the EDS spectrum is the energy in keV, and the vertical axis is the number of X-ray counts or intensity.: The "K" in the vertical axis implies that the values in the vertical axis are multiplied by 1000. The source of Au/Gold is from coating of carbon to make it conductive. Therefore, it was not considered in specifying element compositions.

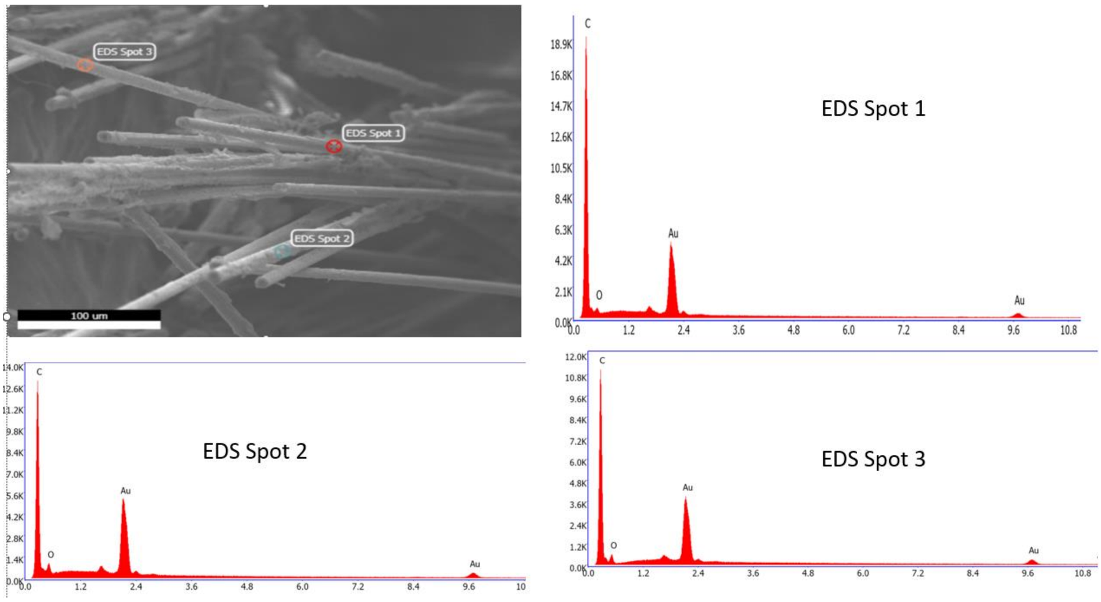


Figure 7. EDS elemental composition analysis of different carbon fiber filaments.

Table 6 shows the composition of carbon and oxygen molecules. The amount of carbon and oxygen in different filaments is different.

Table 6. The concentration of C and oxygen at different spots.

Spot	1	2	3
	Wt. %	Wt. %	Wt. %
C k	98.81	90.40	97.41
O k	1.19	9.60	2.59

4. Conclusion

In this study, the influence of international standards on tensile properties and the fracture surface morphology of continuous carbon fiber reinforced polymer composite have been studied. The 3D printed composite utilized continuous carbon fiber as reinforced and Onyx as matrix materials.

In tensile testing of 3D printed parts, both ASTM D638 and ASTM D3039-3o39M were used to determine the tensile properties of plastics and polymer matrix composite materials. The test done based on the ASTM D638 standard shows lower results due to stress concentration induced by the specimen geometry. The fillet region of the specimens induces crack initiation that causes premature frailer.

The specimens produced based on the ASTM D3039-3039M standard have better tensile performance than the ASTM D638 standard. It has a straight tab and less stress concentration. Therefore, in tensile testing of continuous carbon fiber reinforced 3D printed polymer, ASTM D3039-3039M has better performance.

A study of the fracture surface morphology of the printed composite shows a lack of fusion of carbon fiber and onyx and individual fiber bundles together, so the synergetic effect of the carbon fiber bundle is lacking. This can be improved by increasing printing temperature that enhances adhesion, denser composite, and minimizing voids.

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