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Original Paper

Examination and improvement of direction-dependent surface properties in composite structures produced by the fused deposition modeling method

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ABSTRACT

Additive manufacturing methods, which have begun to be used in final product production beyond producing prototypes, are popular research topics today. The Fused Deposition Modeling (FDM) method, which has a wider usage area and user base, is the most well-known among these methods. The method in which the polymer is extruded in layers has advantages such as design freedom and topology optimization, as well as disadvantages such as surface roughness and low production speed. The number of materials that can be used in the FDM method is diversifying day by day, and polymeric composites can also be produced beyond pure polymers. Using engineering polymers such as polyamide in this production method creates many new opportunities. In addition, the start of production of short fiber reinforced polymeric composites has paved the way to produce high-performance final products. In this study, the effects of parameters such as layer thickness and printing orientation on the surface roughness of samples produced using polyamide and short carbon fiber reinforced polymer matrix composite materials were examined. Chemical surface treatment was applied to the surfaces of 3D-printed samples to improve surface roughness. It was concluded that the increase in layer thickness increases the surface roughness, the -/+45 filling orientation creates higher roughness than the 0 and 90 orientations, and the surface quality can be increased by chemical surface modification.

I. INTRODUCTION

The Fused Deposition Modeling (FDM) method is the additive manufacturing method with the most users in the world [1]. The method, which produces using polymers as raw materials, carries out material extrusion-based production [2]. With the developing material science, it has become possible to go beyond pure polymer production in the FDM method and produce composite structures and even metal products. The mechanical properties of the materials produced by the FDM method vary depending on the production direction, and anisotropic material is produced with this method [3,4]. In addition to the material used, parameters such as fill rate, infill pattern, print speed, layer height, etc. have a great impact on the mechanical properties of the final product produced in the FDM method [5–7]. One of the most important disadvantages of parts produced by additive manufacturing is that the surface quality of the part is not good after production. This requires post-processing in many applications. Many post-processing processes, which can be categorized as mechanical or chemical, have been researched and applied to improve the surface quality of parts produced with FDM [8,9].

Various surface treatments such as chemical treatment, painting, or coating have been tried in the literature to improve surface roughness. Acetone solvents are the most used chemical solvents [10]. Jo et al. and Pestano et al. examined the surface properties of acrylonitrile butadiene styrene (ABS) materials produced by the FDM method post-processed with acetone chemical. It has been determined that surface roughness is reduced by 90% and helps

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to improve the surface quality of ABS-printed parts [11,12]. Rajan et al. examined the effect of Tetra Hydro Fluoride (HF) solution on the surface properties of polylactic acid (PLA) samples produced by the FDM method. It has been observed that the applied coating has a reducing effect on surface roughness [13].

When the mechanical finishing methods for surface quality are examined, it is seen that machining, abrasive, heat cutting, barrel finishing or sanding processes are performed. Boschetto et al. improved the surface quality of parts produced by the FDM method by using computer numerical control (CNC) milling machines [14]. In another study, Moradi et al. examined the surface quality of low-power CO_2 laser finishing applied at different parameters on PLA samples produced by the FDM method. In the study, the parts were cut with a laser to bring them to the final dimensions, and high-quality surfaces were obtained [15]. Additive manufacturing, by its nature, aims to produce final products in one piece, no matter how complex their geometry, without the need for assembly. Mechanically performed post-processing processes have long processing times and create disadvantages in terms of measurement tolerances. Studies carried out in the literature have shown that chemical surface treatments are carried out with general polymer materials such as ABS and PLA, which are the most used in the FDM method. The production of engineering polymers with higher mechanical properties, and even fiber-reinforced polymer matrix composites, with the FDM method is the topic of today's experimental studies[16].

Polyamide6 (PA6) was determined as the matrix material because it is resistant to chemicals and has high mechanical properties. Polyamides are crystalline polymers typically produced by condensation of a diacid and a diamine[17]. Polyamide (PA) is an important thermoplastic with an amide bond (–NH–C=O) in the polymer backbone. PA as a fiber-forming material was invented by Wallace Carothers at DuPont in 1935 and patented the world's first fully synthetic fiber, nylon 6.6.[18]. Polyamide is a material that is included in the thermoplastic polymer types and is used in many engineering applications.

In this study, neat Polyamide6 (PA6) and short carbon fiber-reinforced PA6 matrix composite samples were produced using the FDM method. The effects of production parameters in the FDM method on surface quality and the effects of chemical surface treatments on surface roughness and surface contact angle were examined. The effect of layer thickness on surface roughness and the effect of infill pattern on surface quality were measured. In addition, the effects of carbon fiber reinforcement and reinforcement ratio on the surface quality and the effects of chemical surface treatments applied to all samples after production on the surface quality were investigated.

II. EXPERIMENTAL METHOD

2.1 Materials

In this study, neat PA6 (PA6) material and two different PA6 matrix composites that are reinforced with different ratios by weight of short carbon fiber were used. These composites were shaped as a filament by a twin-screw extruder and since all samples are produced by the FDM method, these materials are in filament form. The matrix material used in neat polyamide and composite structures is the same. The matrix material and neat one of the filaments used in the study is the Ultramid B40LN product of BASF company. The density of the polyamide product is 1.13g/cm^3 and the relative viscosity value is 4. Neat polyamide was used as the control group[19].

The materials used in the study include AC4102 chopped carbon fibers of Dowaksa company. Polyamide matrix filaments containing 10% (PA6CF10) and 20% (PA6CF20) by weight of short carbon fiber were used. Chopped carbon fibers have a density of 1.73g/cm^3, with a tensile strength of 4200MPa and a tensile modulus of 240GPa. The diameters of carbon fibers are 7µm and their length is 6mm[20].

In this study, formic acid was used to improve surface properties. Formic acid, CH_2O_2 (98%+ pure), was obtained from ACROS Organics and used in liquid form. Many studies have reported that formic acid has a corrosive effect on polyamide [21]. For this reason, it is preferred for surface modification.

2.2 Filament Production

All compounds with 2 different fiber ratios were produced with a twin-screw extruder. These compounds were produced by Eurotec Company with a twin-screw extruder in pellet form. An extruder with a counter-rotating screw configuration was used to obtain a homogeneous mixture and fibers and matrix were mixed in time extrusion. A twin-screw extruder unit with a diameter of 18 mm was used in the production of the compound. Pre-drying was carried out at 80°C for 24 hours, and barrel temperatures were between 190-225°C.

All filaments were produced at EG plastic company with a single-screw extruder that brand is SJ. Since the filament diameter of the device used in specimen production with FDM is 2.85mm, all products were produced in these dimensions. Barrel temperatures were between 190-225°C at the single-screw extruder, screw RPM was about 6, and puller RPM was set depending on adjusting the diameter of the filament.

2.3 Production of Test Samples

By using PA6, PA6CF10, and PA6CF20 filaments, sample production for surface contact angle and surface roughness measurement was carried out by the FDM method. As seen in Figure 1, the sample dimensions are 3mm thick and have a width of 12*120mm. The samples were drawn with the Autodesk Fusion 360 CAD program.

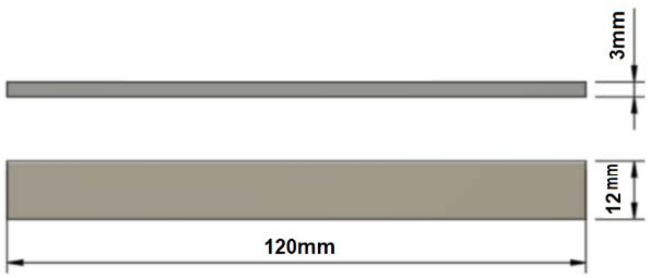

Figure 1. Samples geometry

The production of all samples was carried out at the University of Alberta Multi-Functional Laboratory with the Ultimaker S5 model device [22] and the Ultimaker printcore CC nozzle [23] was used for extrusion head. This special nozzle with a sapphire tip and 0.6mm diameter was used as samples were produced using carbon fiber-reinforced filaments. It is a nozzle specially developed by Ultimaker due to the abrasive effect of carbon fibers. The production parameters of FDM devices are adjusted with CAM programs as shown in Table 1. CAD data created in stl format were converted to gcode files using the CAM program called CURA which is version 4.0.

Table 1. 3D Printing Parameters

Parameters	Values
Print fill	100%
Nozzle Temperature	275 °C
Buildplate Temperature	80 °C
Flow Rate	100%
Print Speed	40mm/s

As a variable parameter for samples, the effect of 3 different layer thicknesses on the surface properties in the Z axis was investigated. Samples with layer thicknesses of 0.1, 0.2, and 0.3 mm were produced as shown in Figure 2. At the X-Y plane, samples were produced in 3 different infill patterns. These are 0, 90, and -/+45 degrees as shown in Figure 3.

Figure 2. Layer height

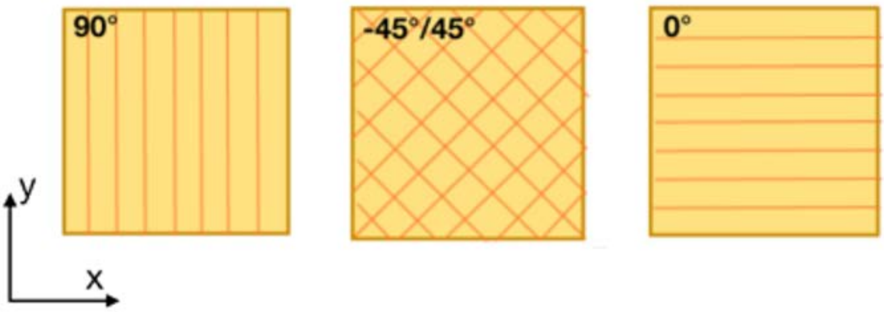

Figure 3. Infill pattern

FDM method sample production was carried out using 3 different filaments shown in Figure 4 with the dimensions shown in Figure 1. For each material type, samples were produced with the same dimensions but in two different build orientations and these are called on-edge and flat as shown in Figure 5.

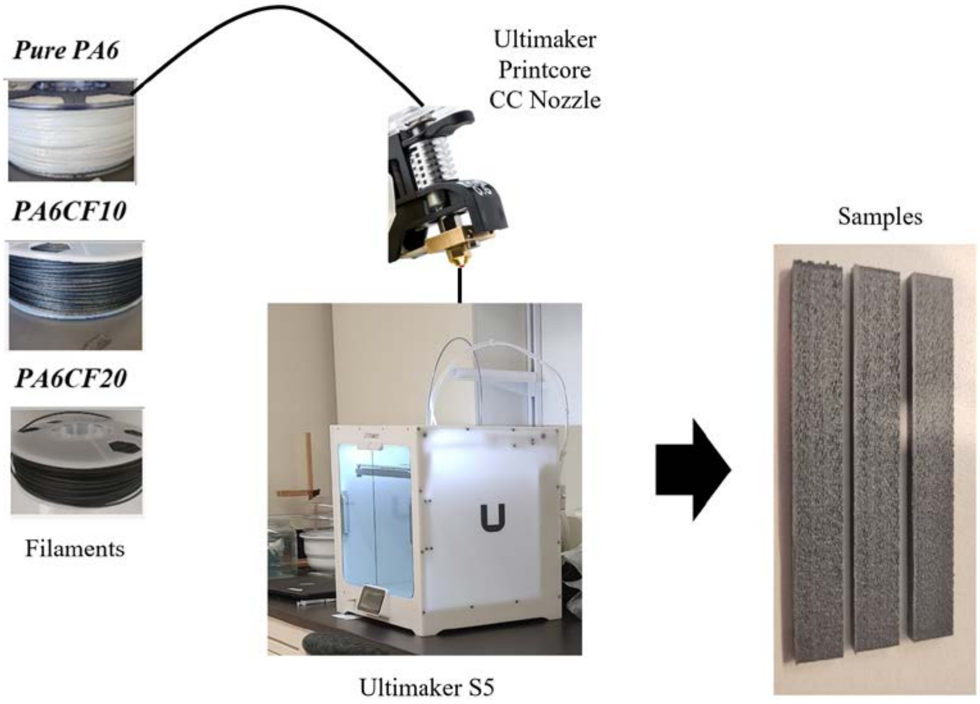

Figure 4. Samples production

The samples were produced with 3 different material groups, they have 6 different variables, and these variables are 3 different layer thicknesses and 3 different filling patterns. A total of 5 different samples were produced from each material group.

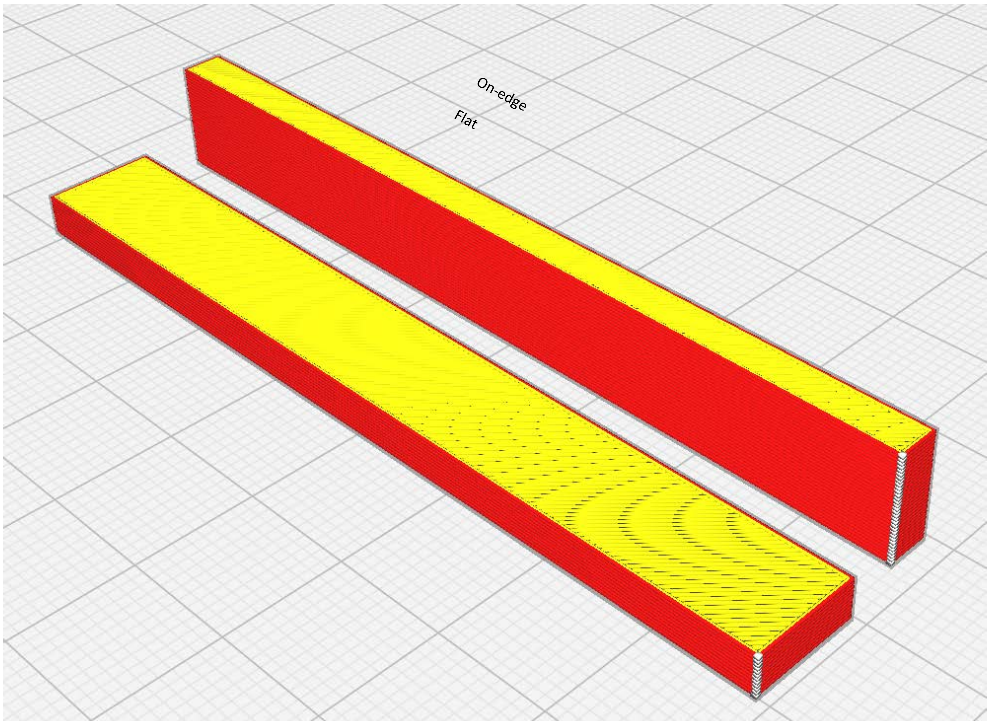

Figure 5. Production build orientations

The effect of the printing direction on the surface roughness for flat samples was examined on the X-Y plane, and for on-edge samples, the effect of layer thickness was examined on the Z-axis.

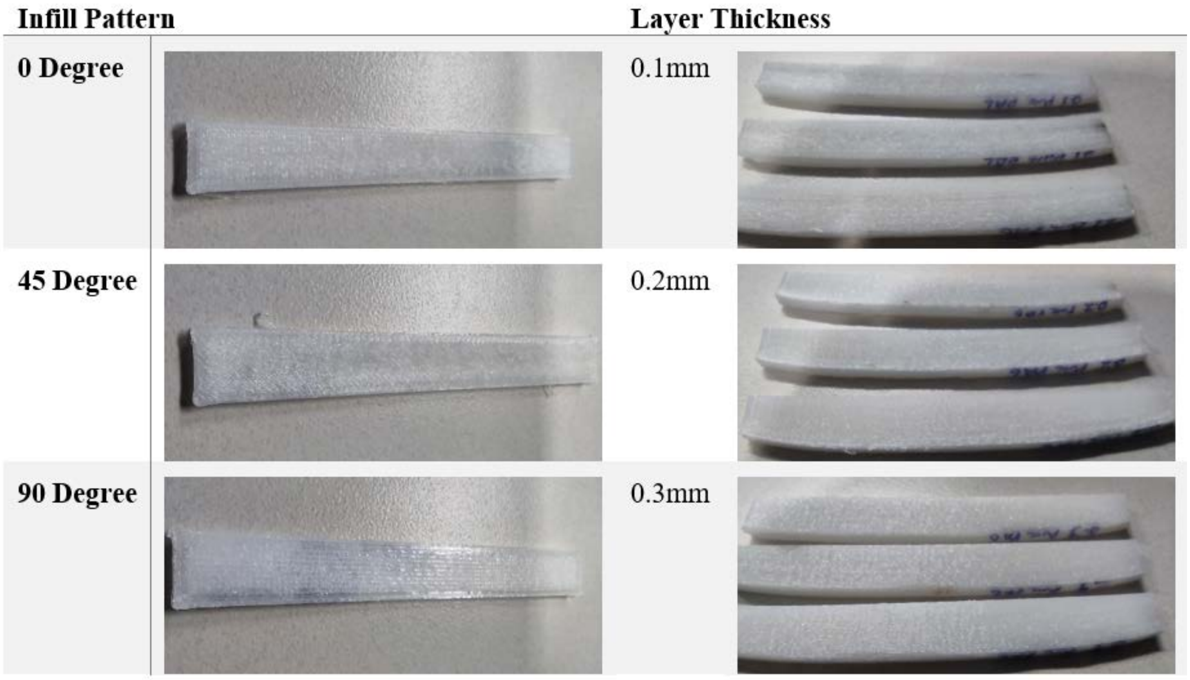

Figure 6. PA6 samples

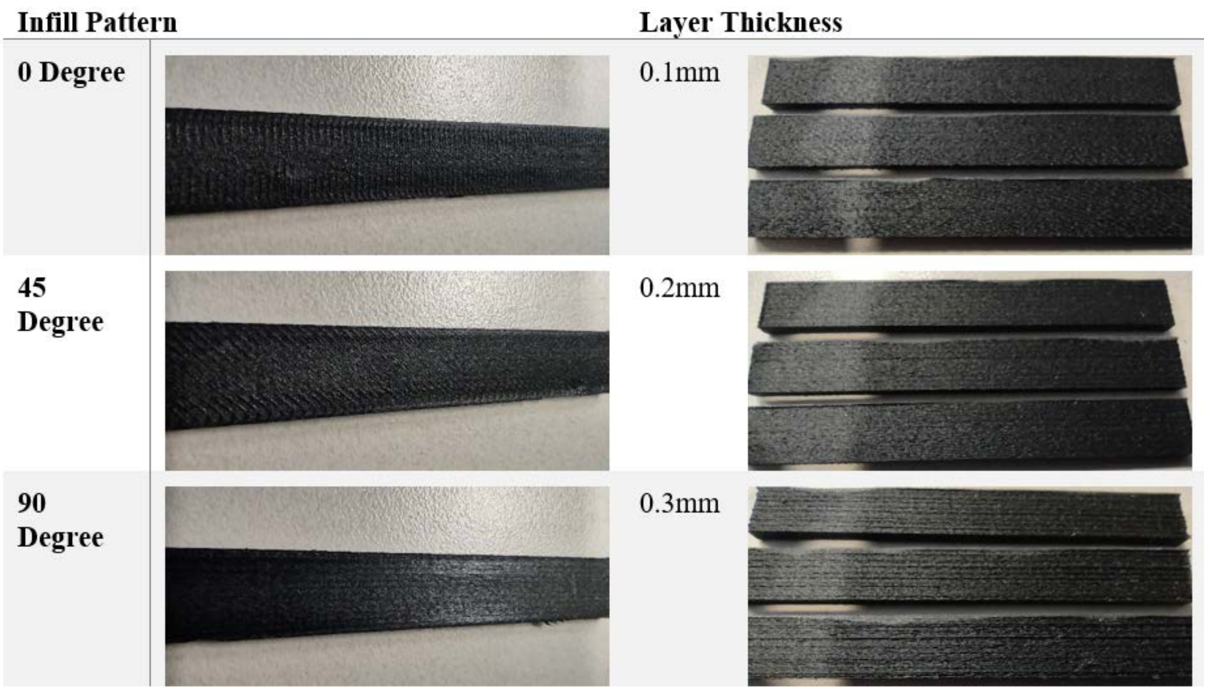

Figure 7. PA6CF10 samples

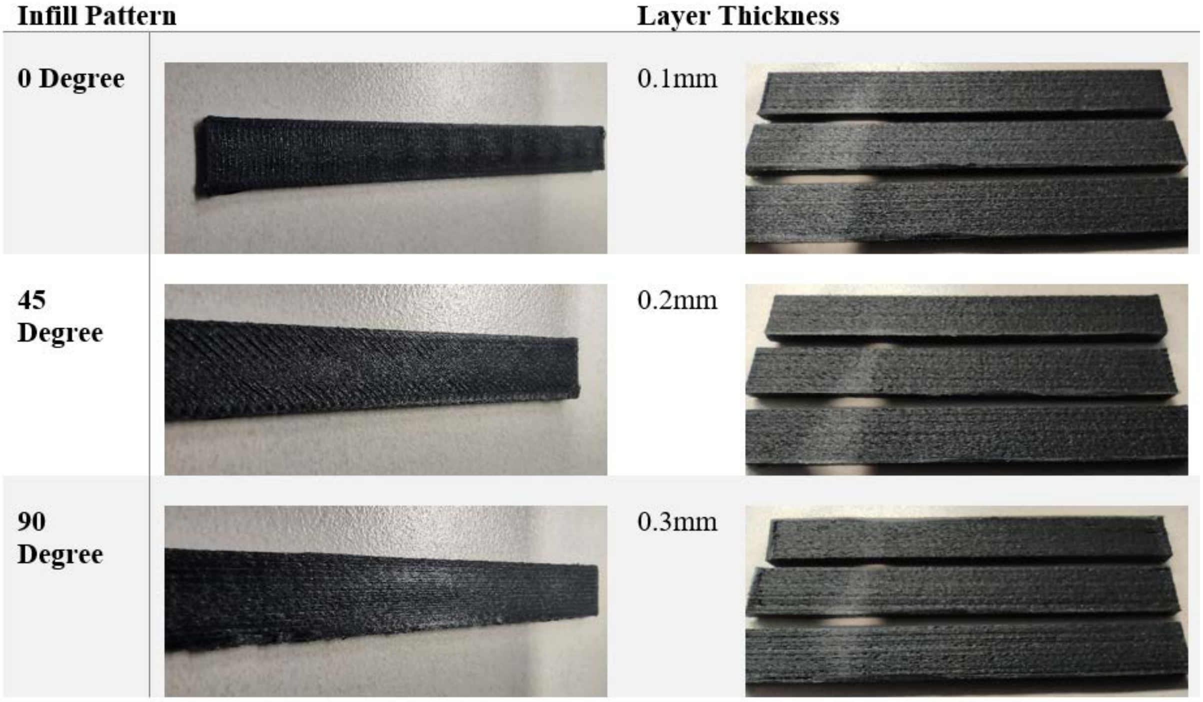

Figure 8. PA6CF20 samples

2.4 Surface Treatment

The chemical treatments applied to the produced samples aimed to improve the surface properties and formic acid was used because it can interact with polyamide with high chemical resistance. To examine the effect of the produced samples on the surface properties, all samples were kept in formic acid. All samples were placed in bottles, sealed with parafilm M PM999 tape, and heated to 68 °C. The samples were stirred in formic acid on a corning pc-420d stirring hot plate at 100 rpm for 30 minutes.

PA6 PA6CF10 PA6CF20

Figure 9. Surface modification in formic acid

To improve the surface properties of all samples produced with a 3D printer, surface treatment was applied with formic acid to examine its effect on the surface properties on both the X-Y plane and Z axis. According to the literature knowledge literature [24–27], formic acid was preferred, and these stated processing values were selected in this study because it was observed to have a corrosive effect on polyamide material.

2.5 Surface Roughness

Surface roughness measurements were made with the Mitutoyo brand SurfTest SJ-301 surface roughness measuring device located in the Mechanical Engineering Department of Ege University Faculty of Engineering. Surface roughness measurements were made on all samples with 0, 45, and 90-degree infill patterns and 3 different layer thicknesses (0.1, 0.2, 0.3 mm), without surface modification and with surface modifications applied.

Surface roughness measurements were carried out according to ISO 1997. In surface roughness measurements, Ra, which is the numerical average value of all peaks and valleys measured along the tested line, and Rz, which is the average value of the consecutive highest peaks and lowest valleys measured, were measured. For each sample, 5 measurements were taken in 5 repetitions at 0.8 cut-offs.

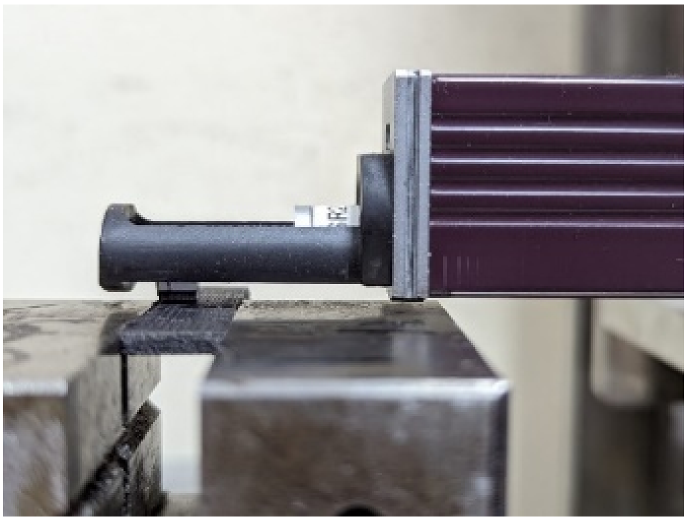

Figure 10. Surface roughness measurement

2.6. Contact Angle Measurement

The wettability changes of the surfaces of the samples produced in the study were investigated by a contact angle test. To determine the change in wettability, contact angle measurements were made on the modified and unmodified surfaces at room temperature. The samples were dried in a vacuum oven at 100°C for 12 hours before testing. Finally, the samples were kept in the desiccator for 90 minutes before contact angle measurements. Measurements were performed using the VCA Optima Contact Angle Surface Analysis System (AST Products, Inc., Billerica, MA, USA) located at the University of Alberta NINT facility. During the contact angle analysis, images were taken in dynamic mode for a 1µL water droplet on the sample surface and then analyzed with software (AST Products, Inc., Billerica, MA, USA) examining the waterdrop shape profile on the sample surface. For each sample, seven consecutive measurements were taken, and mean values were reported as representative measurements.

III. RESULTS AND DISCUSSIONS

The results of roughness values Ra, and Rz against the samples with varied layer thicknesses are tabulated below (Table 2 and Table 3). Analysis of the results: Based on the surface finish results, as shown above, it is evident that as the layer thickness increases, surface roughness values (Ra & Rz) increase. In the table, samples with surface modification applied are expressed as M XXX, and samples without surface modification are expressed as non-M XXX.

Table 2. Surface roughness of X-Y plane (µm)

		M 0 Deg	non-M 0 Deg	M 45 Deg	non-M 45 Deg	M 90 Deg	non-M 90 Deg
PA6	Ra	4.21	16.54	4.83	20.96	1.96	4.00
	Rz	28.51	74.16	42.03	91.53	10.97	19.81
PA6CF10	Ra	7.54	10.22	9.19	11.54	3.90	6.66
	Rz	47.96	58.11	60.09	78.03	22.87	37.24
PA6CF20	Ra	7.84	11.75	10.15	14.73	4.73	7.80
	Rz	48.02	73.40	71.31	103.1	28.34	40.23

Table 3. Surface roughness of Z axis (µm)

		M 0.1mm	Non-M 0.1mm	M 0.2mm	Non-M 0.2mm	M 0.3mm	Non-M 0.3mm
PA6	Ra	16.26	21.49	19.68	23.91	21.21	25.57
	Rz	75.73	122.4	98.6	114.4	94.96	111.9
PA6CF10	Ra	19.59	21.09	20.68	23.0	21.14	26.24
	Rz	110.8	100.2	98.21	122.1	111.1	137.1
PA6CF20	Ra	20.2	24.47	21.34	25.55	22.58	28.13
	Rz	96.49	129.4	114.8	135.2	109.5	145.2

The lowest surface roughness in all samples was observed in samples produced with a 90° infill pattern. Since the 90° infill pattern orientation and the movement pattern of the surface roughness measurement probe were parallel to each other, the lowest values were obtained in these parameters. Increasing the carbon fiber ratio increased the surface roughness in all samples with different infill patterns. The hard structure of carbon fiber and its increased ratio in the matrix has resulted in the formation of rougher surfaces.

The highest surface roughness values were observed in the samples produced with the 45° infill pattern. Producing layers in the -/+45° orientation resulted in rougher surfaces at the moving points that intersected in the coordinate plane. In PA6 unmodified samples, the Ra value increases 5 times when the samples produced with a 90° infill pattern are compared with the samples produced with a 45° infill pattern, while the Ra value increases approximately 4 times when compared to the samples produced with a 0° infill pattern.

A greater improvement in surface roughness was observed in PA6 samples after chemical modification. Since formic acid did not affect the fibers in carbon fiber-reinforced samples, the improvement in surface quality was not as high as PA6. The highest surface roughness values were measured in samples containing 20 percent carbon fiber by weight and produced with a 45° infill pattern. After chemical surface treatment, the surface quality has been increased by 30%. The highest surface quality improvement was observed in the samples produced with 0°

infill pattern. The reason for this is that formic acid creates more contact surfaces at the peaks and the reaction occurs in a larger area.

There was a warping problem during production in the samples produced from PA6 material in on-edge built orientation, but this was not observed in the fiber-reinforced ones. Fiber reinforcement also provides an advantage in shape stability. Since surface roughness measurements were made from the middle parts of all samples, the effect of warping on roughness was tried to be minimized.

When the effects of layer thickness on surface roughness and surface modification in this direction were examined, the highest surface roughness value was measured in PA6CF20 samples produced with 0.3mm layer thickness. Chemical surface treatments showed an improvement of 20%.

Higher surface quality was obtained on the surfaces in the X-Y plane compared to the Z axis. Although more detailed samples were produced with a layer thickness of 0.1mm in the Z axis, surface roughness values in the X-Y plane were not obtained. This is because the nozzle contributes to the smoothing of the surface while moving in the X-Y plane in sample production, while there is no support or corrective mechanism in the Z axis. Selvam et al. examined the surface roughness values at different layer thicknesses on the samples produced from ABS material by the FDM method and measured that the increase in the layer thickness increased the surface roughness [28]. There is a similar situation for PA6 material.

Even if chemical surface treatment was applied on all produced samples, the surface quality X-Y plane values could not be reached in the Z axis. Surface roughness values were measured higher in all parameters and fiber ratios in the Z axis compared to the X-Y plane.

Many surface modifications and coating technologies are applied to optimize wetting and adhesion properties. The surface contact angle is affected by both surface chemistry and roughness. For this reason, the effects of parameter variables and chemical surface modification on surface contact angle were examined within the scope of the study.

The contact angle values of the samples produced with 0° and 90° infill patterns on the X-Y plane were not performed because they would not provide an effective comparison. The contact angle values of the samples produced with only 45° infill pattern on the X-Y plane were examined.

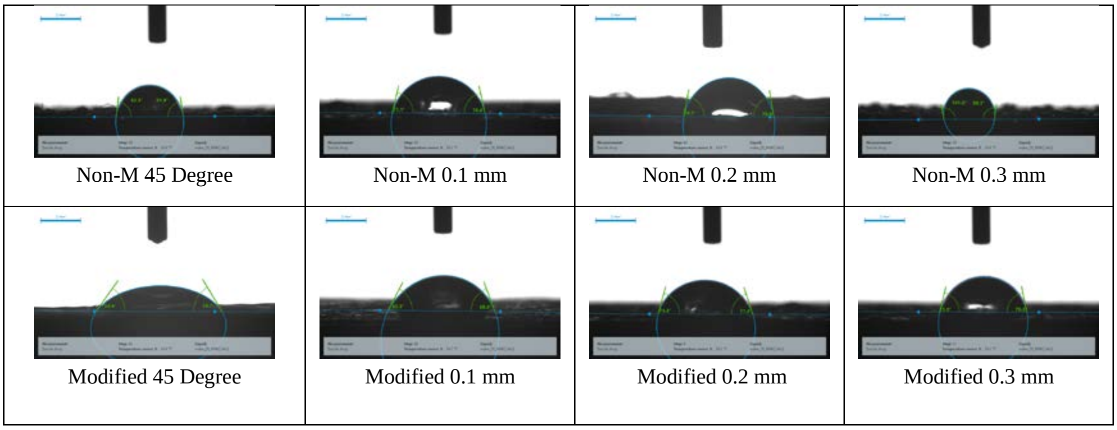

Figure 11. PA6 contact angles

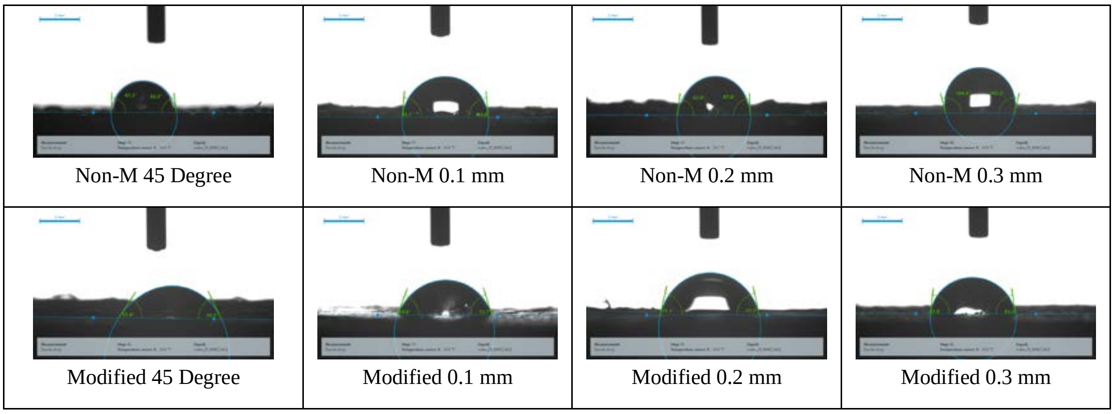

Figure 12. PA6CF10 contact angles

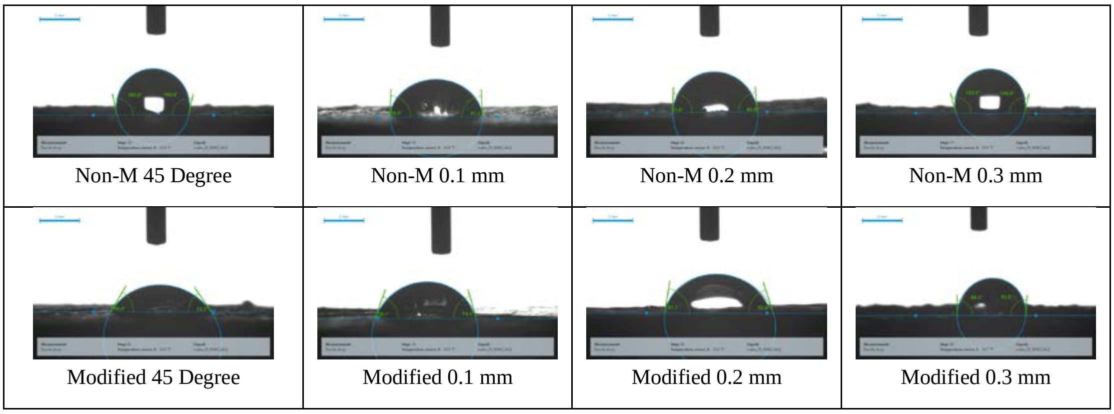

Figure 13. PA6CF20 contact angles

The lowest contact angle value was found on the X-Y plane surface produced with a 45° infill pattern. The contact angle values in the Z axes are higher. Chemical surface treatment decreased the surface roughness and contact angle values in all samples.

The contact angle values of all PA6 samples were lower than the samples containing carbon fiber. This is because polyamide is polar and hydrophilic due to the amide (-CONH) functional group it contains [29]. Carbon fiber reinforcement caused an increase in contact angle values. The increase in surface roughness because of carbon fiber is the reason for this.

As the roughness increased in all samples, the contact angle also increased. The inability of the liquid dripped during the test to penetrate the rough surfaces well and the gas molecules trapped in the roughness because this increase in the contact angle. The roughness on the surface causes discontinuities to form at the liquid-solid interface. Therefore, there is the formation of gas-liquid interfaces in addition to solid-liquid regions. This creates a barrier effect, preventing the liquid from spreading freely on the material surface and reducing wetting [30].

IV. CONCLUSIONS

In this study, the effects of different carbon ratios, production parameters, and chemical surface treatment on the surface properties of composite samples produced by additive manufacturing were investigated. The surface roughness has a linear relationship to the layer thickness. Layer height is the fundamental parameter that influences print quality as it sets the thickness of each layer being printed. The thinner the layer thickness, the surface roughness quality of the 3-D printed object will be better. As the roughness increased in all samples, also the contact angle increased. The contact angle is directly proportional to the roughness. Chemical surface treatment decreased the surface roughness and contact angle values in all samples. The contact angle values on the Z-axis are higher than on the X-Y plane. Chemical surface treatments can be used to reduce the contact angle on samples produced by additive manufacturing. The effects of different chemicals on the surface contact angle of the polyamide matrix can be investigated in future studies.

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