

Surface Roughness Performance Related to the Direction in Composite Structures Produced by the Fused Filament Fabrication Method and Improve to Surface Roughness

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in Materials Science and Engineering

by

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Declaration of Authorship

I, **Özlem DOGRU**, declare that this thesis titled **Surface Roughness Performance Related to the Direction in Composite Structures Produced by the Fused Filament Fabrication Method and Improve to Surface Roughness** and the work presented in it are my own. I confirm that:

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Surface Roughness Performance Related to the Direction in Composite Structures Produced by the Fused Filament Fabrication Method and Improve to Surface Roughness

Abstract

Additive manufacturing is used today in different industries to produce prototypes and even final products. Additive manufacturing offers solutions for the fabrication of complex geometries and is preferred for manufacturing parts in a wide variety of applications. The fused filament fabrication (FFF) method is the additive manufacturing method with the most users worldwide. With the developing material science, it has been possible to produce composite structures and even metal products by going beyond the production of pure polymers in the FFF method. The mechanical properties of the materials produced by the FFF method change according to the production direction. In the FFF method, the production parameters have a significant influence on mechanical properties and material properties. One of the most important disadvantages of parts produced by additive manufacturing is that the surface quality of the parts after production is not good. In this thesis, the surface properties of composite structures produced using pure polyamide and polyamide matrix carbon fiber reinforced filaments by the FFF method were investigated. The production parameters used in the FFF method and the surface roughness performance of the samples depending on the direction were examined. The effect of layer thicknesses on surface roughness and the effect of fill orientation 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. It was concluded that the increase in the layer thickness increased the surface roughness, the -/+45 infill pattern

created higher roughness than the 0 and 90 orientations, and the surface quality could be increased by chemical surface modification.

Keywords: Polymeric Composites, Surface Roughness, Fused Filament Fabrication, Additive Manufacturing

Erimiş Filament Ekstrüzyonu Yöntemi ile Üretilen Kompozit Yapılardan Yöne Bağlı Yüzey Pürüzlülük Performansı ve Yüzey Pürüzlülüğünün İyileştirilmesi

Özet

Eklemeli imalat, bugün farklı endüstrilerde prototipler ve hatta nihai ürünler üretmek için kullanılmaktadır. Eklemeli imalat karmaşık geometrilerin imalatı için çözümler sunar ve çok çeşitli uygulamalarda parça imalatı için tercih edilir. Erimiş filament ekstrüzyonu (EFE) yöntemi, dünyada en çok kullanıcısı olan eklemeli üretim yöntemidir. Gelişen malzeme bilimi ile EFE yönteminde saf polimer üretimi ötesine geçerek kompozit yapılar ve hatta metal ürünler üretmek mümkün olmuştur. EFE yöntemi ile üretilen malzemelerin mekanik özellikleri üretim yönüne göre değişmektedir. EFE yönteminde üretim parametrelerinin mekanik özellikler ve malzeme özellikleri üzerinde büyük bir etkisi vardır. Eklemeli imalat ile üretilen parçaların en önemli dezavantajlarından biri de üretim sonrası parçanın yüzey kalitesinin iyi olmamasıdır. Bu tez çalışmasında, EFE yöntemi ile saf poliamid ve poliamid matrisli karbon fiber takviyeli filamanlar kullanılarak üretilen kompozit yapıların yüzey özellikleri incelenmiştir. EFE yönteminde kullanılan üretim parametrelerinin ve numunelerde yöne bağlı olarak meydana gelen yüzey pürüzlülük performansı incelenmiştir. Katman kalınlıklarının yüzey pürüzlülüğüne etkisi ve dolgu yönlenmesinin yüzey kalitesine etkisi ölçülmüştür. Ayrıca karbon fiber takviyesinin ve takviye oranının yüzey kalitesine olan etkisi ve tüm numunelere üretim sonrası uygulanan kimyasal yüzey işlemlerinin yüzey kalitesine olan etkileri araştırılmıştır. Katman kalınlığındaki artışın yüzey pürüzlülüğünü arttırdığı, -/+45 dolgu yönlenmesinin 0 ve 90 yönlenmelerine göre daha yüksek pürüzlülük oluşturduğu ve kimyasal yüzey modifikasyonu ile yüzey kalitesinin artırılabilceği sonuçlarına varılmıştır.

Anahtar Kelimeler: Polimerik Kompozitler, Yüzey Pürüzlülüğü, Erimiş Filaman Ekstrüzyonu, Eklemeli İmalat

My little munchkin PERA...

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Özlem DOĞRU

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List of Abbreviations

AM	Additive Manufacturing
FFF	Fused Filament Fabrication
PA	Polyamide
PP	Polypropylene
PC	Polycarbonates
PE	Polyethylene
PET	Polyethylene Terephthalate
PEK	Polyether Ketone
PEKK	Polyether Ketone Ketone
PES	Polyethersulfone
ABS	Acrylonitrile Butadiene Styrene
PLA	Polylactic acid
StL	Stereolithography
MJ	Material Jetting
ME	Material Extrusion
SLS	Selective Laser Sintering
CAD	Computer-aided Deesign
CAM	Computer-aided Manufacturing
ASTM	American Society for Testing and Materials,
ISO	International Standards Organization
SEM	Scanning Electron Microscopy
STL	Standard Triangle Language

CF	Carbon Fiber
GF	Glass Fiber
HF	Hybrid Fiber
USA	United States
Rpm	Revolutions per minute
T _g	Glass Transition Temperature
T _c	Crystallization Temperature
T _m	Melting Temperature

List of Symbols

C=O	Carbonyl group
(-OH)	Hydroxyl group
mm	millimeter
cP	Centipoise
η	Viscosity
pa.s	Pascal-second
°C	Celcius
MPa	Megapascal
GPa	Gigapascal
m/s	Meter per second
mm/s	Milimeter per second
Nm	Newton-meter
W/g	Watt per gram
J	Joule
nm	Nanometer
μm	Micrometer
kV	Kilovoltage
Hz	Hertz

Chapter 1

1 Introduction

Engineering work is critical at each of the stages of a product's design, manufacture, and implementation. Correct design and appropriate production method ensure that the final product exhibits the expected properties in the application area. A series of engineering studies can ensure that products are safe, economical, useful and sustainable in the field of application(1). All these stages begin with the determination of the material from which the product will be produced. It is expected that the selected material will meet the expected characteristics (environmental conditions, loads, weight, cost, etc.) from the final product. It must be able to safely meet parameters such as ambient temperature, pressure, etc. in the application area. In addition, it is important that the final product designed has a workable structure in order to reach the final geometry. It must exhibit the expected characteristics under the loads that affect the working conditions, and it is important for sustainability and accessibility that all these processes are cost-effective(2).

Table 1.1: Factors in Final Product Manufacturing Processes

Design	Production	Application
Reproducible Geometry	Formability	Operating in Ambient Conditions
Ergonomics	Affordable Cost	Strength
Fluent Apperance	Sustainable Material	Lightness
		Sustainability
		Affordable price

Materials in materials science; metals, ceramics, polymers and composites. After the material of a designed product is determined, it is very critical to determine the production method. Materials can be shaped with different production methods. It is also possible to process different materials with similar manufacturing methods. Production methods have advantages and disadvantages over each other. In determining the production method, the relationship between the design and the application area of the final product should be considered. The criteria in Table 1.2 should be considered in determining the production method.

Table 1.2: Determination of Production Method

Criteria	Effect
Material Type	Hardness, state of matter, conductivity, thermal resistance limits the production methods.
Geometry	Complex geometry limits production methods.
Production Volume	The final product amount affects the production method's cost and duration.
Production Capacity	The amount of production for a given period influences the choice of method.
Cost	Depending on the production volume and capacity, a cost-effective process is important.
Sustainability	It should be considered on the axes of environmentalism, economic, and accessibility.

1.1 Polymer and Fabrication Techniques

In the modern plastics and composites industry, the polymer is frequently used interchangeably with plastic(3). A chemical substance known as a polymer contains molecules connected in extended, repeating chains. All polymers are formed by the polymerization process, where their constituent elements, called monomers, react together to form polymer chains, i.e., 3-dimensional networks that form polymer bonds.

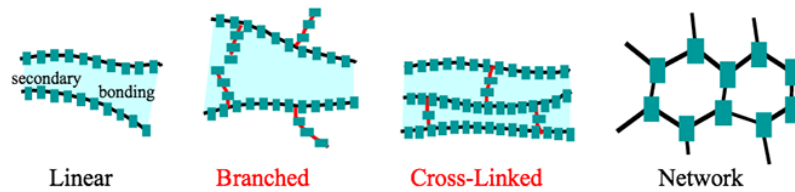


Figure 1.1: Polymer Chains

Polymers have special qualities due to their structure that can be tailored for various uses. They can be found in many different products, including common household items, clothing, toys, building supplies, and insulation(4).

Both man-made and naturally occurring polymers are available. There are synthetic and organic polymers(5). Humanity has been using rubber, a natural polymer, for thousands of years. The natural molecular polymer chains produced by the material give it excellent elastic properties. Shellac, a resin made by the polish beetle in India and Thailand and used as a primer, sealant, and varnish, is another natural polymer(4). Cellulose, an organic substance found in plant cell walls, is the most common natural polymer on the planet(4). It is used to make textiles, paper goods, and other materials such as cellophane(3).

Polystyrene and polyethylene are two examples of synthetic polymers(5). Polyethylene is the most widely used plastic in the world and is used in everything from storage containers to shopping bags (the material used for packing peanuts and disposable containers). Thermoplastics are recyclable synthetic polymers, whereas thermosets are non-recyclable and permanently molded after curing. Some are elastic like rubber (elastomers)(5).

Polymer materials are divided into three groups thermosetting, thermoplastics and elastomers. Polymers can be categorized for better understanding as shown in Figure 1.2.

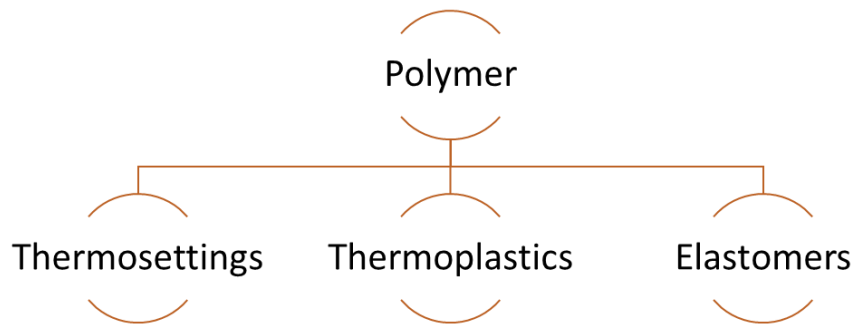


Figure 1.2: Classification of Polymers (6,7)

Polymers exhibit different properties according to their chemical structures, bonds and chains. Polymers, which find use in many different fields today, are obtained from natural or synthetic sources. Properties and examples of elastomers, thermoplastics and thermosets polymers are given in Table 3.

Table 1.3: Properties of Thermoset, Thermoplastic and Elastomer

	Thermoset	Thermoplastic	Elastomer
Properties	High stiffness and strength	Improved impact resistance	Excellent impact resistance
	Adhesion properties	Higher fracture toughness	Very high elongation
		Moderate stiffness and strength	Low stiffness and strength
Examples	Epoxy	Polyamide	Polyurethane
	Polyester	Polyether ketone (PEK)	Rubber
	Bismaleimide		
	Vinyl ester	Polyether ketone ketone (PEKK)	

1.1.1 Elastomers

Elastomers are loosely cross-linked polymers. They have the properties of rubber in terms of flexibility and elasticity(8). Long randomly wound, loosely cross-linked materials can be easily stretched, but will return to their original shape when force or stress is removed. Plastic deformation zones are quite large. They are slightly cross-

linked and amorphous, with a glass transition temperature well below room temperature.

The elastomer's primary uses are gaskets, molded flexible parts, and adhesives, and it is used in vehicle manufacturing, food manufacturing, scientific applications, and chemical processes(5,9).

1.1.2 Thermosets

Thermoset polymers are formed as a result of a chemical reaction in two steps. First, they are produced as macromolecular chains like reactive thermoplastics. In the second stage, these macromolecular chains form cross-links with the effect of temperature and pressure(10). The cross-linking of thermoset polymers strengthens the molecular bonds and makes the polymer durable. Thermoset polymers do not soften when heated, due to the cross-linking molecular bonds they have. As these polymers are reheated, they do not become fluid, on the contrary, they decompose with the increase in temperature(11). Today's polymer sector has challenges with recycling thermoset polymers, which are widely utilized.

1.1.3 Thermoplastics

Polymers that soften or melt when heated are called thermoplastic polymers. The macromolecules that make up thermoplastic polymers are bonded to each other by weak van der Waals forces(12).

When thermoplastic polymers are heated, the intermolecular forces are significantly reduced, so the material begins to soften. With the increase in temperature, the material becomes flexible and becomes a viscous liquid at high temperatures(13). It becomes solid again when allowed to cool. This cycle can be repeated many times, which is an advantage of recycling. However, of course, the properties of thermoplastic polymers deteriorate with more than one heating-cooling cycle.



Figure 1.3: Ultramid B40LN Polyamide Pellet

Thermoplastic materials are divided into two groups according to the arrangement of the macromolecules they have. These are called crystalline and amorphous structures. While the crystalline macromolecules are characterized in an ordered array, the amorphous macromolecules are randomly arrayed (14). Polymers such as polyamide can have a high degree of crystallinity(15). However, it is not possible to make a perfect crystalline thermoplastic because of the complex structures. Therefore, it can be called semi-crystalline or partially crystalline (16). The crystallization of polymers is largely dependent on the thermal processes during their production and results from the more intense aggregation of macromolecules. Crystal structure affects the mechanical properties of polymers. Unlike the recycling problems of thermoset polymers, thermoplastic polymers can be recycled and reused. In addition, biodegradable thermoplastic polymers are also widely used today. These advantages of thermoplastics have paved the way for their use as matrix material in polymer matrix composites. In many areas, studies are carried out to replace thermoset matrices. It is a critical area for the solution of a sustainable world and environmental problems.

1.2 Composite Materials and Manufacturing

Combining two materials with various structural qualities creates a composite material (physical and chemical). These substances come together to create a unique substance. When they are combined, a strong feature such as stronger, lighter will emerge and these features can be improved. The combined materials do not mix completely and do not completely lose their chemical properties(17).

Composites consist of a matrix reinforced with reinforcing materials such as particles or fibers (such as glass, carbon or aramid)(18).

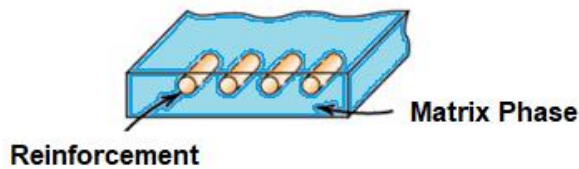


Figure 1.4: Composite Structure Form

The matrix transmits the weight between the fibers and shields them from external and environmental harm. In order to reinforce the matrix and prevent fractures and breakdowns, fibers provide it strength and rigidity. Composites may include fillers, additives, and core components that are intended to enhance the production process, the final product's look, and its functionality(17). From the matrix and fiber reinforcement, which cannot form composite materials, the matrix forms the main component. Its main task is to wrap the reinforcement component around it and to keep it together and in the desired form.

The matrix keeps the reinforcement phase together by preserving the designed material form and transmits any applied force to the reinforcement phase without being destroyed, via the interface bond, and distributes the force. An ideal matrix material should be able to wrap the fibers properly and form a good interface. The mechanical properties of the matrix material play an important role in the performance of the load-bearing reinforcing elements in composite structures. Composite materials are divided into 3 according to the matrix material(17,19).

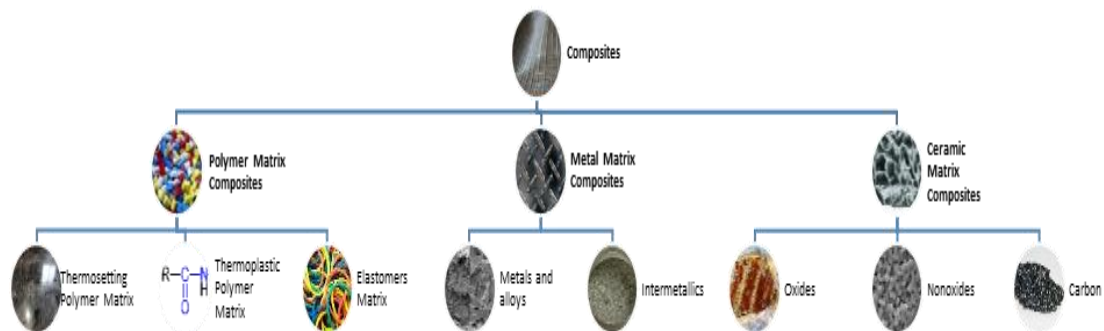


Figure 1.5: Classification of Composites

According to their resources, fibers are split into two categories: natural and synthetic. Natural fibers are produced from renewable resources and are the next generation supplements to replace petroleum-based materials and synthetic fibers. Natural fibers are fibers obtained from plants, animals or minerals. Although natural fibers are low

cost and biodegradable, they do not have high thermal resistance. It also has serious problems with moisture absorption(20).

The flexibility of synthetic fibers is very high. They can carry heavy loads without breaking. Fabrics made from these fibers are cheaper and more durable compared to natural fibers(21).

It also has some serious disadvantages such as high costs, high energy consumption in the processing and manufacturing processes, recycling problems and non-renewable properties, CO₂ emissions and health hazards when inhaled.



Figure 1.6: Carbon Fibers

1.2.1 Composites with Polymer Matrix

Polymer matrix composites are generally petrochemical based products. Polymer matrix composites are the most widely used composites in the industry because they are inexpensive and easy to handle compared to other composites. Polymeric composites are materials that are resistant to corrosion, suitable for long-term use, easy to process, formable, and have a high load capacity per unit mass (22).

Polymer matrix composites are preferred in aerospace, aerospace, and defense industries. These composites, which are used for reinforcement, are generally used by reinforcing with glass and carbon.

Composites with polymer matrix are divided into two as thermo-set and thermoplastic matrix composites.

Table 1.4: Polymer Matrix and Properties(18,23,24)

	Thermosetting	Thermoplastic
Properties	High stiffness and strength Adhesion properties	Better impact resistance Higher fracture toughness Moderate stiffness and strength
Examples	Epoxy Polyester Bismaleimide Vinyl ester	Polyamide (PA) Polycarbonates (PC) Polyethylene (PE) Polyethylene Terephthalate (PET) Polyether ketone ketone (PEKK)

1.2.1.1 Thermosetting Matrix Composites

Thermoset matrix composites are composite materials in which thermoset polymers are used as matrix. With thermoset polymers, they are liquid at room temperature by nature, and after curing, they cannot be restored to their original state when reheated. The use of thermoset matrices is limited due to disadvantages such as high raw material cost, difficulty in production, recycling problems and low forming ability at room temperature. Polymers such as polyester, vinyl ester and epoxy are often used as matrix in thermoset composite materials(25). Among these thermoset polymers, polyester is more economical than others and does not require heating after curing. However, its mechanical strength and adhesion are lower. Epoxy, on the other hand, is a thermoset with high costs but the highest mechanical properties. It is a formulation of vinyl ester, epoxy resin and methacrylic acid, with mechanical performance between epoxy and polyester(26). Thermoset matrix composite materials are reinforced with reinforcing elements such as particles and fibers to form composite materials.

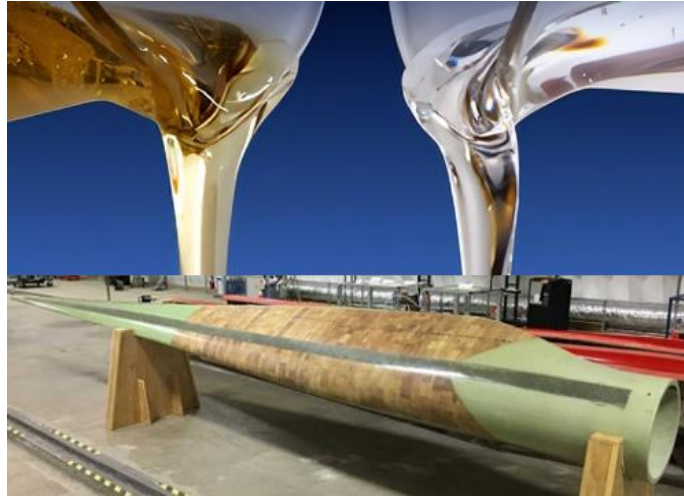


Figure 1.7: Epoxy Matrix Wind Turbine Blade(27)

Thermoset matrix composite product is shown as figure 1.7 and production methods are given in figure 1.8.

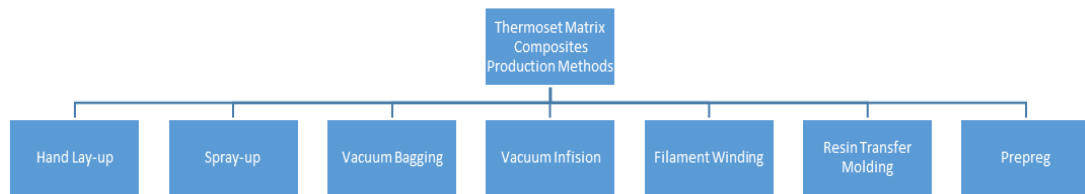


Figure 1.8: Thermoset matrix composite production methods

1.2.1.2 Thermoplastic Matrix Composites

It is the name given to composite materials in which thermoplastic polymers are used as matrix material. Thermoplastics are inherently solid at room temperature and become fluid by decreasing their viscosity with heat(28,29). The most important advantage of thermoplastics is that they become solid again when they lose heat. Thermoplastic materials are reinforced with reinforcement elements in different forms to form composite materials. Fibers of different forms and sizes or particles of different sizes can be used as reinforcement. The most important advantage of thermoplastic matrix composites is their recyclability. Studies in this field are critical for a sustainable world. The polymers in table 1.5, which show high performance among thermoplastic polymers, are used as matrix materials and find many application areas. Thermoplastics such as PEEK (Polyether ether ketone), PPS (Polyphenylensulfid), PA (Polyamide), PC (Polycarbonate), PLA (Polylactic acid) and ABS (Acrylonitrile Butadiene Styrene) are matrix material types commonly used in industrial applications(30,31).

Table 1.5: Properties of Selected Thermoplastic Matrix

	PEEK	PPS	PA	PC	PLA	ABS
Density (G/Cm³)	1.30-1.32	1.36	1.03-1.16	1.2	1.25	0.9-1.53
Tensile Strength (Yield) (Mpa)	90-11	28-93	40-86	62.1	59	40
Elongation (%)	25	6	60	110	7	50
Melting Point (°C)	340-344	280-282	211-265	265	190-220	200-280

Table 1.5 lists some characteristics of thermoplastic polymers deemed appropriate for applications requiring high performance. These polymers have a glass transition temperature (T_g) that is rather high. These characteristics enable them to exhibit good dimensional stability at high temperatures. The size and flexibility of additional chemical groups or chain links affect the true value of T_g. For industrial applications, the high T_g value expands the application area. Thermoplastic matrix composite production methods are included in figure 1.9

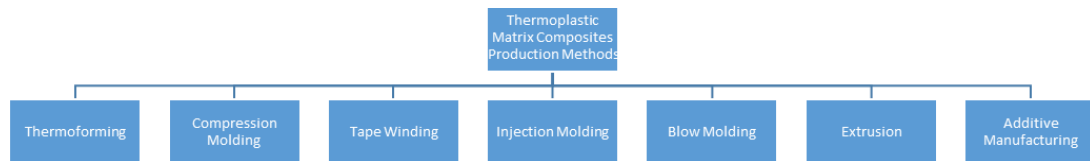


Figure 1.9: Thermoplastic matrix composite production methods

1.2.2 Thermoplastic Matrix Composite production methods

1.2.2.1 Thermoforming

Thermoforming is a plastic-forming process in general. At the beginning of the process, thin plastic sheets are first heated for easy shaping. The heated layer is shaped and cooled by vacuum or pressure. Thus, the final product is obtained. Both the automobile and toy sectors frequently employ thermoform packaging.

In addition to its advantages such as low cost, fast turnaround time, easy prototype production, it also has disadvantages such as complex shape constraints.

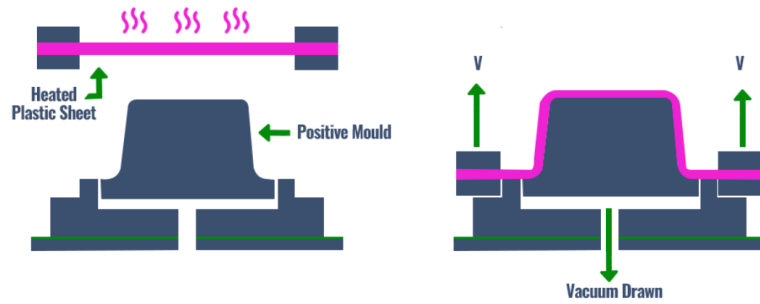


Figure 1.10: Thermoforming Principle(32)

1.2.2.2 Compression Molding

Compression molding is the process of forming preformed or heated raw material in a heated mold cavity. The material is shaped and cooled under high pressure in a closed mold.

With this method, complex parts of various lengths and thicknesses can be produced. The final products produced have high strength. In addition, it is widely used because of its low cost and high production capacity.

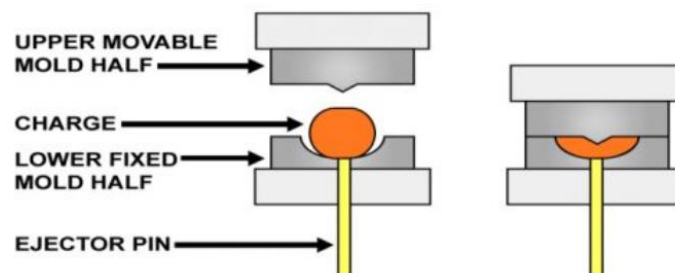


Figure 1.11: Compression Molding(32)

1.2.2.3 Tape Winding

The strip winding process allows composite parts made of strip to be produced automatically and efficiently in the first step forming process without additional curing steps. Using tape wrapping technology, high pressure vessels and pipes can be designed and manufactured in large volumes to meet the increasing demands of industrial applications.

The process has advantages such as short cycle time and easy quality control.

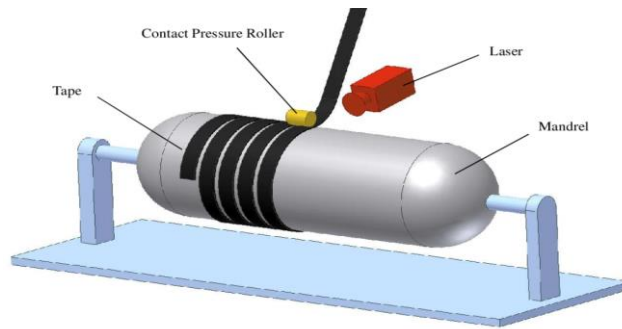


Figure 1.12: Tape Winding(33)

1.2.2.4 Injection Molding

Injection molding is a method of obtaining a final product by injecting heat-melted plastic materials into a mold, followed by cooling and solidification. Injection molding is the most widely used method for forming thermoplastics.

Injection molding is a manufacturing process that allows parts to be produced in large volumes. Typically used as a mass production process to produce thousands of identical products. Injection molding materials include metals, glass, polymers and food products, but are most commonly used in the manufacture of thermoplastics(34).

Although the initial design and mold costs are high, the cost decreases as the number of parts increases in large volume and mass production. Fire rate is at minimum.

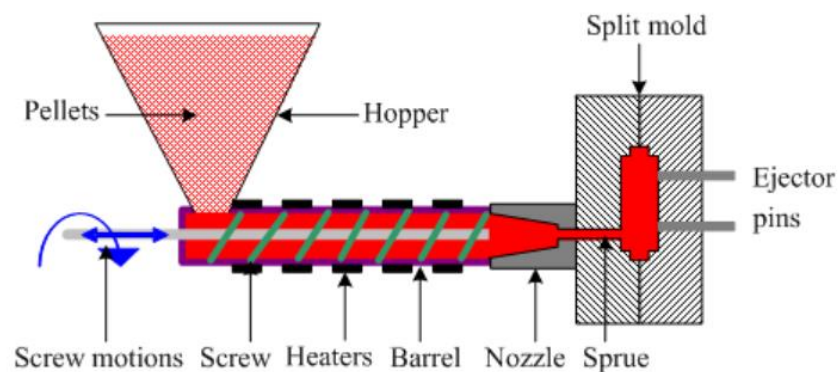


Figure 1.13: Injection Molding(35)

1.2.2.5 Extrusion

The word extrusion is taken from the Latin word extrudere, which means out (ex) and push (trudere). In the extrusion process, a pellet or dry powder plastic is heated and passed through a die(36,37). The screws will melt and mix the material and force it to

come out through the opening in the mold. An extruder can be single screw or twin screw. The material being extruded determines which extrusion technology is required. Twin screw extruders are mainly used to combine with different plasticizers, fillers, colorants and other components in line with basic plastics. Extrusion is suitable for making pipe, rod, profile, film or sheet plastic products of various widths and thicknesses(38).

The fibers added to strengthen thermoplastic matrix composites can be in continuous form or in short forms. While methods such as thermoforming, tape winding and compression molding are used in the production of thermoplastic matrix composites containing continuous fiber reinforcement. Short fiber reinforced thermoplastic composites are produced in injection molding, blow molding and extrusion methods. One of the innovative production methods of thermoplastic composites is additive manufacturing methods. It is possible to produce short and continuous fiber reinforced thermoplastic composites with additive manufacturing methods(39,40).

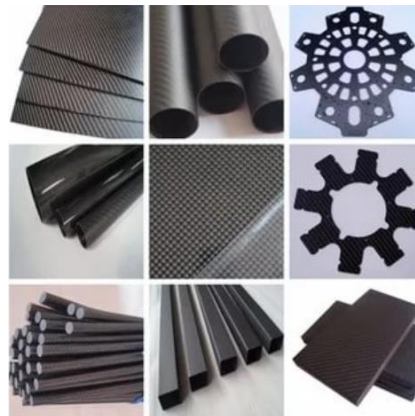


Figure 1.14: Thermoplastic Composite Products

1.3 Additive Manufacturing

Additive manufacturing is the formalized term for methods formerly called Rapid Prototyping and now commonly referred to as 3D Printing. Contrary to traditional machining techniques, additive manufacturing refers to the processing of computer-aided drawing models (CAD models) by layering on materials (41). The concept of Rapid Prototyping is defined as the process of quickly creating a representative system or part of the product before the final version is formed or commercialized during the product development stages. In short, it is the presentation of the physical product in

order to quickly transform the design into a product. A prototype or base model is produced from which the final product will be derived(42). Improvements in the quality of the parts produced by additive manufacturing methods ensure that the outputs are very close to the final product. Today, many parts can be produced directly by pending manufacturing methods. Therefore, additive manufacturing methods have gone beyond prototype development in many different industries and are also used to produce final products. The ISO/ASTM 52900:2015 standard (43) contains the fundamental ideas of additive manufacturing techniques. A technical committee within ASTM International is working to establish new terminology in this field.

The principle of operation, the type of material employed, and the type of energy used are all taken into consideration when categorizing methods that combine components by stacking layers upon layers. Additive manufacturing techniques are categorized into seven separate categories in the American Society for Testing and Materials (ASTM) standard, which is based on the manufacturing process. These; Binder Sputtering, Directed Energy Deposition, Material Extrusion, Material Sputtering, Powder Bed Fusion, Sheet Lamination and photopolymerizationThe cold spray technique has also been created and used recently in addition to the ASTM classification (44). Table 1.6 includes additive manufacturing methods and their definitions.

Table 1.6: Additive Manufacturing Methods (ASTM)(35)

Methods	Description
Binder Jetting	To joining powder components, a liquid bonding agent is carefully placed
Directed Energy Deposition	Materials are fused by melting as the substance is deposited using concentrated heat energy
Material Extrusion	A nozzle or aperture is used to disperse material in a certain way
Material Jetting	Build-material droplets are deliberately deposited
Powder Bed Fusion	Areas of a powder bed are fused using thermal energy
Sheet Lamination	An item is made by bonding material sheets together
Photopolimerization	By using light-activated polymerisation, liquid photopolymer in a vat is selectively cured

The basic principle of additive manufacturing technologies is that a model created using a three-dimensional Computer Aided Design (3D CAD) system can be produced

directly without the need for process planning(45). Although this is not as simple as it may seem at first glance, additive manufacturing technologies significantly simplify the process of producing objects with complex geometry in one piece, using CAD data directly. The following process steps should be clarified in the parts production processes with the general production methods.

- Analysis of part geometry
- the order of operation of the part,
- what equipment to use
- which processes to use
- assembly stages
- post-processing

Parts that are difficult to produce with conventional production methods, impossible to produce in one piece, or requiring multiple processes can be easily produced with additive manufacturing methods. In the production phase of additive manufacturing methods, some basic dimensional details are needed, the parameters suitable for the additive manufacturing method are created and the finishing steps are needed(46).

When we separate the additive manufacturing methods according to the type of material used, the main methods preferred in the production of polymer products by additive manufacturing methods are FFF and Stereolithography (SLA) in the material extrusion category, selective laser sintering (SLS) in the powder bed fusion category and polyjet method that produces parts by binder spraying(47). More than 51% of the materials processed by additive manufacturing are polymer materials(48). The FFF method is the most widely used and favored technique for molding polymers in additive manufacturing. The FFF method's operating concept is shown in Figure 1.15.

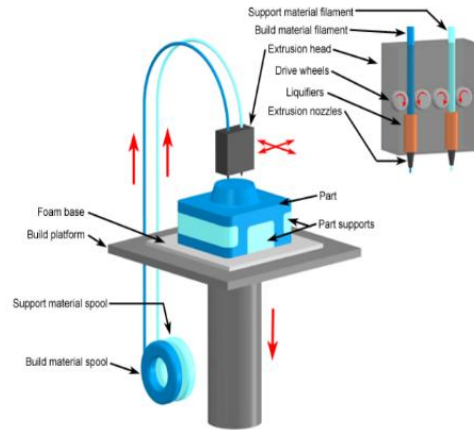


Figure 1.15: Illustration of fused filament fabrication (49)

1.3.1 Stereolithography (SLA)

The first 3D printing process is called SLA. Chuck Hull developed stereolithography, got a patent for it, and began selling it in 1986. The process of creating layer-by-layer models by curing photosensitive polymers with ultraviolet (UV) light that acts as a catalyst is known as SL(50).

Solid state laser is typically used by SLA printers to cure items. Dot laser-based 3D printing has the drawback of potentially taking longer to trace a cross-section of an item than DLP, which cures each layer individually.

The SL method, shown in Figure 1.16, is among the most popular rapid prototyping techniques. Today, a layer precision of 10 μ m can be achieved with the micro stereolithography method (50). SL printing requires a delicate process. Excessive curing can cause a break between the substrate and the top printed layer. Also, because the resin is very viscous (hatched line shape), the layer thickness can vary. The type of stereolithography that uses more than one material is called "multi-material stereolithography"(44).

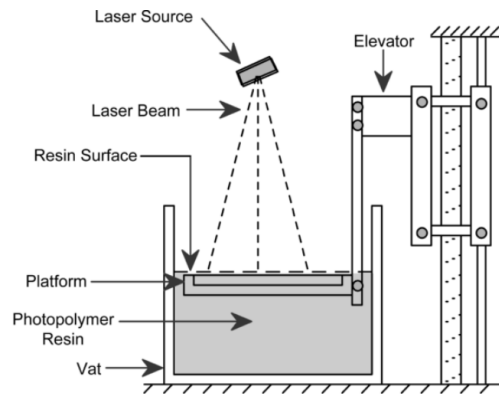


Figure 1.16: Stereolithography (SLA)(51)

1.3.2 Selective Laser Sintering (SLS)

Selective laser sintering is the term used to describe the process of creating an item using powder bed fusion technology and polymer powder (SLS). The polymer powder is first heated to a temperature slightly below the polymer's melting point. A very fine layer of powdered material is then deposited on the platform. After that, a CO₂ or fiber laser starts scanning the surface(52). The cross-section of the part is solidified in the appropriate layer by selective sintering of the laser powder. When the entire section in the relevant layer has been scanned, the build platform moves down the layer thickness. The coating equipment then deposits a new layer of powder on the laser scanned layer, and the laser sinters the next section of the object onto the previously solidified sections(53).

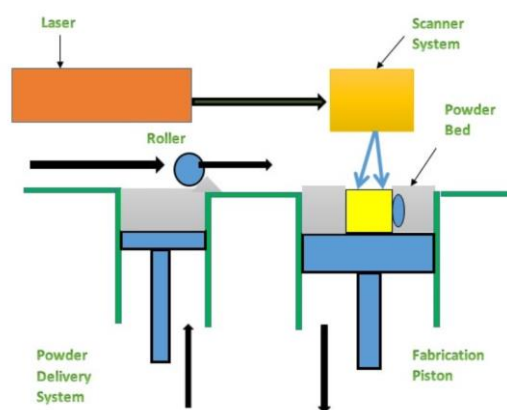


Figure 1.17: Selective Laser Sintering (SLS)(54)

Repeat these actions until all items have been fully made. Until the conclusion of manufacturing, unsintered powder is left in situ to support the item. As a result, fewer or no support structures are required.

Micro-selective laser sintering (μ SLS) is essentially selective laser sintering (SLS) on a small scale and is often referred to as micro-laser sintering. While SLS usually refers to a process with plastics, here μ SLS more commonly refers to a laser sintering process with metals. μ SLS can produce true 3D metal parts with resolution below 5 μ m and throughput greater than 60 mm³/hr(55).

Nylon-based powders are melted into solid plastic using selective laser sintering (SLS). SLS components may support moving hinges and snap connections and are strong, ideal for functional testing, and constructed of genuine thermoplastic material. The components are more robust than the SL, although their surfaces are rougher. SLS does not require support structures, so it is possible to enclose multiple parts of the entire build platform within a single structure. This makes it suitable for higher part positioning than other 3D printing processes. Many SLS parts are used today for prototyping designs to be injection molded.

1.3.3 Polyjet

PolyJet is a 3D printing technology that creates parts by spraying thousands of photopolymer droplets onto a build platform and solidifying them with a UV light. It is one of the fastest and most accurate 3D printing technologies currently available. The anatomy and printing process of the PolyJet 3D printer is the same as a material jet 3D printer. It can produce parts with multiple properties such as colors and materials(56).

PolyJet printers consist of a material hopper, a build platform, and a section where UV lights and jet print heads are mounted. Before starting to print, the photopolymer resin must be poured into the material container and heated(57). This allows the substance to reach the desired viscosity.

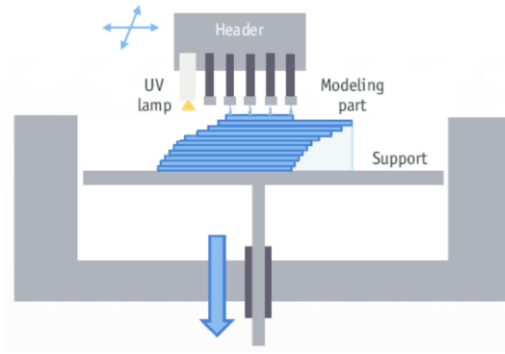


Figure 1.18: Polyjet(58)

The carriage moves along the build platform's X-axis to start the printing process. The print heads move to selectively spray the resin onto the build platform in the form of droplets. Sprayed polymers are solidified with UV lights immediately after. Because there is more than one print head, different materials can be printed at the same time. The rendering platform advances one layer once each layer is finished, and this procedure continues until the portion is complete. Even bio-resins may be used with PolyJet 3D printers to produce a variety of materials(59).

1.3.4 Fused Filament Fabrication (FFF)

FDM (Fused Deposition Modeling) or FFF (Fused Filament Fabrication) is a technology used in three-dimensional printing technologies to create strong, durable and dimensionally stable parts with its dimensional accuracy and repeatability(60,61). It is the most widely used 3D printing technology. It is an additive manufacturing technology that uses thermoplastic as a raw material. It is based on the principle of melting thermoplastic parts made into filaments and applying them to the production table layer by layer. All geometries in your mind can be created with the FFF method. For this reason, you can use the parts produced with this technology as end-use components in the aerospace industry, in the defense industry or in the automotive industry to meet all your prototype needs.

The most extensively used and reasonably priced forms of 3D printing technology globally are FFF devices. In principle, it operates by feeding a temperature-controllable printer nozzle into the extrusion head of a 3D printer that travels in a cartesian system. The printer nozzle is heated to the desired temperature, whereupon a motor pushes the filament through the heated nozzle, causing it to melt. While the

pressure required for extrusion is provided by the engine pushing the filament, the amount of material sent by this engine per unit time is determined. In the FFF printer, a plastic filament is extruded layer by layer by melting it into the build platform by this nozzle. It is a quick and affordable approach to creating physical models. FFF has certain applications for functional testing, however, the method is constrained by the need for parts with weak construction and somewhat rough surface finishes(62).

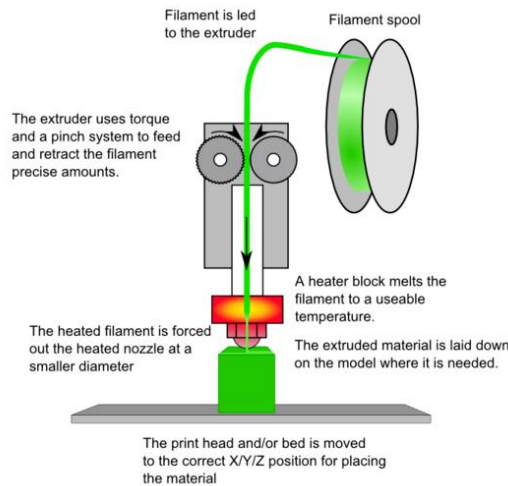


Figure 1.19: FFF(63)

With the increase in the variety of materials used in this field, the development of 3D printers that can move more precisely, the development of post-production material-specific finishing techniques, and most importantly, the research studies carried out in this field are making progress in the way of being used in the production of final products day by day

1.4 Surface Characterization Techniques

Various methods are used to study the surface properties of a material. In order to examine the surface properties of the manufactured and processed parts, surface roughness measurement, imaging with an optical microscope, contact angle measurement, and examination with an electron microscope are performed(64,65).

1.4.1 Surface Roughness

Average roughness, which is sometimes represented as "Ra," is a standard unit of measurement for surface roughness. Ra is the surface's computed average between peaks and valleys. Less variance between peaks and troughs on a surface results in a smoother surface as Ra values decrease. For instance, a lego block will have a low Ra value, similar to the touchpad on a laptop(66). These items become extremely rough and coarser with a greater Ra value, making them unfit for their perhaps desired application. This comparison of Ra values highlights how crucial it is to decide on the required level of surface roughness for a product before the manufacturing process starts. Without making these decisions, a product's machining surfaces may not be as accurate as desired(5). The charts below demonstrate how Ra (the numerical mean of all peaks and valleys during the course of the test) and Rz differ from one another (mean of consecutive highest peaks and lowest valleys).



Figure 1.20: Sample of Surface Roughness Result (67)

Ra - The numerical average of all peaks and valleys over the length of the test.

Rz - Average of consecutive highest peaks and lowest valleys. The distance between the highest peak and the lowest valley, the distance between the second highest peak and the second lowest valley, etc. This is usually done for the five largest deviations and then an average is calculated.

Rp - The calculated distance between the highest peak of the profile and the mean line within the evaluation length.

R_v - The calculated distance between the lowest valley of the profile and the mean line within the evaluation length.

R_{max} - The largest consecutive deviation between the highest peak and lowest valley calculated within the evaluation length.

RMS - Calculated within the rating length, this is the root mean square mean of the change in profile height from the mean line(67).



Figure 1.21: Surface Roughness Device (67)

1.4.2 Contact Angle Measurement

A quantitative measure of wetting a solid with a liquid is called the contact angle (θ). The liquid forms an angle on the solid surface. This angle varies according to the type of solid and liquid contacted(68). The contact angle is the degree of wettability, and the magnitude of this angle depends on the magnitude of the cohesion and adhesion forces. Materials are defined as hydrophobic if the contact angle is greater than 90 degrees – hydrophobic (does not wet), if the contact angle is less than 90 degrees – hydrophilic (wetting), if the contact angle is greater than 140 degrees – super hydrophobic, and if the contact angle is very close to 0 degrees – super hydrophilic(69,70).



Figure 1.22: Contact Angle Device(71)

1.4.3 Confocal Microscope

Using fluorescent optics in a confocal microscope, laser light focuses on a specific spot in the sample, causing emission of fluorescent light at that point. By scanning the sample in a raster pattern, images of a single optical plane are created. 3D objects can be visualized by scanning several optical planes and stacking them using an appropriate microscopy deconvolution software (z-stack).

Confocal microscopy offers several advantages over traditional wide-field optical microscopy, including the ability to control depth of field, the elimination or reduction of background information away from the focal plane (which causes image degradation), and the ability to collect serial optical sections from thick samples.

Chapter 2

2 Experimental

Plastic injection molding is a manufacturing method that is widely used in polymer production, especially in the automotive and machinery sectors. In order to manufacture a polymer product with this method, appropriate mold design and manufacturing is a difficult, time-consuming and costly process. This approach is inadequate considering evolving technology and rising standards for quality, particularly when producing delicate and complicated parts in tiny quantities. The developments in additive manufacturing methods and the beginning of obtaining high quality products create important opportunities at this point. Additive manufacturing is a technology used in the manufacture of prototypes and products, and this technology has several advantages, such as the production of complex geometries without waste of material and the need for expensive molding machines, finishing and machining tools. However, it also has disadvantages regarding geometric tolerance, precision, surface finish and strength.

The application areas for these techniques will be expanded once the flaws in the surface quality of the objects created by additive manufacturing are fixed. Products produced using pure thermoplastic filaments with the FFF method show lower mechanical properties in terms of strength and functionality than parts produced by injection. This situation restricts the manufacturing of the parts to be used as the final product by the additive manufacturing method. Various studies on the improvement of the surface quality of pure polymer materials produced by the FFF method are available in the literature. Today, however, it is possible to produce products that can be used in engineering applications with higher mechanical properties with the FFF method. The use of composite filaments is the most important indicator of this. In this thesis, the focus is on composite products produced by the FFF method. In the thesis

study, studies were carried out to measure the surface roughness value and to make the surface quality smoother on the parts produced by the FFF method using polyamide matrix fiber reinforced materials. The effect of infill pattern and layer thickness on the surface properties was investigated with the FFF method.

2.1 Materials

2.1.1 Polyamide

Polyamides are crystalline polymers typically produced by condensation of a diacid and a diamine. Polyamide (PA) is an important thermoplastic with amide bond ($-\text{NH}-\text{C}=\text{O}$) 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.(48). Polyamide is a material that is included in the thermoplastic polymer types and is used in many engineering applications. It is resistant to chemicals and has high mechanical properties.

The matrix material of the filaments used in the thesis study is the Ultramid B40LN product of BASF company. The density of the polyamide product is 1.13g/cm³ and the relative viscosity value is 4. Pure polyamide was used as the control group. The effects of fiber reinforcement at different ratios on the surface roughness and surface energy of the samples produced by additive manufacturing were investigated.

2.1.2 Carbon Fiber

The filaments used in the thesis include AC4102 chopped carbon fibers of Dowaksa company. Polyamide matrix filaments containing 10% and 20% by weight of short carbon fiber were used. Chopped carbon fibers have a density of 1.73g/cm³, with a tensile strength of 4200MPa and a tensile modulus of 240GPa. The diameters of carbon fibers are 7 μ and their length is 6mm.



Figure 2.1: PA6CF20 Filament

2.1.3 Formic Acid

Many studies have reported that formic acid has a corrosive effect on polyamide (77). For this reason, it is preferred for surface modification. Formic acid, CH_2O_2 (98%+ pure), was obtained from ACROS Organics and used in liquid form.



Figure 2.2: Formic Acid

2.2 Methods

2.2.1 FFF Method

By using pure polyamide, 10% by weight carbon fiber reinforced and 20% by weight carbon fiber reinforced filaments, sample production for optical imaging of the surface, surface energy and surface roughness measurement was carried out by FFF method. As seen in figure 2.3, the sample dimensions are 3mm thick and have a width of 12*120mm. The samples were drawn with Autodesk Fusion 360 CAD program.

The production parameters of FFF devices are adjusted with CAM programs. CAD data created in stl format were converted to gcode file using the CAM program called CURA.

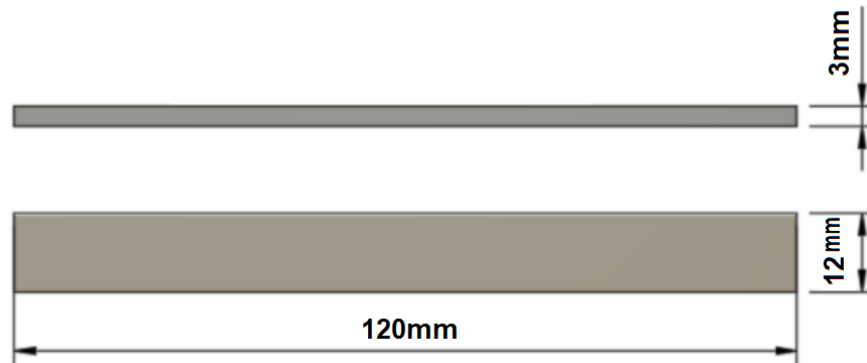


Figure 2.3: Produced Samples Dimensions

The production of all samples was carried out at the University of Alberta Multi-Functional Laboratory with the Ultimaker S5 model device.

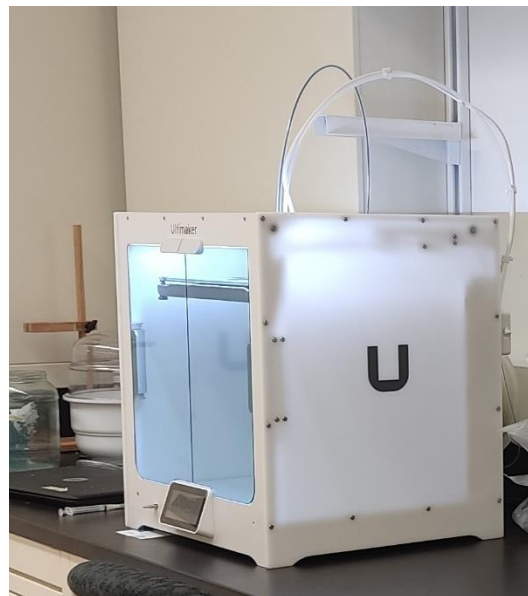


Figure 2.4: Ultimaker S5 FFF Device

The characteristics of the device where sample production is carried out are given in Table 2.1.

Table 2.1: Ultimaker S5 FFF Device Properties

Nozzle Temperature	~280°C (CC Nozzle: ~300°C)
Printing Volume	330 X 240 X 300 Mm
Xyz Accuracy	X;6.9, Y;6.9, Z;2.5 Micron
Layer Sensibility	20 - 200 Micron
Filament Diameter	2.85mm
Print Head Travel Speed	30 - 300 Mm/S
Build Plate Temperature	20 – 140°C

All samples were produced with an ultimaker printcore CC nozzle. This special nozzle with a sapphire tip 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.

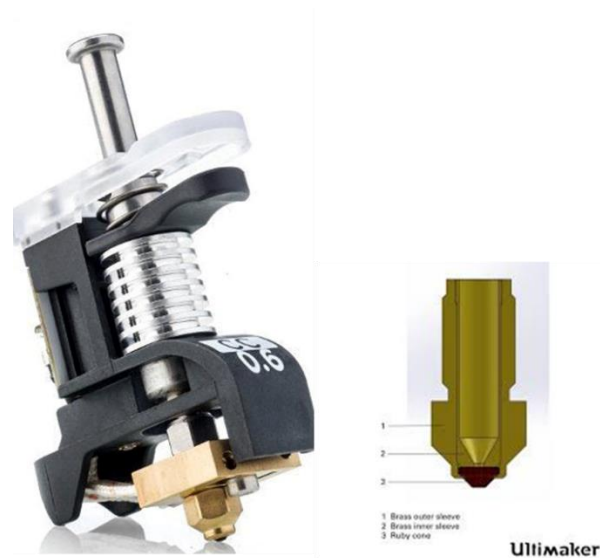


Figure 2.5: Printcore CC Nozzle

Each material group is produced in 3 different infill patterns and 3 different layer thicknesses. The effect of the infill pattern on the surface properties in the X-Y axis was investigated.

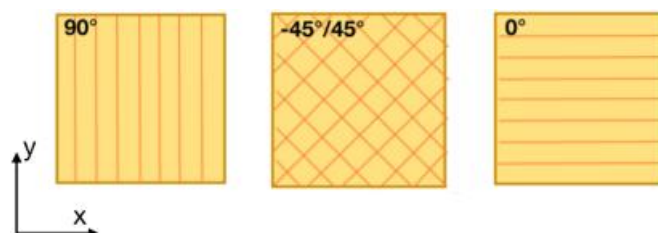


Figure 2.6: Infill Patterns

In addition, the effect of layer thickness on the surface properties in the Z axis was investigated.

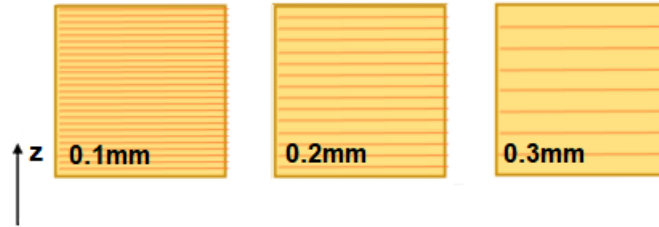


Figure 2.7: Layer Thickness

Using 3 different materials, 6 different samples were produced from each material group. Since surface modification will be applied to each sample group, 5 productions were made.







Infill Pattern	Layer Thickness	
0 Degree		0.1mm 
45 Degree		0.2mm 
90 Degree		0.3mm 

Figure 2.8: Pure PA6 Samples

Infill Pattern	Layer Thickness
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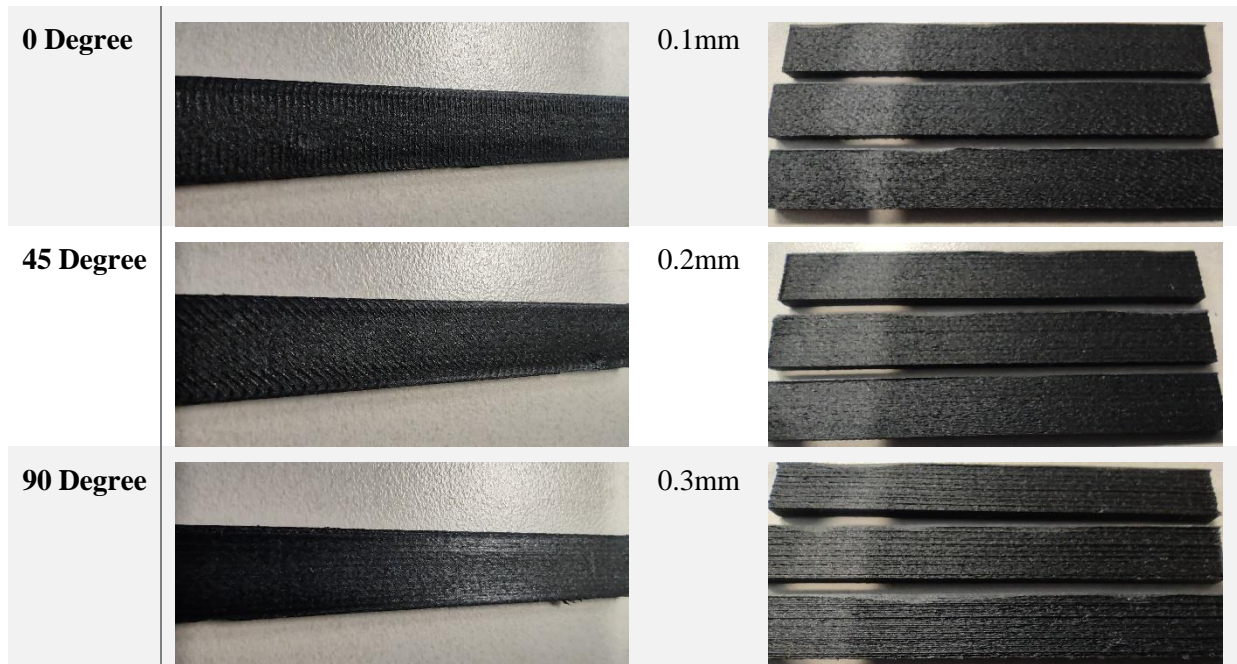


Figure 2.9: PA6CF10 Samples

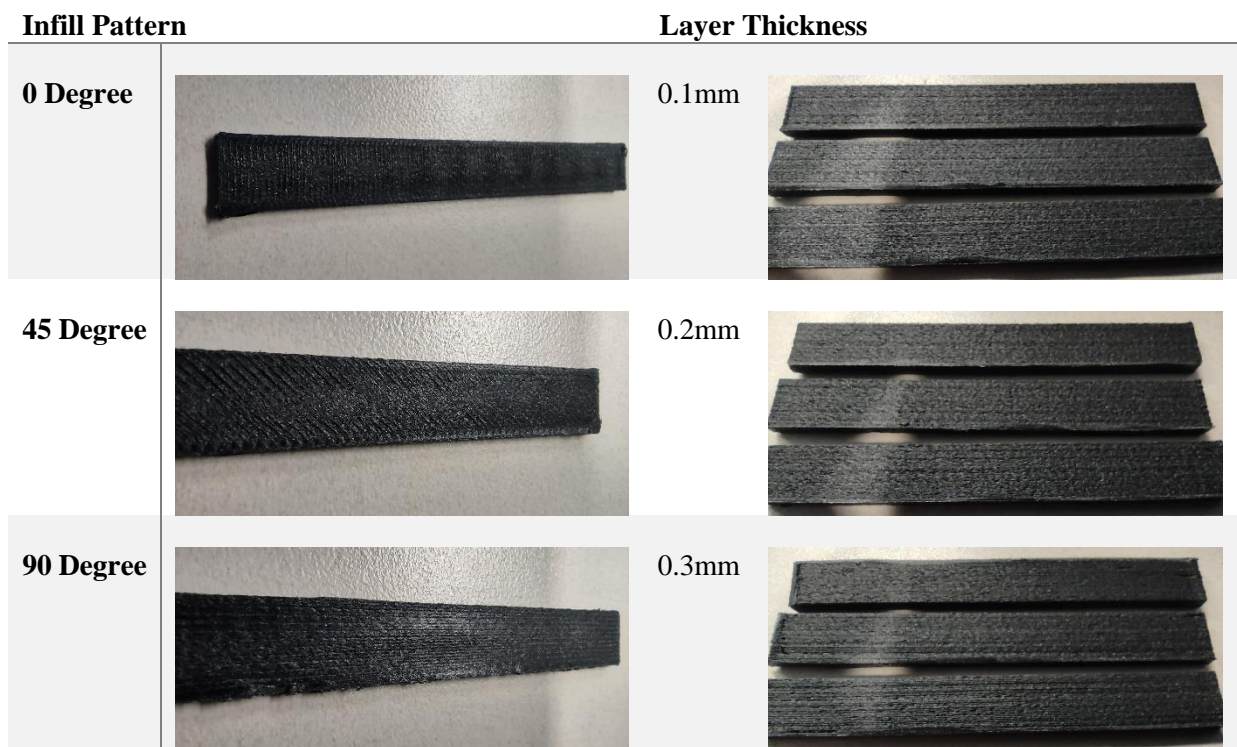


Figure 2.10: PA6CF20 Samples

2.2.2 Surface Modifications

The most important disadvantage of the FFF method is the poor surface properties. For this reason, post-processing operations are often required. Infill pattern and layer thickness affect the surface properties of samples produced with FFF. What different

carbon ratios and production parameters affect the surface properties was investigated in the thesis study. In addition, it is aimed to improve the surface properties with chemical processes. For this reason, formic acid, which can interact with polyamide with high chemical resistance, is used.

In order to examine the effect of all produced samples on the surface properties, all samples were kept in formic acid. The samples were taken into the bottle, sealed with parafilm M PM999 tape, and heated up to 68 °C. The samples were stirred in formic acid on a corning pc-420d stirring hot plate at 100 rpm for 30 minutes.

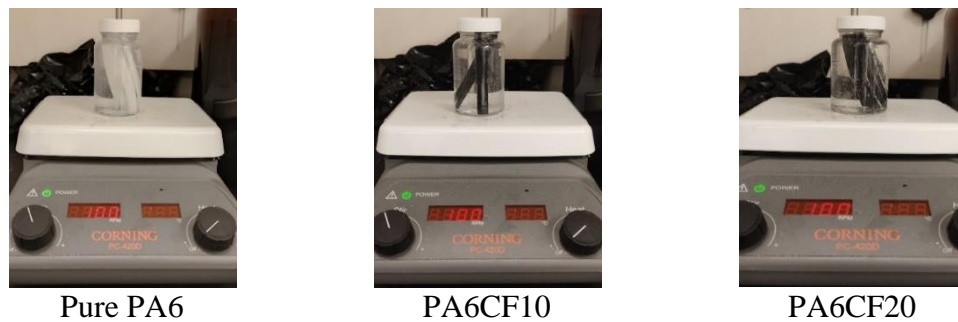


Figure 2.11: Surface Modifications in formic acid



Figure 2.12: Modified Pure PA6 (M Pure PA6)

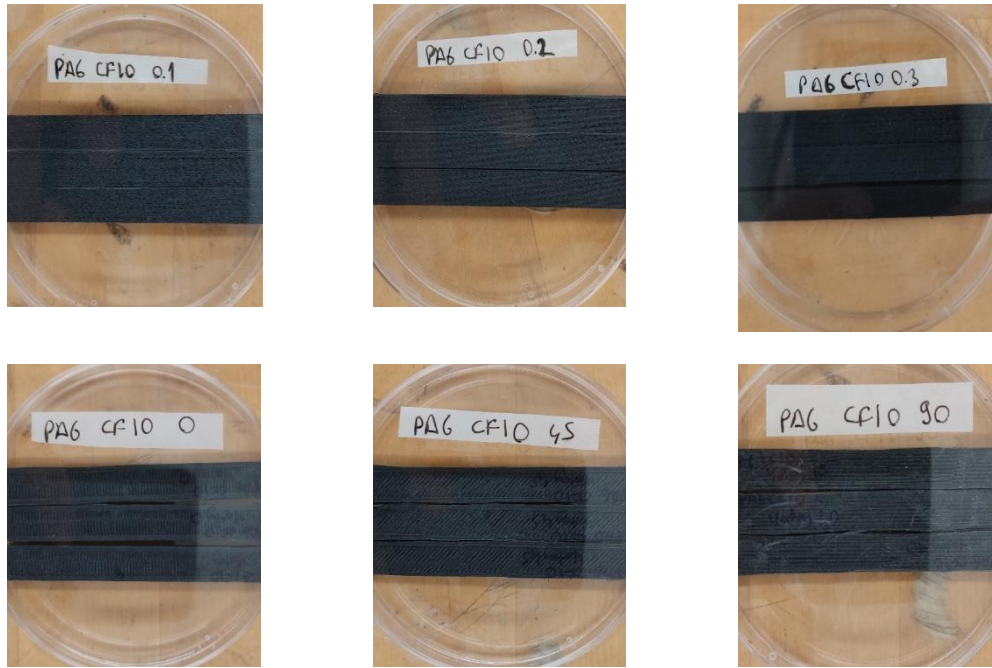


Figure 2.13: Modified PA6CF10 (M PA6CF10)



Figure 2.14: Modified PA6CF20 (M PA6CF20)

2.3 Characterization

2.3.1 Surface Roughness

Surface roughness measurements of all samples produced in the thesis study were carried out. The changes in the surface roughness of the samples produced in different layer thicknesses and infill patterns with additive manufacturing and the changes in the surface roughness after the modification applied to these samples were investigated.

Surface roughness measurements were made with the Mitutoyo brand SurfTest SJ-301 surface roughness measuring instrument located in Ege University Engineering Faculty Mechanical Engineering Department. Surface roughness measurements of the samples with 0, 45, and 90-degree infill patterns and 3 different layer thicknesses (0.1, 0.2, 0.3mm) and measurements of the surface modifications of all these samples were carried out.

Measurements were carried out according to the ISO 1997 standard. In each sample, 5 measurements were taken with 5 repetitions at 0.8 cut-offs.

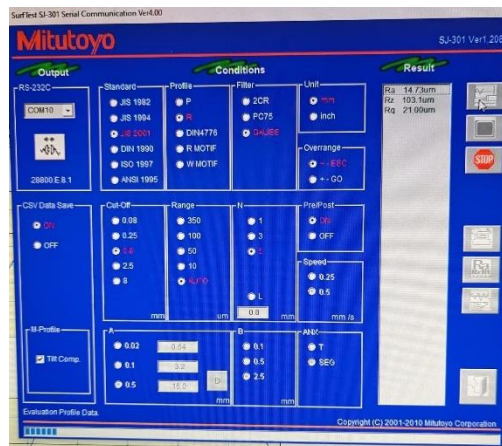


Figure 2.15: Surface Roughness Measurement Software

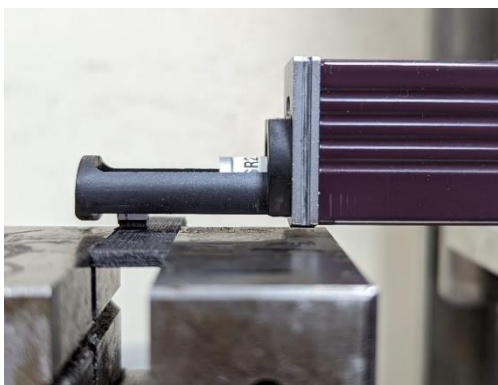


Figure 2.16: SJ-301 Surface Roughness Probe

2.3.2 Contact Angle Measurement

The wettability changes of the surfaces of the samples produced in the thesis study, which were produced with 45-degree infill patterns and 0.1, 0.2, and 0.3mm layer thicknesses, with and without surface modification, were investigated by 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 1 μ L 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 are reported as representative measurements.

2.3.3 Confocal Microscope

The surface properties of all samples were examined under an optical microscope. Modified and unmodified surfaces were imaged using the Olympus IX81 confocal microscope and cellSens software.

Chapter 3

3 Results and Discussions

Additive manufacturing is a technology used in the manufacture of prototypes and products, and this technology has several advantages, such as the production of complex geometries without waste of material and the need for expensive molding machines, finishing and machining tools. Additive manufacturing methods, which were used only to produce prototypes in the past, have paved the way to produce more durable commercial parts with the development of new materials today. Additive manufacturing methods provide an important alternative to produce parts with complex geometry, which cannot be produced in one piece with traditional manufacturing methods, and for sectors where piece-based production is made instead of mass production.

One of the additive manufacturing methods is the Fused filament extrusion (FFF) method. FFF is an additive manufacturing method produced by material extrusion. The FFF method is a cost-effective and easily accessible method compared to other additive manufacturing methods. In addition, it is possible to use it in different applications with a wider range of materials. It is an additive manufacturing method, also called three-dimensional printing, in which the computer-aided design (CAD) model is divided into slices with data in the appropriate format and produced layer by layer by the device using thermoplastic filament.

Apart from the variety of materials used in the production of parts with the FFF method, there are different parameters used for production. These parameters, which can be exemplified as nozzle temperature, layer thickness, nozzle diameter, bed temperature, printing speed, fill rate, filling pattern, affect the mechanical properties, characterization and geometry of the produced part. The parts produced by additive

manufacturing exhibit different properties depending on the direction. Changing the process parameters in parts produced for different applications with additive manufacturing methods has a significant effect on mechanical properties such as tensile strength, surface roughness, geometric tolerance, toughness and hardness. In addition, the surface quality of the parts produced by additive manufacturing is not good compared to the products produced by injection. This negative surface quality limits the application areas of additive manufacturing methods.

In this thesis, the changes in the directional surface properties of the composite structures produced using the fused filament extrusion (FFF) method and the improvement of the surface properties with chemical treatments were carried out. Experimental research on the parts produced by the FFF method using polyamide matrix carbon fiber reinforced material was carried out by changing the layer thickness and infill pattern parameters. Chemical finishing applications were made to improve the surface roughness of the parts produced by the FFF method. In this scope, the optimization of the FFF process parameters and the effect of the chemical treatments applied to the post-production part were examined.

3.1 Surface Roughness

Before smoothing pictures of flat and curved surfaces, the rasters are clearly visible. In the case of flat surfaces, they are in a straight direction whereas curved surfaces are in oblique direction. Inside the raster, for both surfaces, some marks or globules are spotted. The flat surface shows both big and small spots and the curved surface shows only small spots. These spots indicate comparatively more rough areas.

The results of roughness values R_a , R_q , and R_z against the samples with varied layer thicknesses are tabulated below. Analysis of the results: Based on the surface finish results, as shown above, it is evident that as the layer thickness is increasing, surface roughness values (R_a , R_q & R_z) are increasing. The layer thickness is the only parameter considered for the surface roughness study, while other parameters were considered for the built time study. The relationship between the layer thickness and roughness values is shown below, which is showing a linear regression.

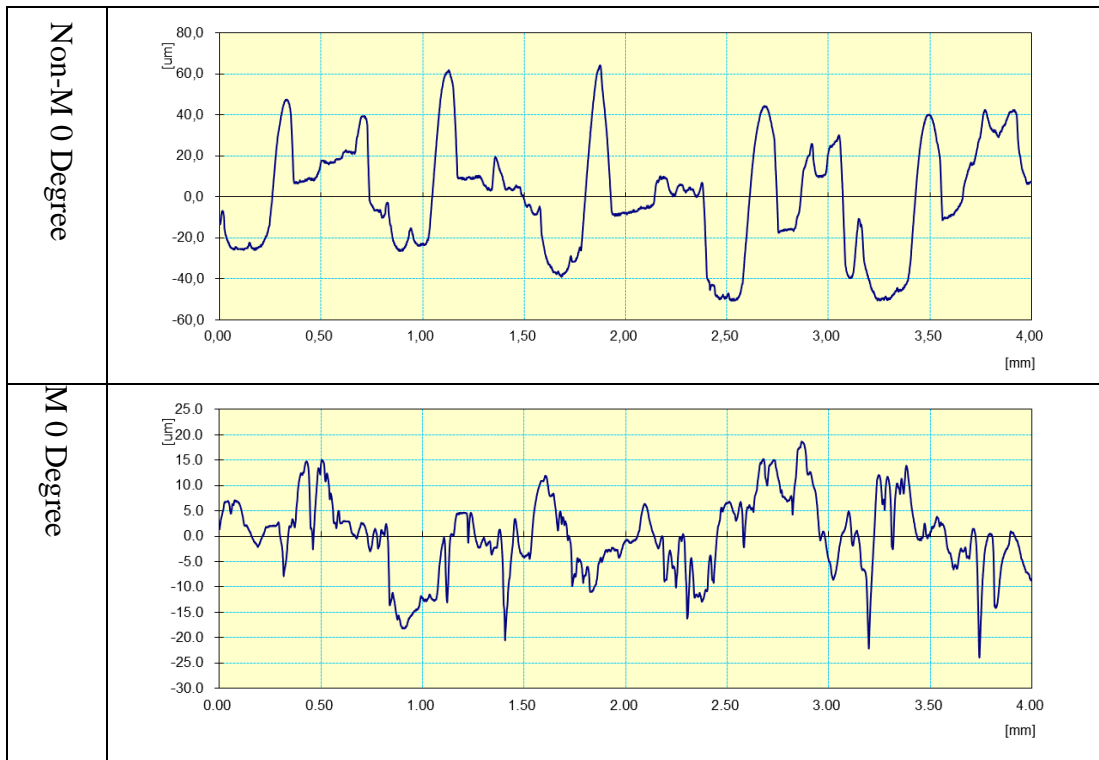


Figure 3.1: Pure PA6 Surface Roughness (0 Degree)

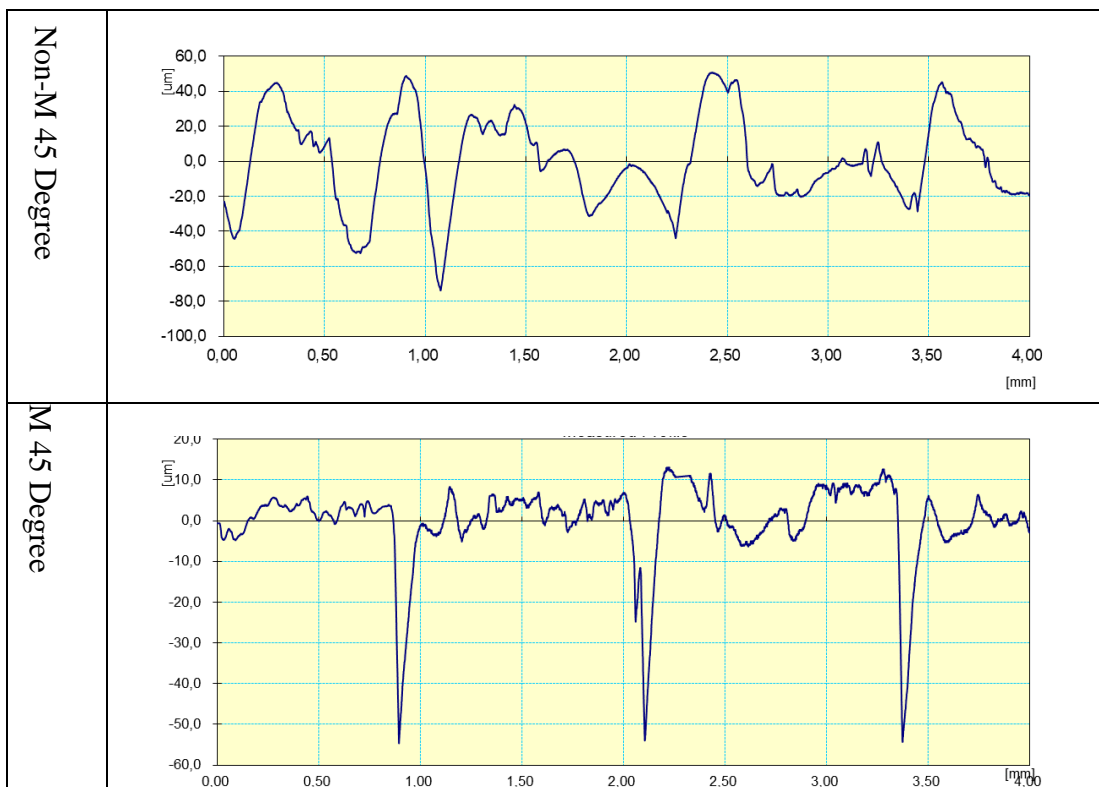


Figure 3.2: Pure PA6 Surface Roughness (45 Degree)

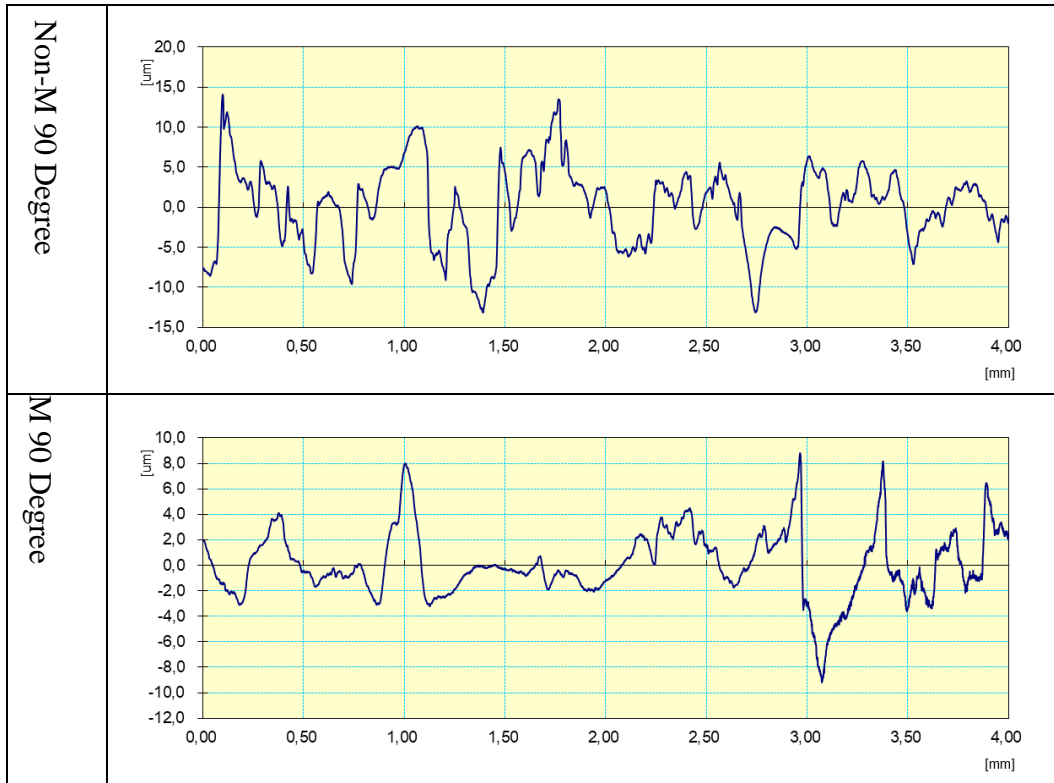


Figure 3.3: Pure PA6 Surface Roughness (90 Degree)

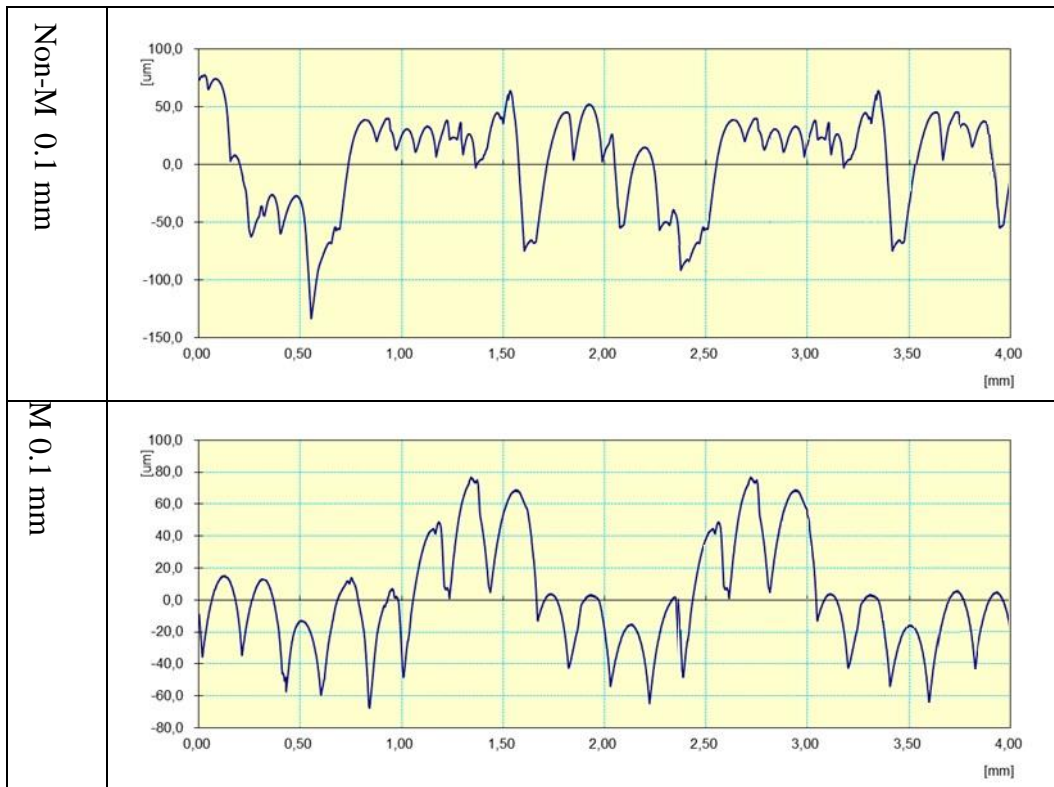


Figure 3.4: Pure PA6 Surface Roughness (0.1mm)

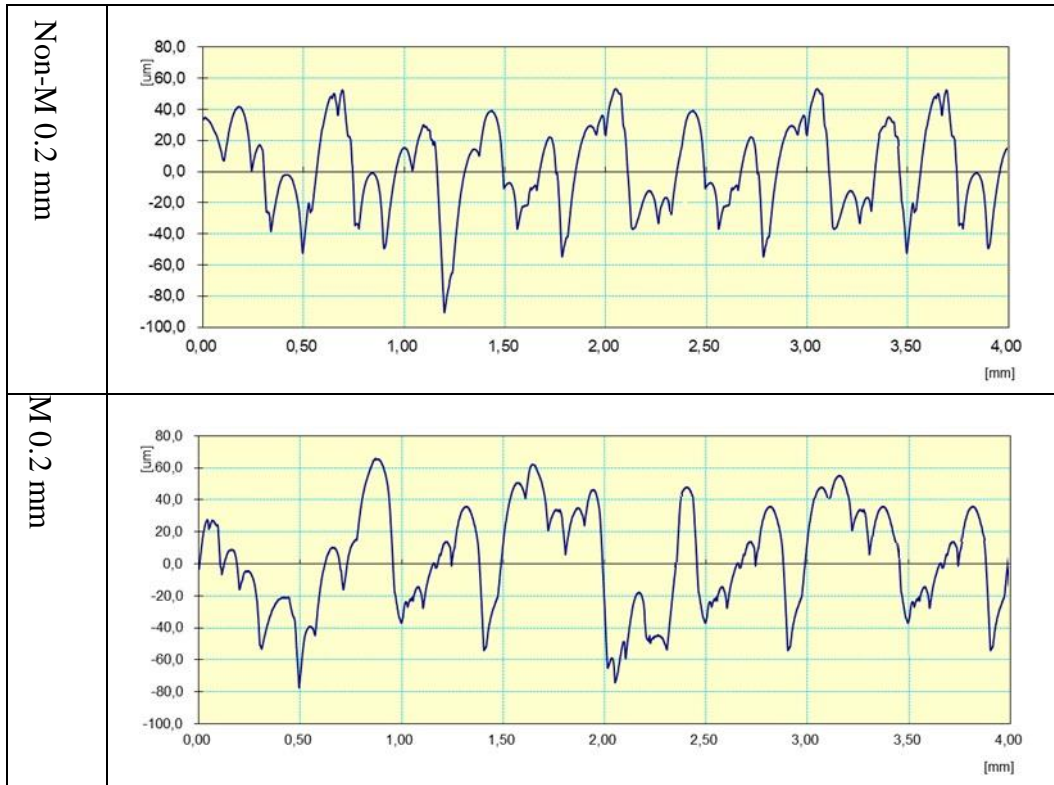


Figure 3.5: Pure PA6 Surface Roughness (0.2mm)

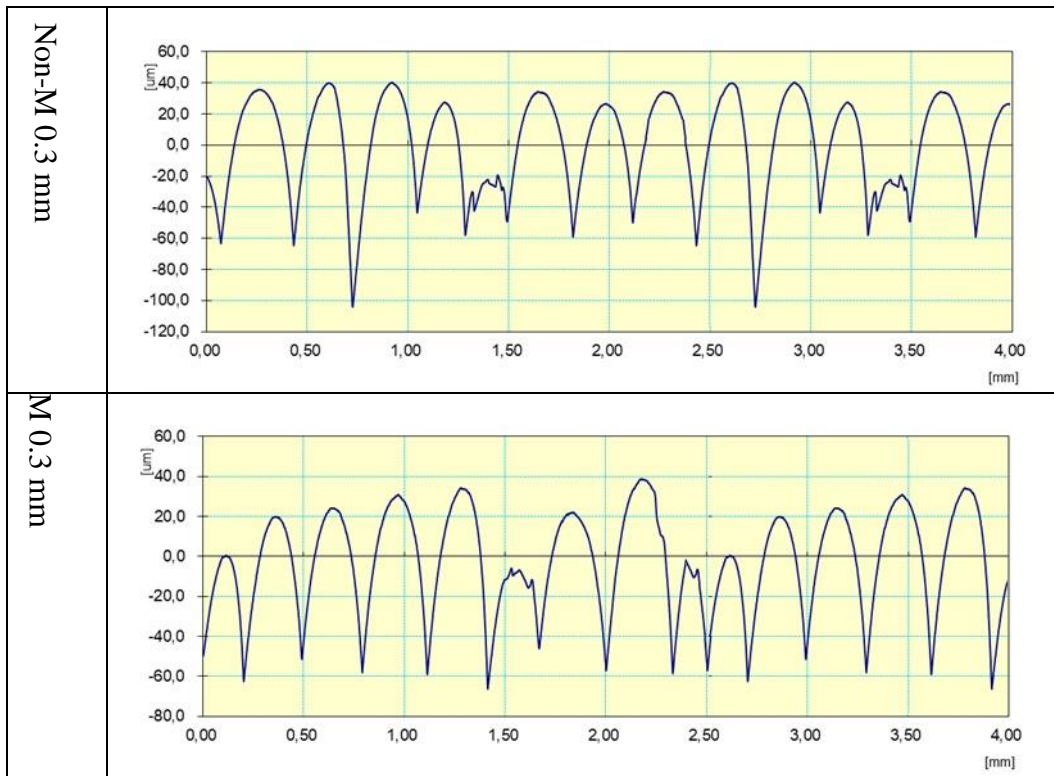


Figure 3.6: Pure PA6 Surface Roughness (0.3mm)

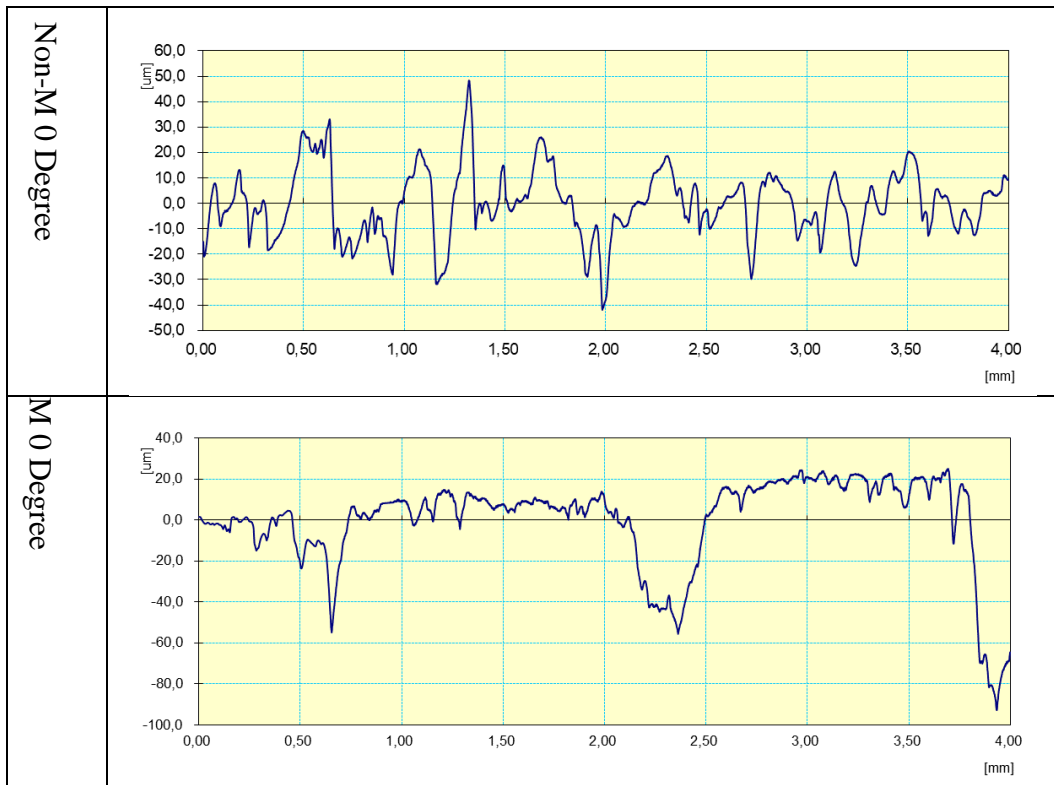


Figure 3.7: PA6CF10 Surface Roughness (0 Degree)

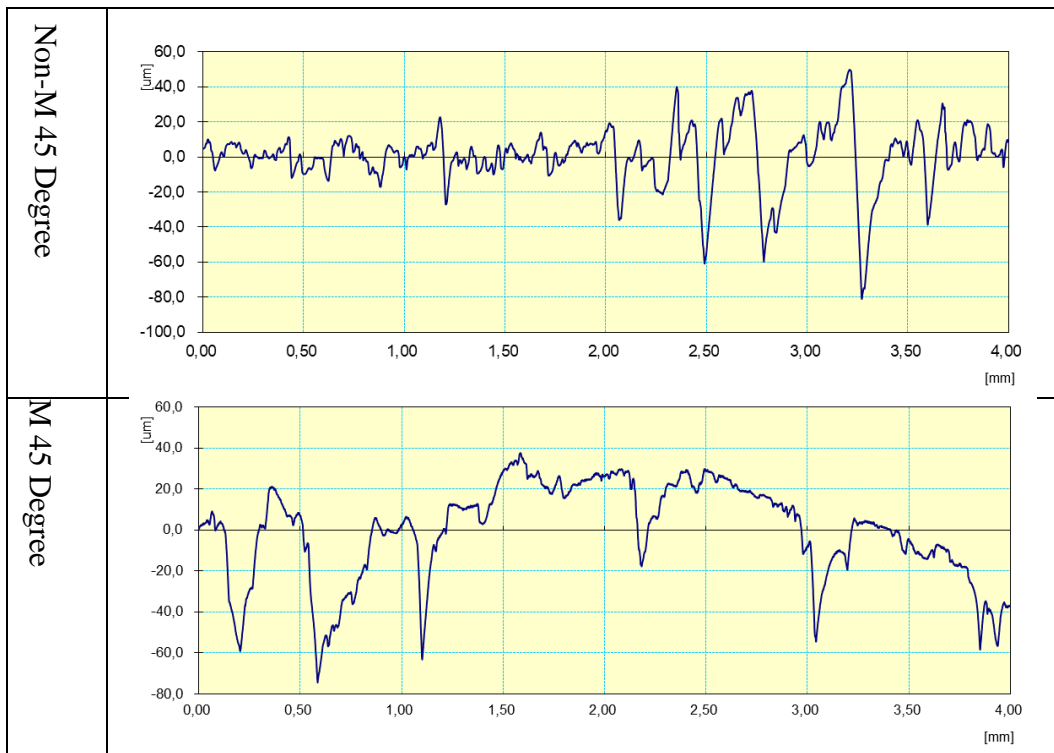


Figure 3.8: PA6CF10 Surface Roughness (45 Degree)

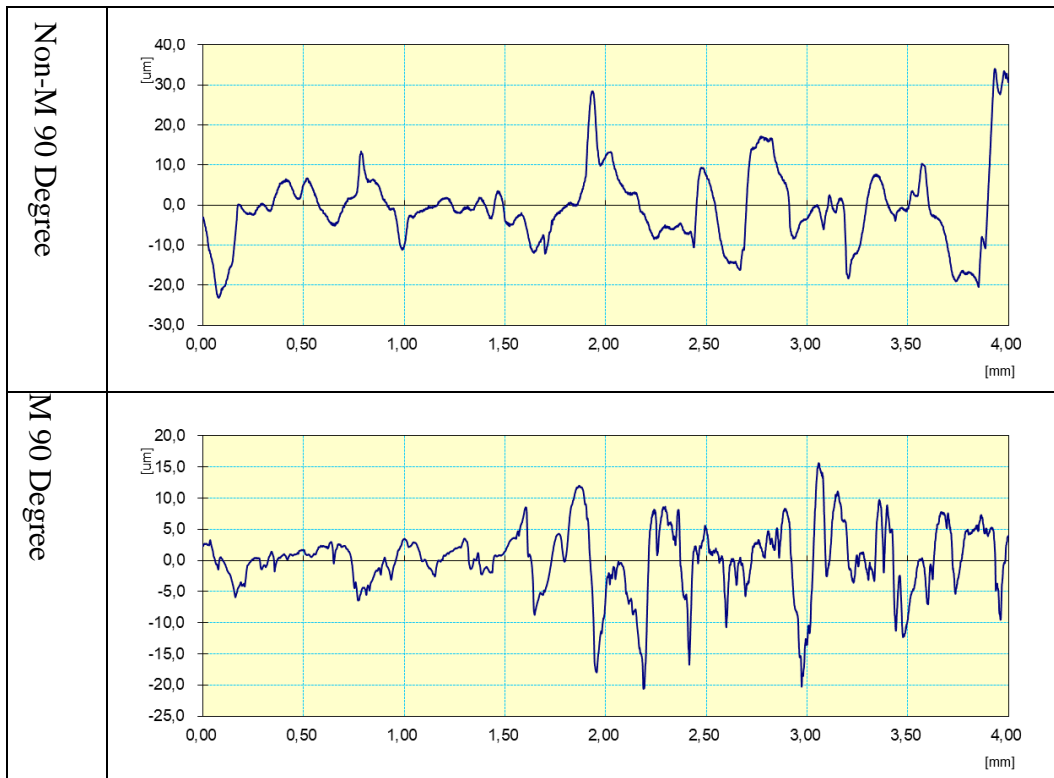


Figure 3.9: PA6CF10 Surface Roughness (90 Degree)

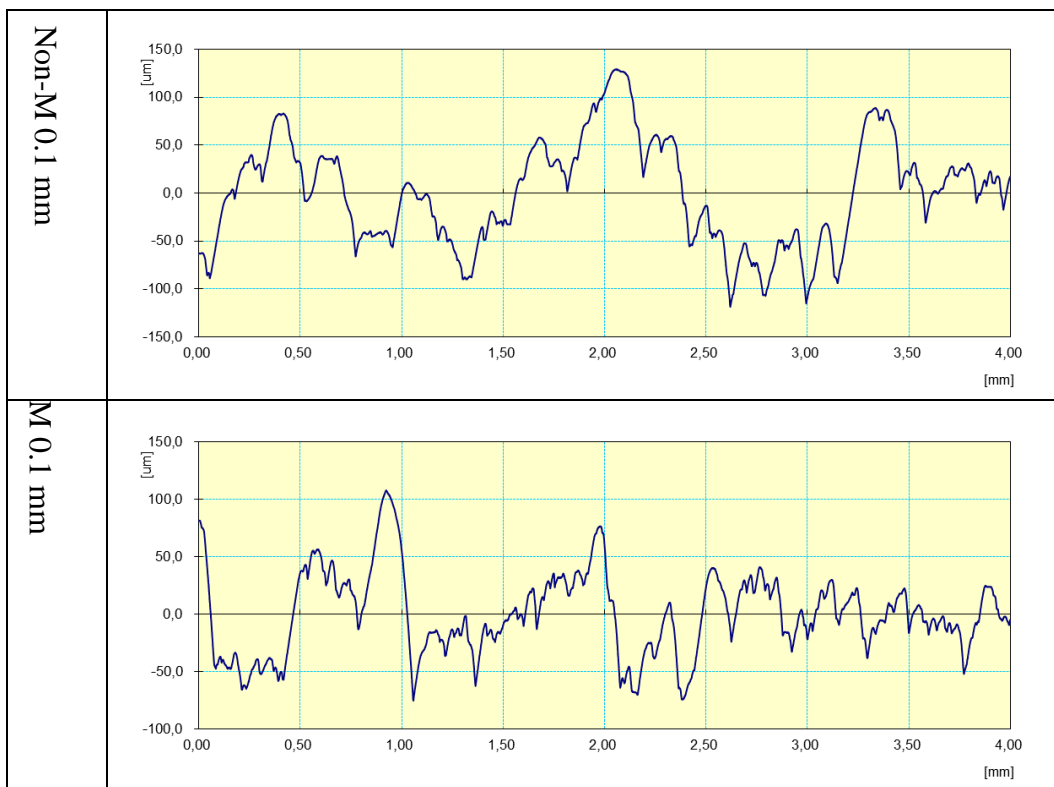


Figure 3.10: PA6CF10 Surface Roughness (0.1mm)

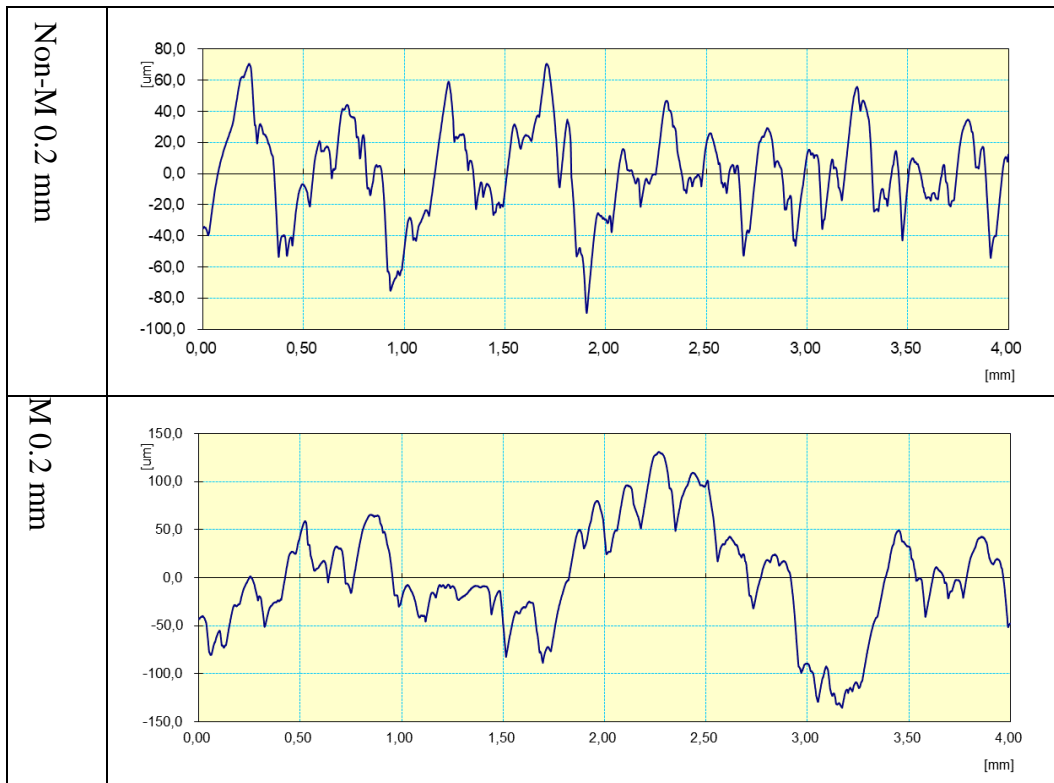


Figure 3.11: PA6CF10 Surface Roughness (0.2mm)

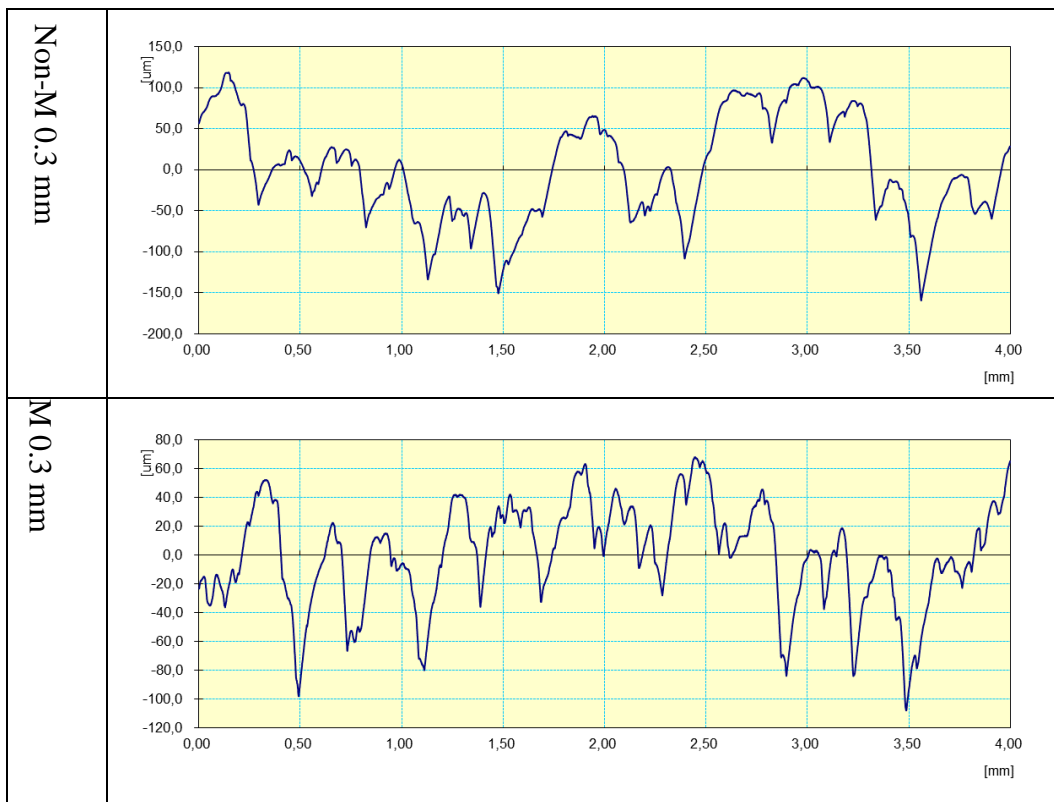


Figure 3.12: PA6CF10 Surface Roughness (0.3mm)

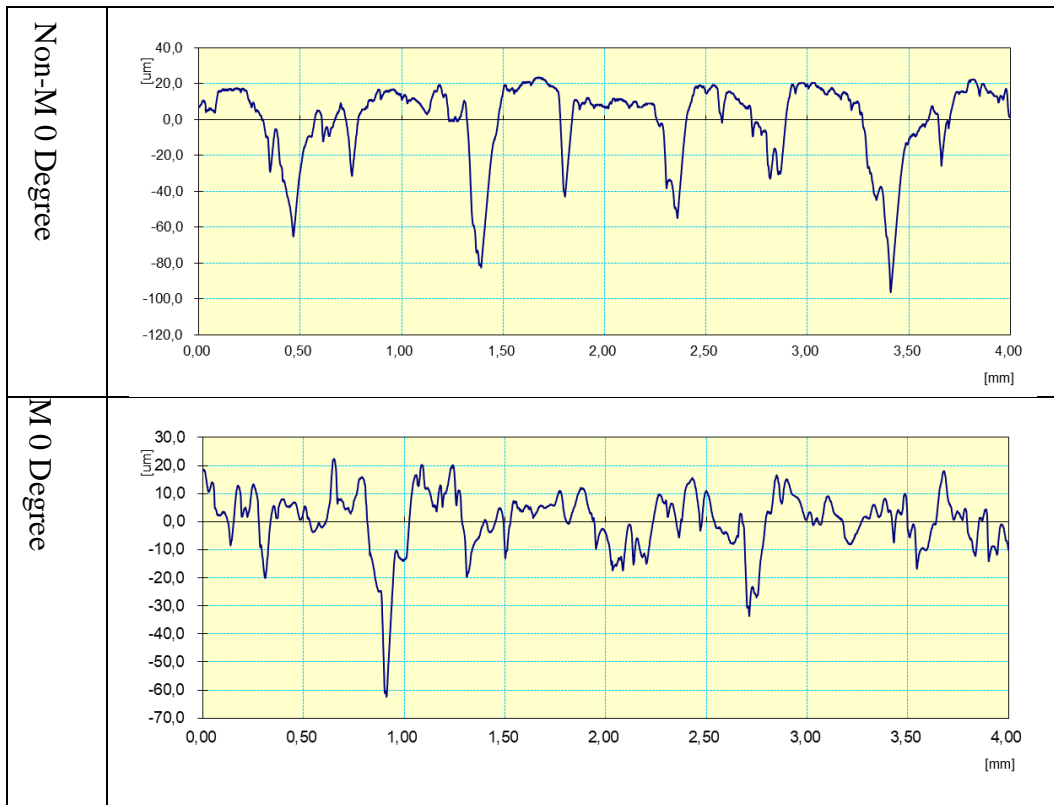


Figure 3.13: PA6CF20 Surface Roughness (0 Degree)

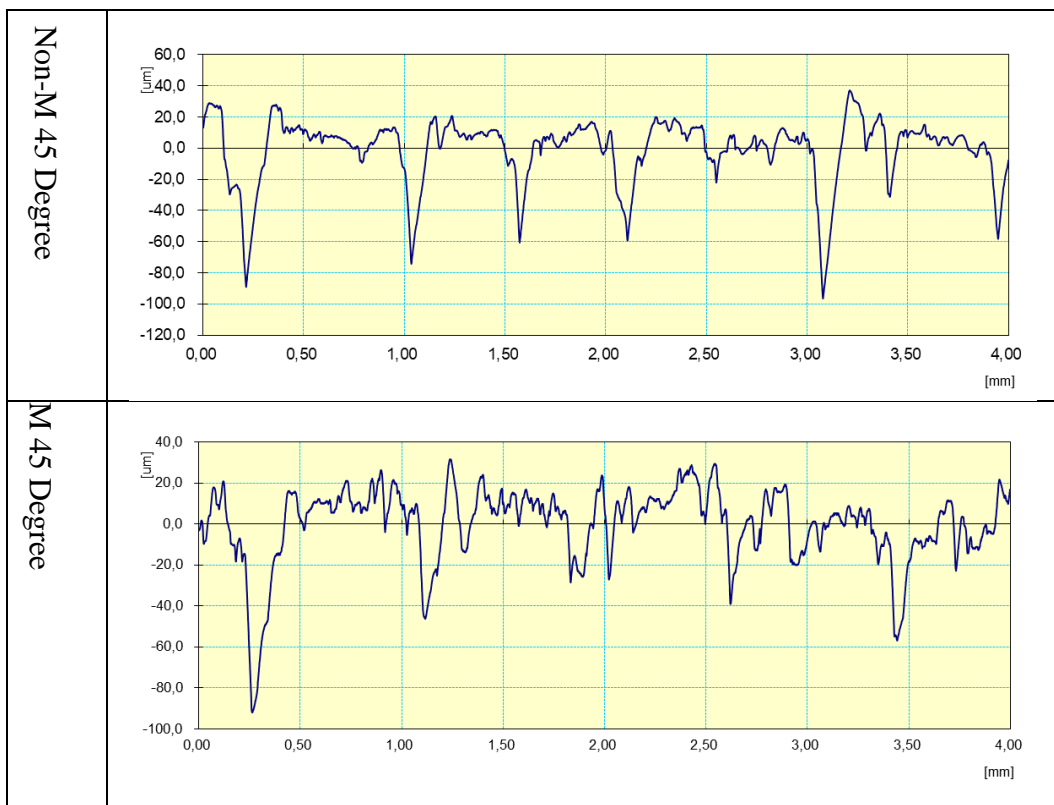


Figure 3.14: PA6CF20 Surface Roughness (45 Degree)

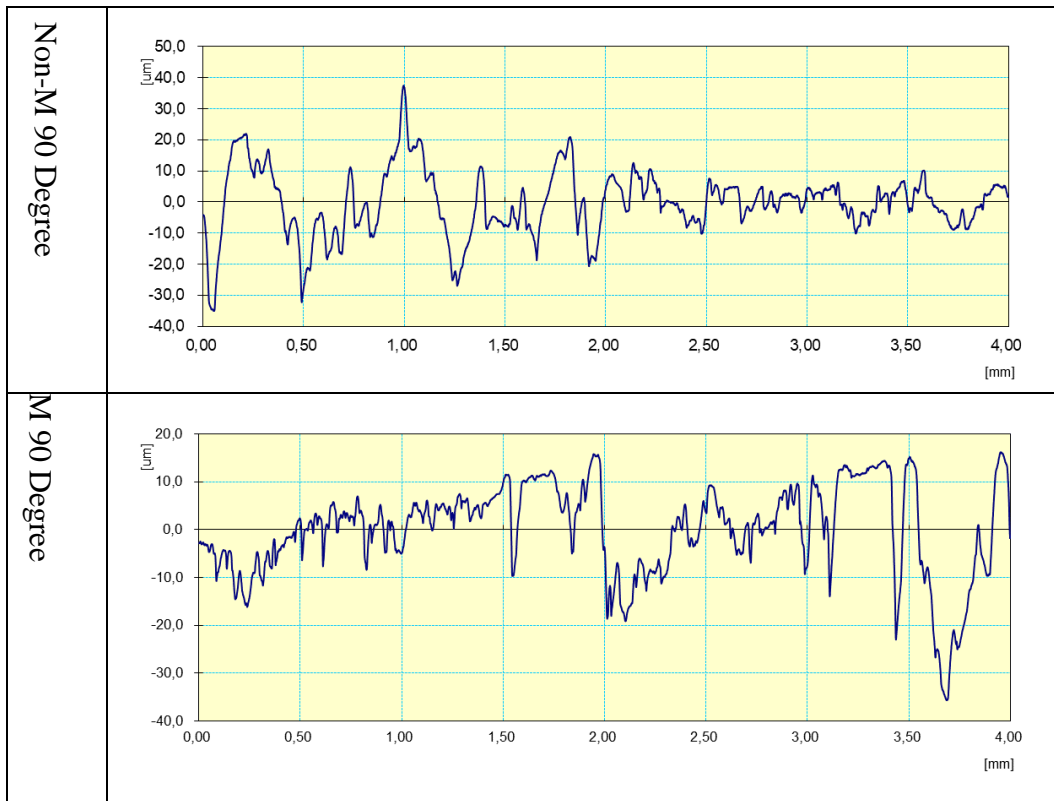


Figure 3.15: PA6CF20 Surface Roughness (90 Degree)

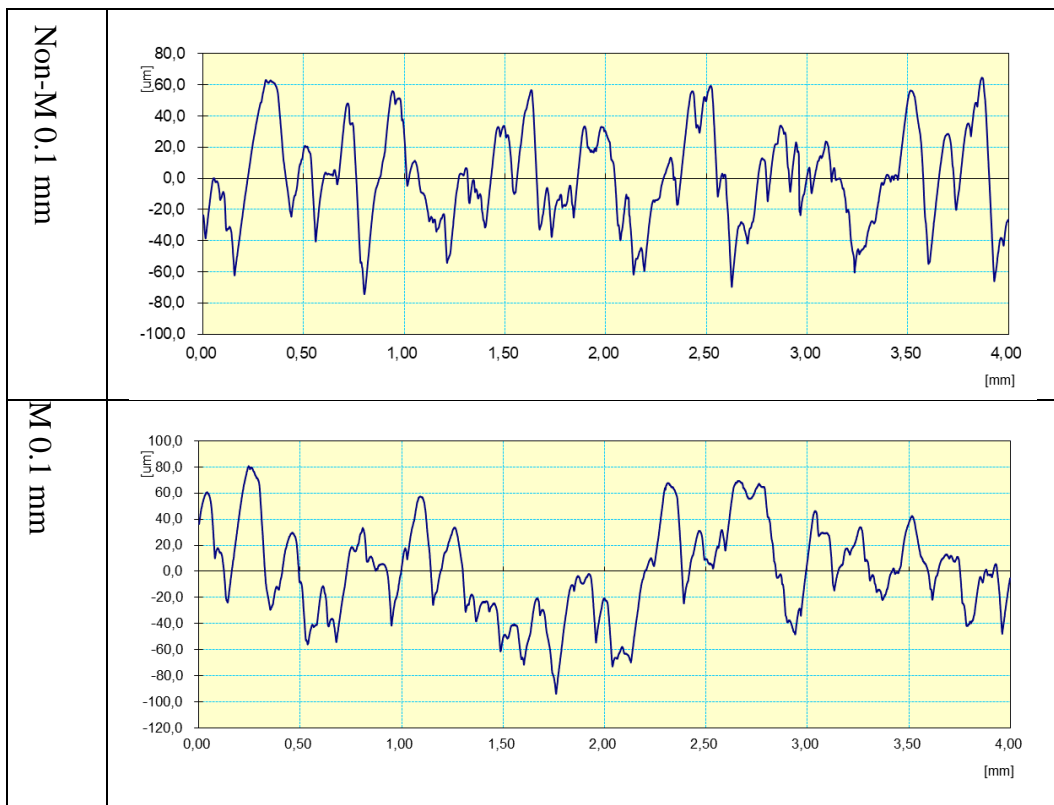


Figure 3.16: PA6CF20 Surface Roughness (0.1mm)

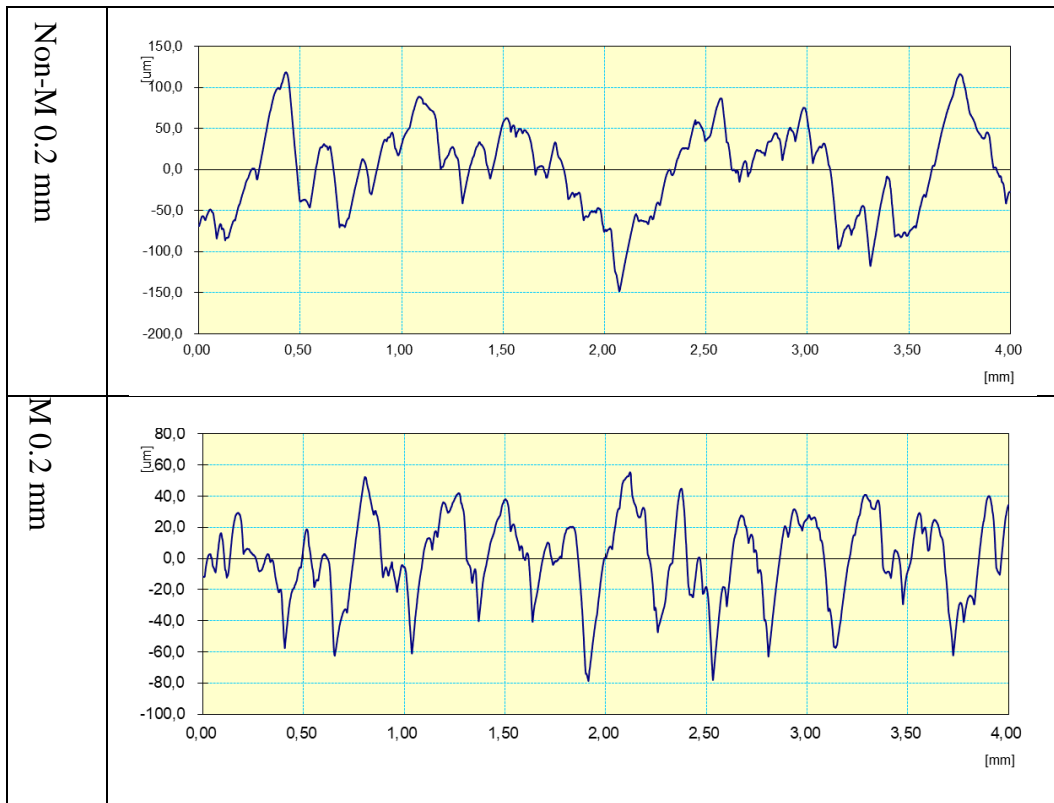


Figure 3.17: PA6CF20 Surface Roughness (0.2mm)

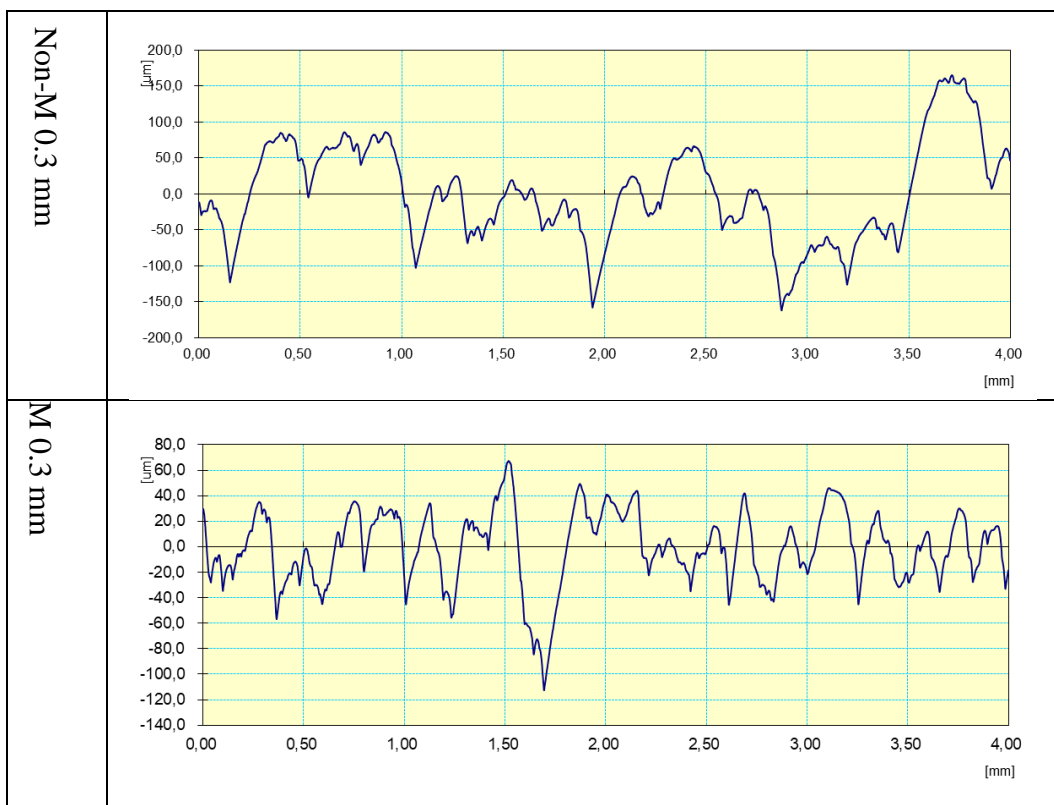


Figure 3.18: PA6CF20 Surface Roughness (0.3mm)

Table 3.1: Surface Roughness of X-Y Axis

		M 0 Deg	Non-M 0 Deg	M 45 Deg	Non-M 45 Deg	M 90 Deg	Non-M 90 Deg
Pure Pa6	Ra	4.21um	16.54um	4.83um	20.96um	1.96um	4.00um
	Rz	28.51um	74.16um	42.03um	91.53um	10.97um	19.81um
Pa6cf10	Ra	7.54um	10.22um	9.19um	11.54um	3.90um	6.66um
	Rz	47.96um	58.11um	60.09um	78.03um	22.87um	37.24um
Pa6cf20	Ra	7.84um	11.75um	10.15um	14.73um	4.73um	7.80um
	Rz	48.02um	73.40um	71.31um	103.1um	28.34um	40.23um

Table 3.2: Surface Roughness of Z Axis

		M 0.1mm	Non-M 0.1mm	M 0.2mm	Non-M 0.2mm	M 0.3mm	Non-M 0.3mm
Pure Pa6	Ra	16.26um	21.49um	19.68um	23.91um	21.21um	25.57um
	Rz	75.73um	122.4um	98.60um	114.4um	94.96um	111.9um
Pa6cf10	Ra	19.59um	21.09um	20.68um	23.00um	21.14um	26.24um
	Rz	110.8um	100.2um	98.21um	122.1um	111.1um	137.1um
Pa6cf20	Ra	20.20um	24.47um	21.34um	25.55um	22.58um	28.13um
	Rz	96.49um	129.4um	114.8um	135.2um	109.5um	145.2um

Surface roughness measurements of pure PA6, 10 wt% carbon fiber reinforced, and 20 wt% carbon fiber reinforced samples were performed. Surface modification with formic acid was applied to the same samples and the changes in surface roughness after chemical treatment were investigated.

All samples were produced in 0.45 and 90 infill patterns in the X-Y axis and in layer thicknesses of 0.1mm, 0.2mm and 0.3mm in the Z axis.

The changes in the surface roughness depending on the infill pattern were investigated in the samples produced in different infill patterns on the X-Y axis. In addition, the effects of different carbon fiber additive ratios on the surface roughness were observed.

The lowest surface roughness in all samples was observed in samples produced with 90° infill pattern. Since the 90° infill pattern orientation and the movement pattern of the surface roughness measurement probe are parallel to each other, the lowest values were obtained in these parameters. The increase in carbon fiber ratio increased the surface roughness in all samples with different infill patterns. The rigid structure of carbon fiber and the increase in its ratio in the matrix caused the formation of rougher surfaces. Microscope images also support this.

The highest surface roughness values were observed in the samples produced with 45° infill pattern. The production of layers in the $\pm 45^\circ$ orientation resulted in the formation of rougher surfaces at the moving points that intersect in the coordinate plane. In pure PA6 unmodified samples, when the samples produced with 90° infill pattern were compared with the samples produced with 45° infill pattern, the Ra value increased 5 times, when the samples produced with 0° infill pattern were compared, the Ra value increased approximately 4 times.

A greater improvement in surface quality was observed in pure 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 pure PA6. Although the surface roughness, which increased with the increase in fiber content, was reduced as a result of chemical modification, higher surface roughness was observed in the samples with high carbon fiber content. 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.

The changes in the surface roughness depending on the layer thickness were investigated in the samples produced with different layer thicknesses in the Z axis. In addition, the effects of different carbon fiber additive ratios on the surface roughness in the Z axis were investigated.

Since all produced samples are produced in a frame, the infill pattern is independent of the infill pattern on the surfaces in the Z axis. The projections of the layer thicknesses on the Z axis have the same pattern.

An increase in surface roughness occurred with the increase in layer thickness in all pure PA6 samples with and without chemical treatment. After chemical surface treatment, improvement in surface quality is observed in all layer thicknesses. Carbon fiber additive and additive ratio did not change the surface roughness in the Z axis. Extruding the material with a nozzle from an area of 0.6mm in diameter in the X-Y axis, but with much lower layer thicknesses prevented the fibers from forming a rough surface. As the layer thickness increased, the effect of the fiber additive ratio on the surface roughness also increased.

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 axis 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 axis were not obtained. This is due to the fact that the nozzle contributes to the smoothing of the surface while moving in the X-Y axis in sample production, while there is no support or corrective mechanism in the Z axis.

Even if chemical surface treatment was applied on all produced samples, the surface quality X-Y axis 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 axis.

3.2 Contact Angle Measurement

Many surface modifications and coating technologies are applied to optimize wetting and adhesion properties. Surface energy is affected by both surface chemistry and roughness.

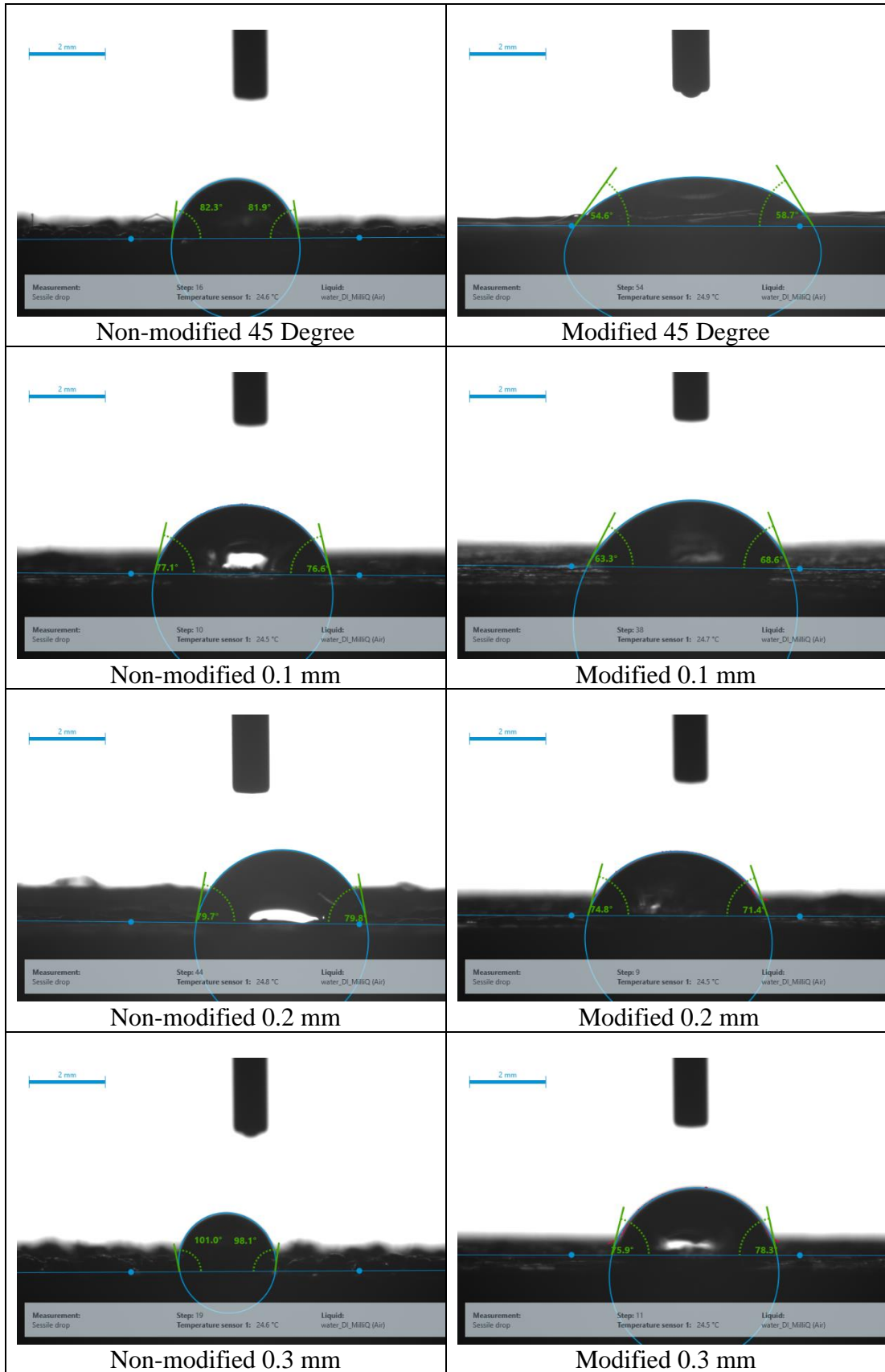


Figure 3.19: Pure PA6 Contact Angles

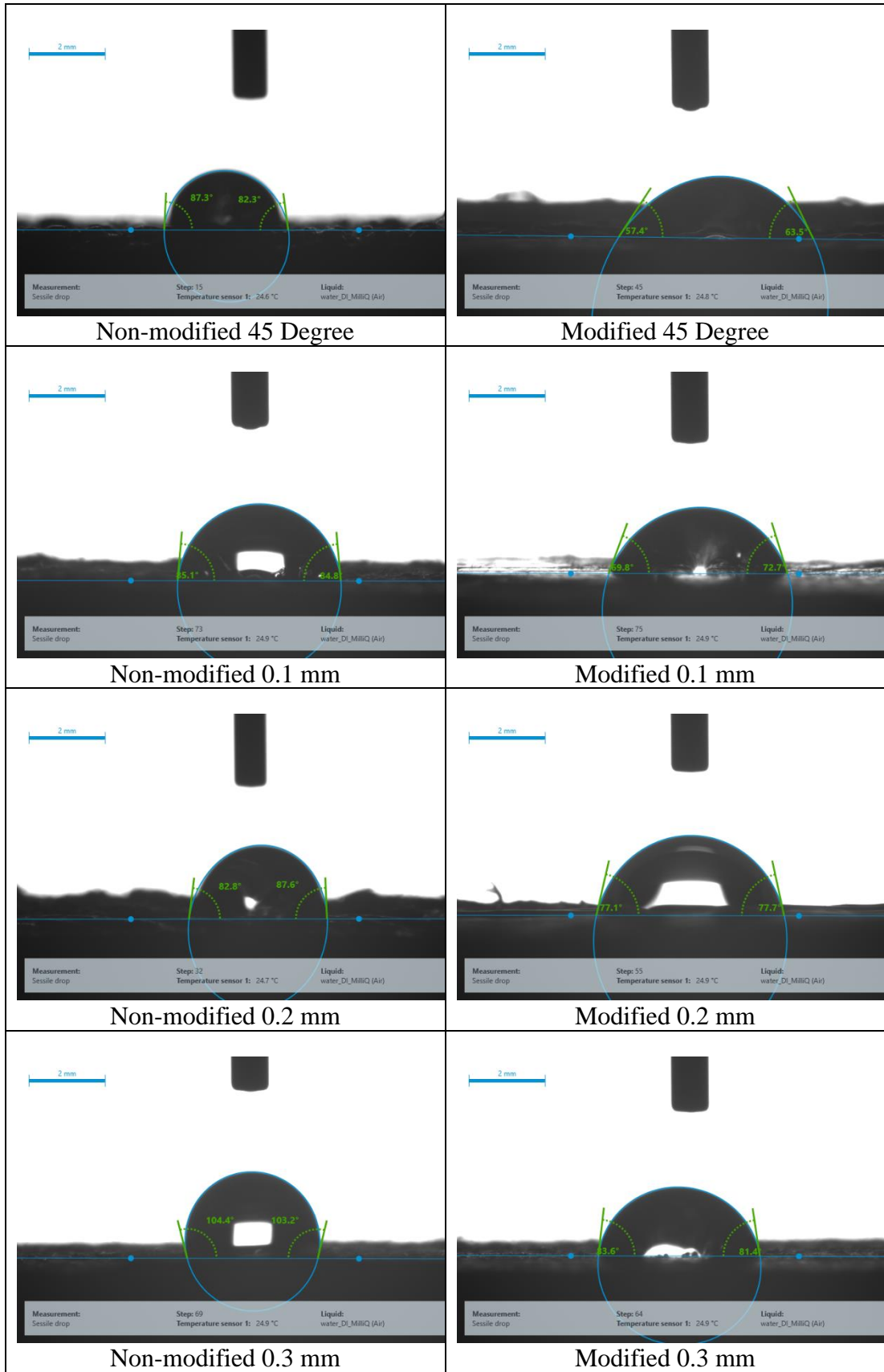


Figure 3.20: PA6CF10 Contact Angles

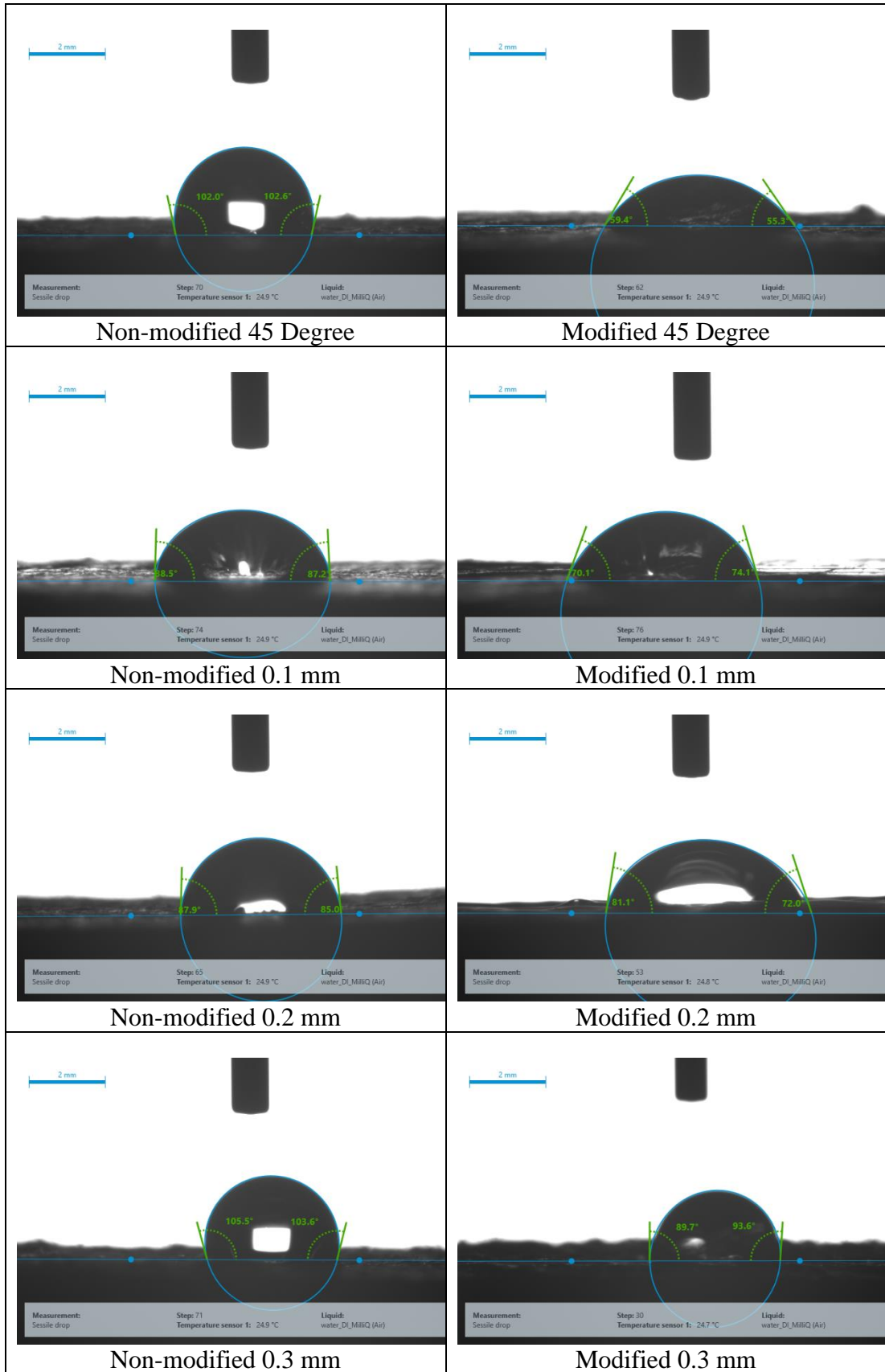


Figure 3.21: PA6CF20 Contact Angles

Table 3.3: Contact Angles

	45	0,1	0,2	0,3
PUREPA6 M	54,6 - 58,7	63,3 - 68,6	74,8 - 71,4	75,9 - 78,3
PUREPA6 NON-M	82,3 - 81,9	77,1 - 76,6	79,7 - 79,8	101 - 98,1
PA6CF10 M	57,4 - 63,5	69,8 - 72,7	77,1 - 77,7	83,6 - 81,4
PA6CF10 NON-M	87,3 - 82,3	85,1 - 84,8	82,8 - 87,6	104,4 - 103,2
PA6CF20 M	59,4 - 55,3	70,1 - 74,1	81,1 - 72	89,7 - 93,6
PA6CF20 NON-M	102 - 102,6	88,5 - 87,2	87,9 - 85	105,5 - 103,6

The contact angle values of the samples produced with 0° and 90° infill patterns on the X-Y axis 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 axis were examined.

The lowest contact angle value was found on the X-Y axis 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 Pure PA6 samples were lower than the samples containing carbon fiber. This is because polyamide is polar and hydrophilic (-CONH) due to the amide (-CONH) functional group it contains (72). Carbon fiber reinforcement caused an increase in contact angle values. The increase in surface roughness 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 cause 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 (73).

3.3 Confocal Microscope

In this section, confocal microscopy results will be presented. Images were taken to measure the contact layer's height and extrusion width. Also, structural differences were explained between surface modifications.

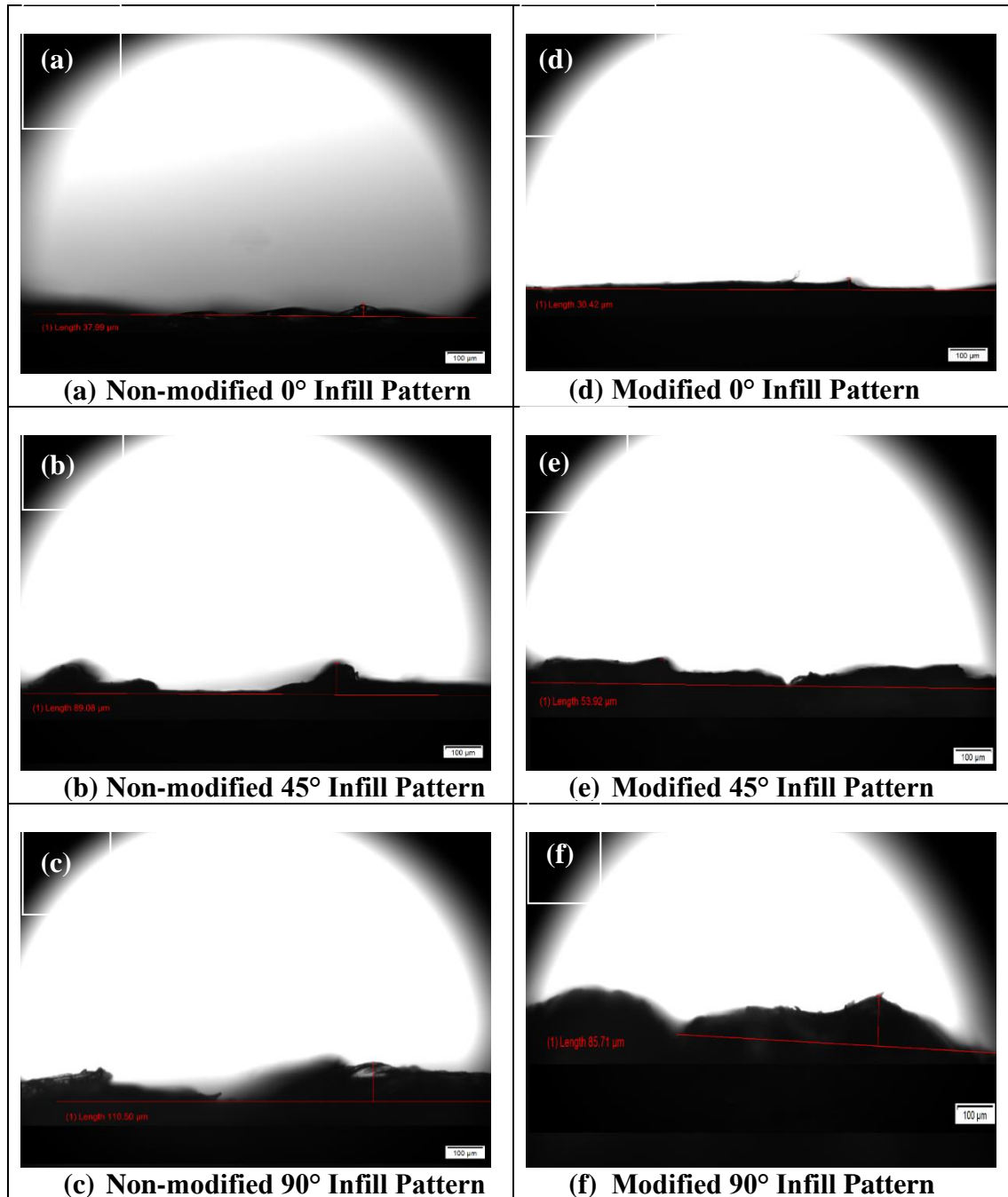


Figure 3.22: Pure PA6 X-Y Axis Confocal Images

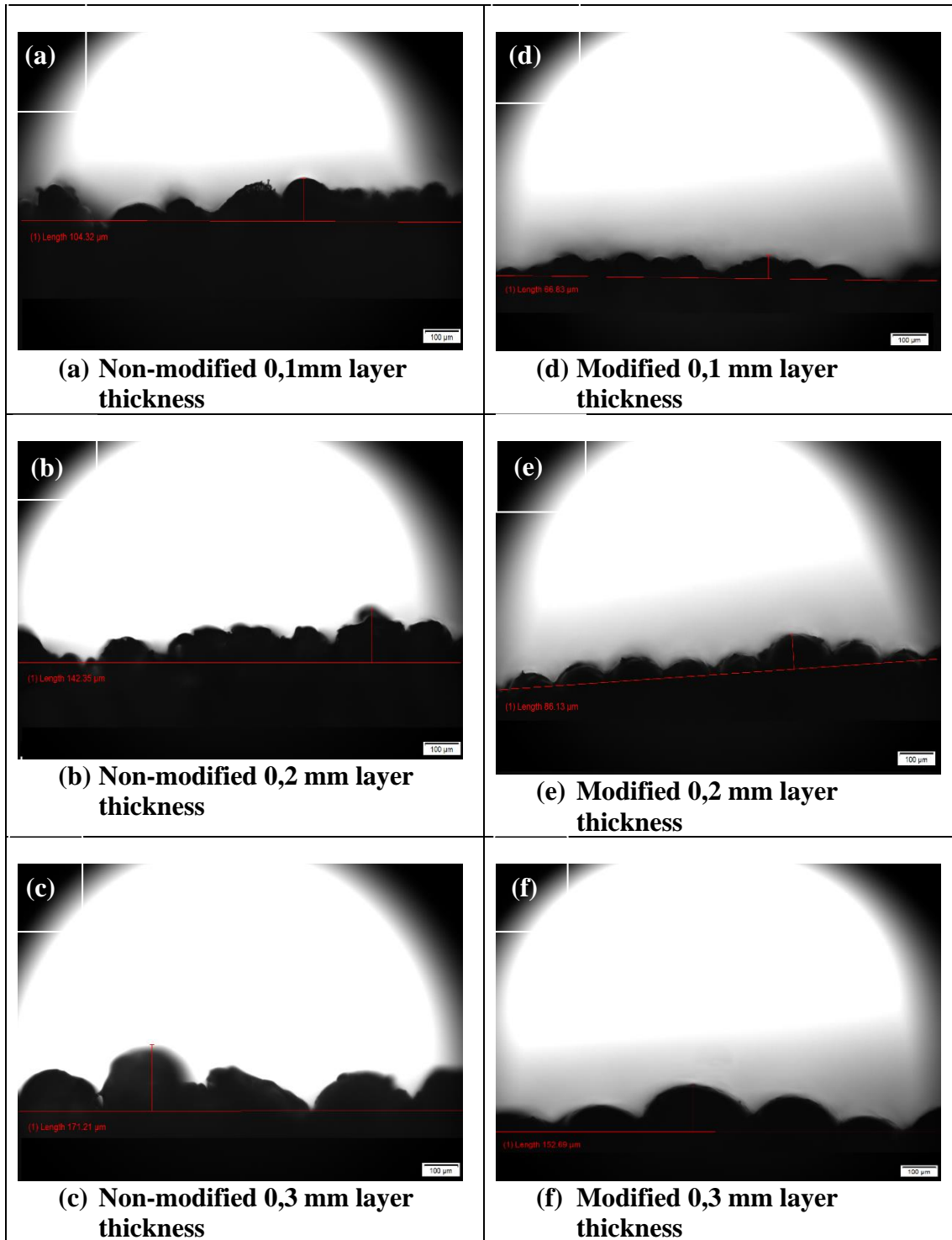


Figure 3.23: Pure PA6 Z Axis Confocal Images

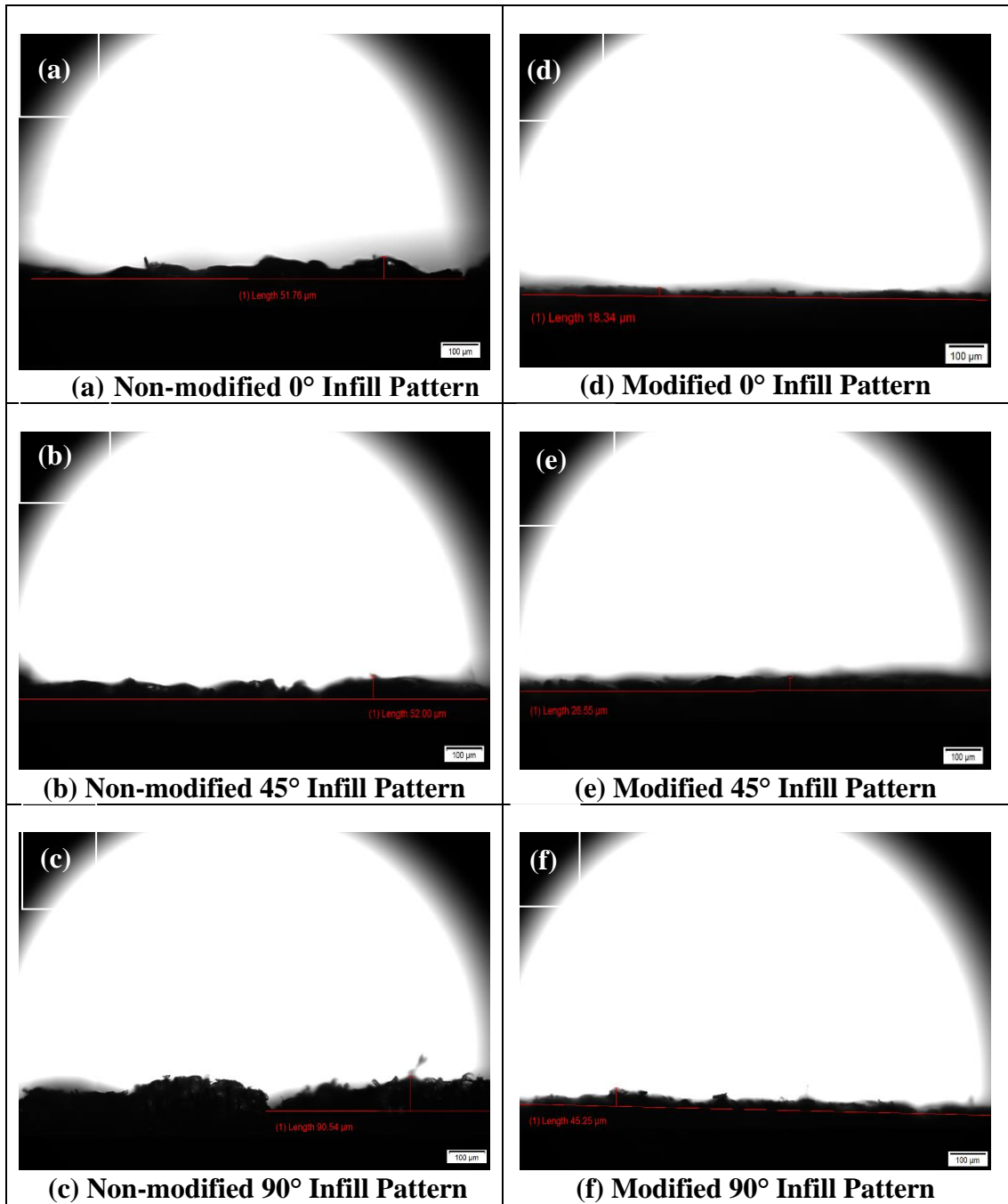


Figure 3.24: PA6CF10 X-Y Axis Confocal Images

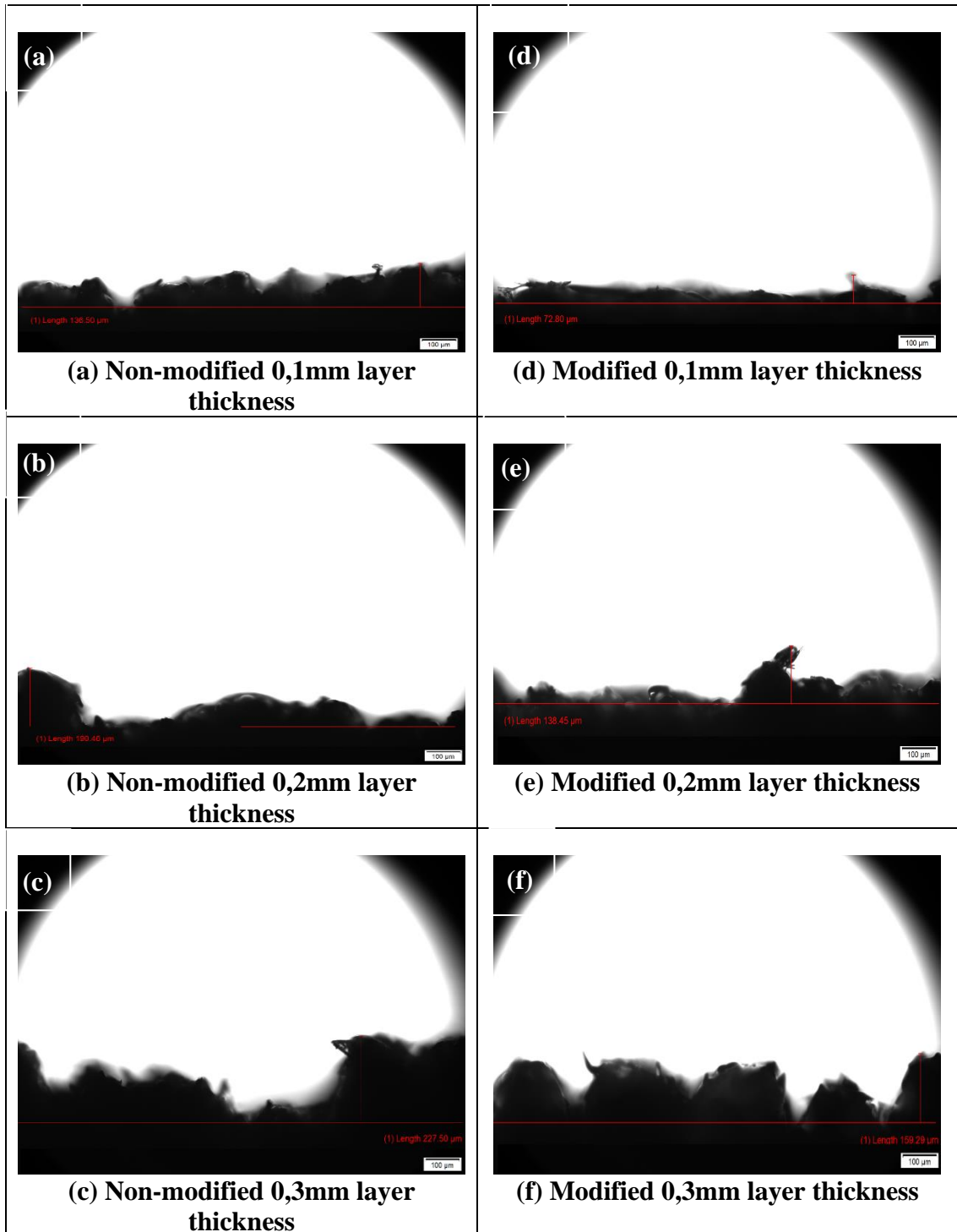


Figure 3.25: PA6CF10 Z Axis Confocal Images

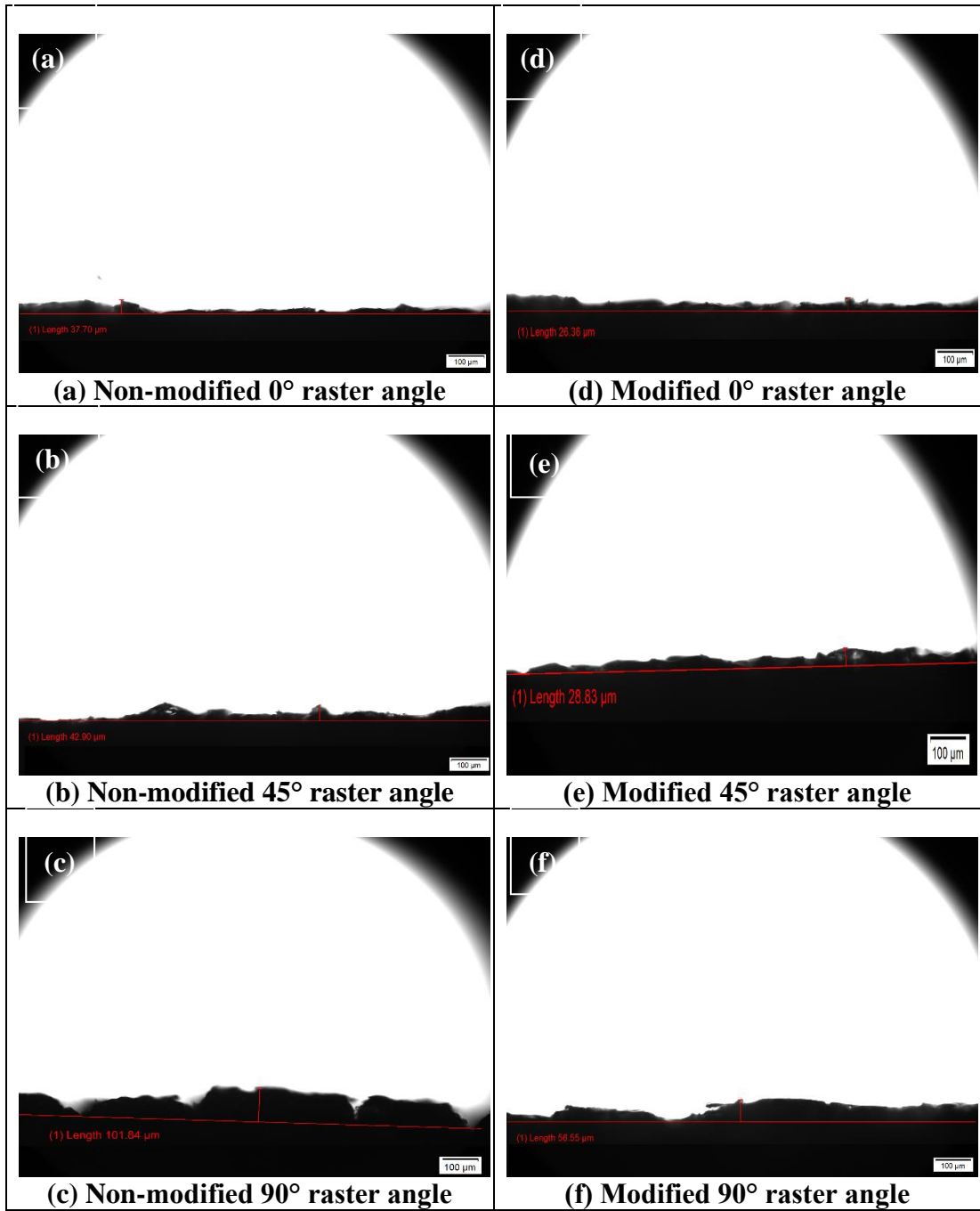


Figure 3.26: PA6CF20 X-Y Axis Confocal Images

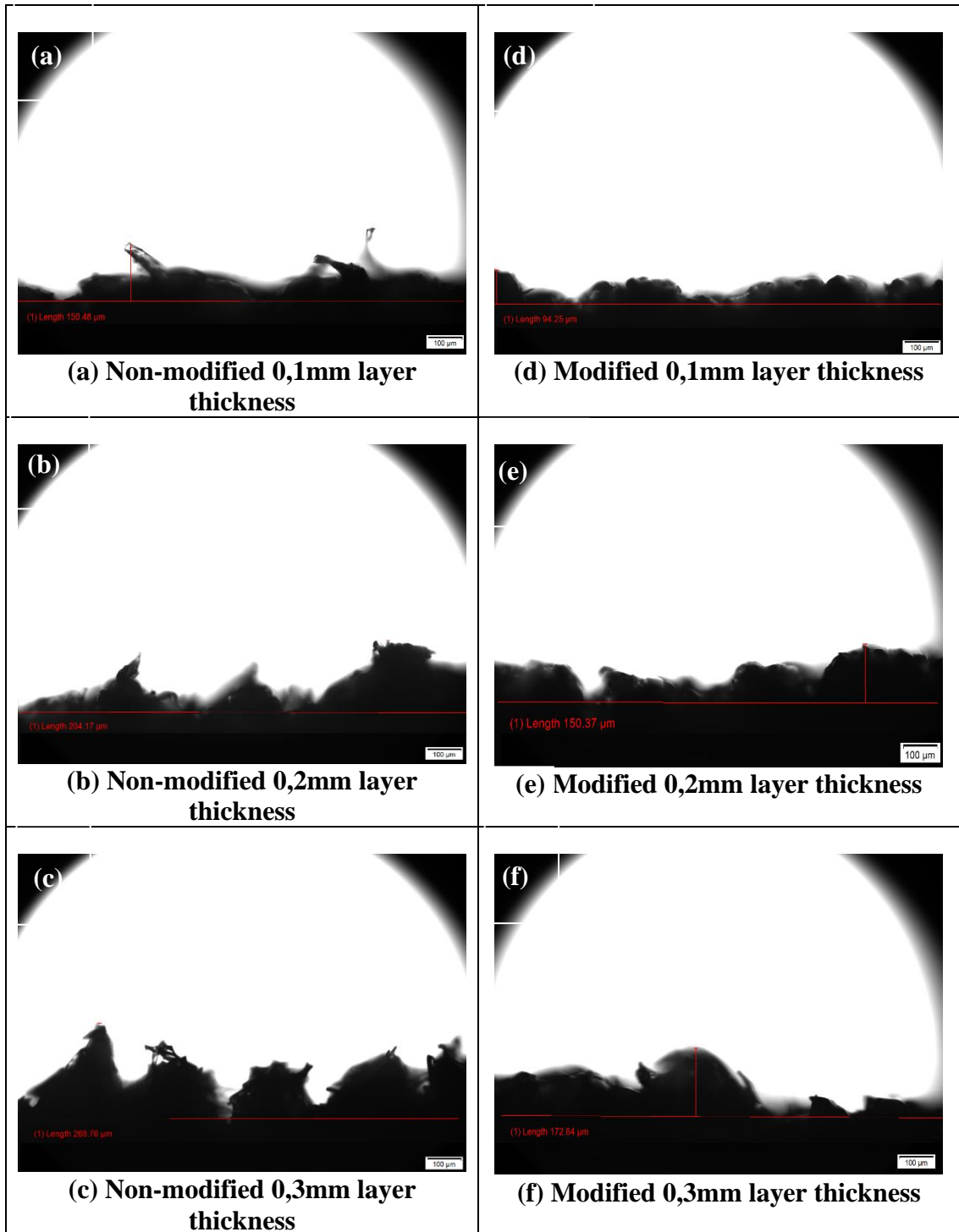


Figure 3.27: PA6CF20 Z Axis Confocal Images

Chaidas et al. surface roughness measurements were performed on PLA samples produced at different nozzle temperatures with the FFF method. It was observed that the surface roughness parameters decreased as the nozzle temperature increased (74). The effects of different nozzle temperature parameters on the surface roughness of

PA6 matrix samples can be studied. Selvam et al. examined the surface roughness values at different layer thicknesses on the samples produced from ABS material by the FFF method and measured that the increase in the layer thickness increased the surface roughness (75). There is a similar situation for PA6 material. In Confocal microscope images, with the increase in layer thickness, it is seen that rougher surfaces are formed in the samples produced by the FFF method.

The changes in the surface roughness depending on the infill pattern in the X-Y axis were also examined with a confocal microscope. A similar study was carried out for PLA material by Alsoufi et al. Alsoufi et al. examined the surface roughness values of the samples produced with 0°, 45° and 90° infill patterned and 0.3, 0.2, 0.1mm layer thickness parameters (76). Similarly, it was observed that the surface roughness values increased with the increase in layer thickness. The highest surface roughness was observed in PLA material produced with a 45° infill pattern. The effects of the infill pattern on surface roughness are similar for PLA and PA6, and confocal microscope images confirm this. In samples produced with a 45° infill pattern, the crossing of the extruded material masses in each layer over the nozzle increases the surface roughness. This situation reveals a feature created by the infill pattern parameter in the method, regardless of the material.

This study shows that an innovative approach to performing surface modification on PA6 matrix composite samples produced by the FFF method with chemical surface treatments(77). Chemical treatments applied to the PA6 matrix improved the surface quality. This may create new areas for polymers produced by additive manufacturing. It can also enable an engineering polymer such as PA6 to be used more effectively and to be preferred in final product production.

Chapter 4

4 Conclusion

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. However, reducing the layer thickness also means more need to time for manufacturing. The layers and the time would be required for 3D printing can be proportionately set by changing layer thickness.

From the results, we can conclude that the surface roughness is directly proportional to the layer thickness, and as the layer thickness increases surface roughness also increases.

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 in the Z axes are higher than in the X-Y axis. 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 energy of the polyamide matrix can be investigated in future studies.

In this thesis study, chemical polishing was applied to improve the surface quality of FFF carbon fiber reinforcement PA6 matrix composite samples. The modification in the surface morphology, surface roughness, contact angle, and confocal microscopy was studied. After chemical polishing, several defects such as gaps and voids were completely detached and a more smoother surface was obtained.

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