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Research Article

Analysis of the Mechanical and Physical Behaviors of 3D-Printed PEEK Structures under Different Parameters

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ABSTRACT

In this study, the mechanical and physical properties of polyetheretherketone (PEEK) material produced through three-dimensional (3D) printing under different production parameters were investigated. For this purpose, the results obtained by applying heat treatment to the produced samples were examined. Comparisons based on mechanical properties were supported by 3D surface profiles and XRD analyses. According to the obtained results, low nozzle diameter and high printing temperature led to an increase of up to 8% in the elastic modulus and up to 33% in tensile strength of the samples. The heat treatment applied in the study increased tensile strength by up to 8% and elastic modulus by up to 9% in samples produced with a low-diameter nozzle. In conclusion, this study aims to provide a dataset for 3D PEEK designs, intending to enhance the performance of PEEK materials that can be used in medical and industrial applications.

1. Introduction

While implants made of metals and alloys are widely used, they often fail to fully meet the requirements for implants due to their high elasticity modulus and low rigidity [1]. Long-term results of implants made from these materials reveal issues such as stress shielding and wear, reducing the lifespan of the implants [2]. Polyetheretherketone (PEEK), a high-temperature polymer with mechanical properties similar to natural bone, has become increasingly popular in medical applications[3].

PEEK, a member of the polyaryletherketone (PAEK) family, stands out for its excellent biocompatibility, radiation transparency, low rigidity, and biochemical stability. Due to these properties, PEEK is considered a promising alternative to commonly used titanium (Ti) alloys [4]. The bone-like characteristics of PEEK are attributed to its elasticity modulus of around 3.6 GPa, and it has been reported to produce significantly fewer artifacts under X-ray radiation compared to Ti alloys [5].

Despite being widely used in solid forms (bulk) for applications such as spinal and dental implants [6], there is ongoing research on the 3D printing of PEEK implants using techniques like Fused Deposition Modelling (FDM) or Fused Filament Fabrication (FFF). In these studies,or Fused Filament Fabrication (FFF). In these studies, the production of implants with complex geometries from polymer-based materials like PEEK is investigated. The high-temperature nature of PEEK necessitates the optimization of parameters for these methods. Achieving the desired rigidity in devices using this material depends on dimensional accuracy, which is challenging due to the high melt viscosity of PEEK [7]. An example summary of parameters used in the literature for the mentioned production methods is provided in Table 1, indicating six parameters that need to be optimized.

Table 1. Manufacturing parameters of the sample PEEK used in the literature for the FFF method.

References	P1* (°C)	P2* (°C)	P3* (°C)	P4* (mm)	P5* (mm/s)	P6* (mm)
[8]	420	160	90	-	12	0.15
[9]	420-430-440	250	-	0.2-0.4-0.6	5-10-15	0.1
[10]	430	-	-	0.2	40	0.2
[11]	470	-	-	0.4	50-10	0.15-0.30
[12]	440	-	-	0.4-0.6-0.8	17-20	0.1-0.25-0.35
[13]	400	100	-	0.4	20	
[14]	360:20:480	-	100-150-200	-	-	-
[15]	334	-	-	0.4	-	0.2-0.3-0.4
[16]	400	100	-	0.4	-	-

* P1- Nozzle temperature, P2- Bed temperature, P3- Chamber temperature, P4- Nozzle diameter, P5- Printing speed, P6- *Layer thickness.*

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Parameter optimization is crucial for achieving the ideal crystallization of PEEK. Upon examining the parameters, it is observed that three parameters are associated with temperature (P1, P2, and P3), two parameters with dimensions (P4 and P6), and one parameter with production speed (P5). PEEK, with a melting temperature of 340 °C [2] is recommended to be printed at temperatures above 380 °C [7]to ensure optimal crystallization. However, despite the melt PEEK temperature being above 380°C in the initial layers of production, it rapidly decreases to the glass transition temperature (140-150°C) [17]. This temperature difference leads to the emergence of different internal structures. It has been reported that a polymer that is not fully crystallized will have a stiffness of approximately 1.9 GPa in the amorphous state or approximately 2.4 GPa in the semi-crystalline state [18].

In this study, various production parameters were considered to enhance the rigidity and mechanical properties of 3D-printed PEEK material. Additionally, a comparative evaluation of the mechanical effects and surface characteristics was conducted through heat treatment. Tensile specimens were produced with dimensions specified by ASTM standards and tested according to the same standards to identify parameters that provide the desired rigidity. The surface morphologies of the produced specimens were examined using an optical microscope and 3D profilometer to identify production defects caused by the parameters.

2. Material and Method

2.1. PEEK Sample Printing and Post-Processing Operations

In the experiments conducted in the study, commercially available pure PEEK filament with a diameter of 1.75 mm (Evonik Naturel PEEK, Evonik Industries AG, Essen, Germany) was used. The material properties of the PEEK filament are provided in Table 2.

Table 2. Material properties of polyetheretherketone (PEEK) filament.

Material Properties	Values
Glass transition temperature	143ºC
Melting temperature	343ºC
Density	1.30 gr/cm^3
Tensile strength	96 MPa
Tensile modulus	3500 MPa

PEEK samples were produced using a 3D PEEK printer (Magic HT M, IEMAI Intelligent Technology Co. Ltd., China) [19, 20]. The technical specifications of the printer are provided in Table 3.

Table 3. Technical specifications of the 3D PEEK printer.

Properties	Values
Print dimensions	220x220x200 mm
Print temperature	450ºC
Bed temperature	150ºC
Chamber temperature	90ºC
Number of extruders	1
Filament diameter	1.75 mm
Layer thickness	0.05-0.3 mm
Position accuracy	X/Y:8.65 µm, Z:2.75 µm
File type	STL,OBJ,3MF,GCODE
Power	200-250 V, 50-80 Hz 950 W

Drying protocol of PEEK filaments can also influence the properties of printed PEEK parts [21]. To prevent bubbles from impacting the prints and the molding, the PEEK filament needs to be dried before the experiment [22, 23]. For these reason and to achieve a stable melt flow in the PEEK filament, drying process was applied in an oven at a constant temperature of 80°C for 7 hours before each production. One hour before the production, the device was started to stabilize the predetermined temperature values (chamber, nozzle, and bed temperatures) for production. The samples to be produced for tensile tests were designed in accordance with ASTM D638 Type-I standards [24] considering the 3D model dimensions shown in Figure 1. The 3D model was sliced in SuperSlicer software, and the necessary G-code for production was obtained.

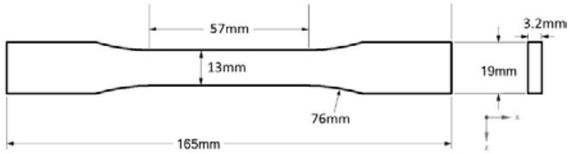


Fig. 1. Dimensions of tensile specimen designed according to ASTM D638 Type-I standard.

For all productions, the bed temperature was set at 150°C, chamber temperature at 90°C, and the printing speed at 15 mm/s. Other main parameters determined for the production throughout the study are listed in Table 4.

Table 4. Main parameters determined for production and their nomenclature.

Nozzle Diameter (mm)	Nozzle Temperature(°C)	Layer Thickness	Heat Treatment
0.4	410	0.2	As-build (P1)
			Heat treated (P1HT)
0.2	420	0.1	As-build (P2)
			Heat treated (P2HT)

The other critical parameters used in production are wall count, scanning strategy, infill ratio, and skirt structure. The wall count parameter used here is a scanning strategy that surrounds the sample in one turn, as shown in Figure 2. A 45° cross-angle scanning method was determined to achieve a more homogeneous printing area within the structure, and a 100% infill ratio was considered. Skirt structures, used only in the first layer of the printed sample, allow the part to adhere more tightly to the bed, preventing warping during production and ensuring stabilization. For the stabilization process in this production, an outward 3 mm skirt structure was used in the first layer. The preview of these parameters in the software is presented in Figure 2.

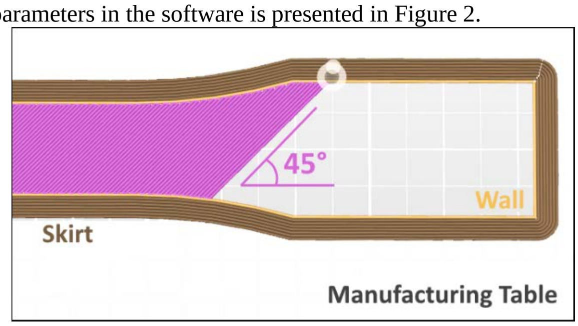


Fig. 2. Pre-production preview of the sliced sample.

PEEK is a high-temperature polymer, and therefore, determining the effects of thermal treatments on its crystal structure and mechanical properties is crucial. To investigate these effects, the produced samples were subjected to heat treatment. The samples were treated at a constant temperature of 300°C for 90 minutes and then gradually cooled in a controlled manner to room temperature over 6 hours at the end of the 90-minute period. This process was conducted to examine changes in the material's crystal structure and mechanical properties.

2.2. Characterization of PEEK Samples

To determine the suitability of the phase structures of the filament used in the FFF device after production and heat treatment, X-ray Diffraction (XRD) was employed using Explorer equipment from GNR, Italy. This method is a reliable and widely used technique to analyze changes in the crystal structure of materials. Through XRD analysis, changes in the structural properties of the filament and the effects of heat treatments have been better understood. Monochromatic Cu-Kα radiation (λ = 0.15418 Å) was used, and XRD data were collected in the range of 0-50° with a scanning speed of 0.02°/s. The peaks obtained from PEEK samples in this analysis will be compared with the literature in the next section.

Following the sizing according to ASTM D638, the samples were mechanically tested in the next stage. The mechanical and surface morphological properties of parts printed under different parameters were determined. Static tensile tests were conducted at a speed of 2 mm/min using a universal static tension-compression device (Model 8872, Instron, MA, USA). Each mechanical test was performed using three samples produced under the same process conditions. Video extensometer was used to track points identified on the front surface of the samples for evaluating mechanical test results. Surface images were taken from cubic samples using an optical microscope with 20x magnification. Roughness values from the front and side surfaces of the same cubic samples were obtained using Bruker-Contour GT 3D Optical Profilometer.

3. Results and Discussion

The analysis results of 3D-printed PEEK samples under different parameters are presented below. XRD analysis was conducted on the samples, and the graph shown in Figure 3 was obtained.

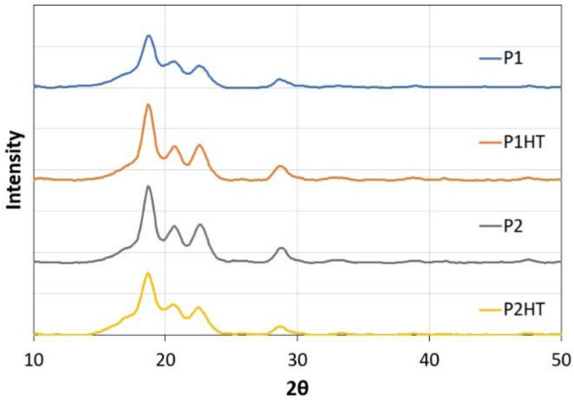


Fig. 3. XRD patterns of the produced samples.

Figure 3 displays X-ray diffraction diagrams covering the 2θ range of 10 to 50 degrees for PEEK samples. Specifically, peaks at 18°, 21°, 23° and 29° are attributed to the orthorhombic PEEK crystal planes (1 1 0), (1 1 1), (2 0 0) and (2 1 0), respectively [13, 25].

In Figure 4, optical images and profilometer images taken from the side surfaces of the produced PEEK samples are presented. The topologies of the surfaces were examined by taking profilometer images from samples produced using two different nozzle diameters (0.2mm-0.4mm).

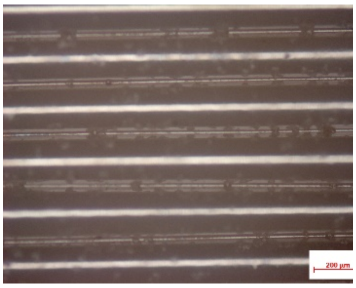
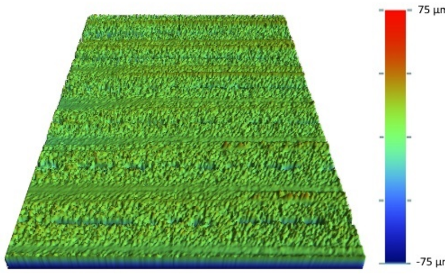
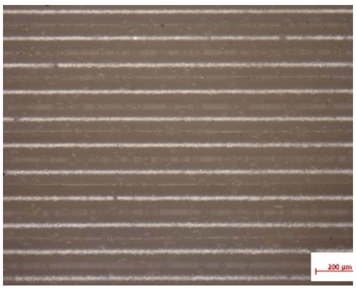
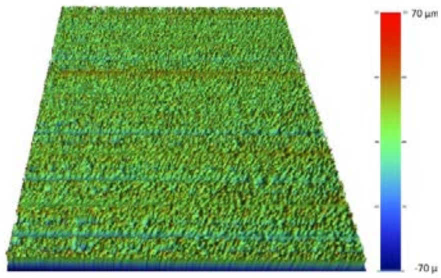
Parameter	Optical microscope	Profilometer
P1		 Ra = 10.796 µm
P2		 Ra = 10.592 µm

Fig. 4. Side views of the samples produced in different layers, taken from the optical microscope and profilometer.

When a 0.4 mm nozzle diameter and 0.2 mm layer thickness were used in the production of samples, it was determined through the experiment that surface roughness and surface fluctuations were higher compared to the test samples produced with a 0.2 mm nozzle diameter and 0.1 mm layer thickness. The impact of different layer heights and nozzle diameters on smoothness is clearly evident (Figure 4). Due to the varying print temperatures in the two different productions, differences in surface roughness were observed due to diffusion occurring between PEEK molecules. The layer thickness, a crucial parameter of additive manufacturing, significantly influences the vertical smoothness. As the layer thickness increases in the compared samples, an increase in surface roughness and fluctuations in the vertical direction is observed. During 3D printing, larger nozzle diameter and layer thickness allow the molten material to spread over a larger area, while smaller nozzle diameter and layer thickness allow finer details to be captured.

Figure 5 presents the results of tensile strengths of the samples. It is observed that the tensile strengths of samples (P2) with reduced nozzle diameter (0.2 mm) and layer thickness (0.1 mm) increased by approximately 33%. Additionally, while the effect of heat treatment on the P1 samples was minimal, a heat-treated P2 sample achieved an approximately 8% increase in tensile strength.

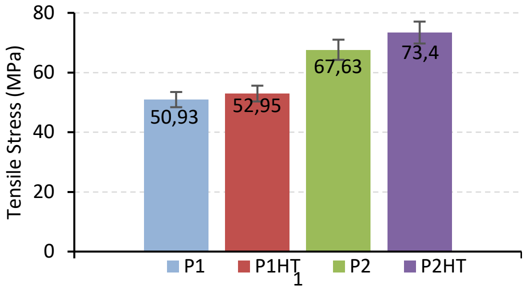


Fig. 5. Tensile strength results of the tested samples.

The stress-strain results drawn using the force-elongation data obtained from the static tensile test are presented in Figure 6. The results are plotted by considering the average of three repeated experiments conducted in the same series. According to these results, the P2H (0.2 nozzle-heat-treated) parameter exhibits the highest strength and yields the best mechanical properties. The P1 (0.4 nozzle-unprocessed) sample is identified as having the weakest mechanical behavior among the cases.

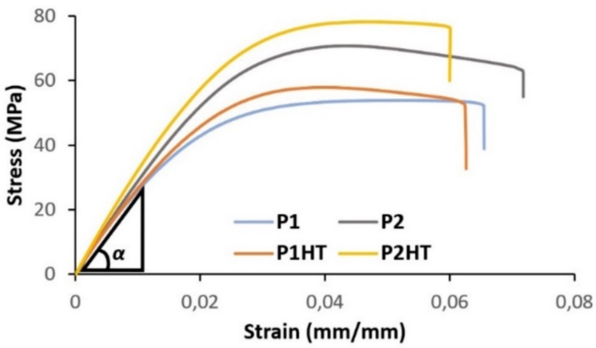


Fig. 6. Tensile test setup and tensile curves of PEEK samples.

The average modulus of elasticity was calculated for each test by considering the range of strain values between 0 and 0.01 in the stress-strain data. Subsequently, the average values of the modulus of elasticity were calculated by averaging three repeated experiments for each sample, and the results are presented in Table 5.

Table 5. Comparison of the mean modulus of elasticity values.

Process Parameters		Sample 1	Sample 2	Sample 3	Average Elasticity Modulus (GPa)
0,4 nozzle	As-buid material (P1)	2.669	2.569	2.558	2.598
	Heat treated (P1HT)	2.492	2.513	2.642	2.549
0,2 nozzle	As-build material (P2)	2.866	2.933	2.630	2.809
	Heat treated (P2HT)	3.217	3.224	2.806	3.082

When examining the elasticity modulus values provided in the table, it is determined that the sample produced with the P2HT parameter exhibits the most rigid behavior. Samples with a nozzle diameter of 0.2 mm are observed to have more rigid elastic values. Additionally, upon reviewing Table 5, it is understood that the heat treatment has no significant effect on the elastic properties of samples produced with a 0.4 mm nozzle diameter.

This study aims to investigate the mechanical and physical properties of 3D-printed PEEK material under different parameters. In this context, the mechanical and physical properties of samples are compared based on nozzle diameter, nozzle temperature, bed temperature, and the application of heat treatment. Other critical parameters such as wall count, scanning strategy, and infill ratios were also considered during the production process. Except for nozzle diameter and heat treatment, all specified parameters were kept constant during the production of the samples. All samples were produced using a 45-degree crosshatch scanning strategy, providing greater contact with the material's printing surface. This was found to have a significant impact, ensuring a stronger bond and higher resistance in the final product.

According to the XRD analysis results, similar to many studies in the literature, the peaks at 18°, 21°, and 23° correspond to the (110), (113), and (200) planes, respectively [19, 26]. When examining the peak points, expansions are observed. The broad crystallization peaks indicate that the samples are semi-crystalline polymers [27]. The XRD profile of all four samples includes an amorphous region with a density not exceeding 20° at 2ϴ. Additionally, there are peaks with lower intensity corresponding to different crystallographic planes indicating the presence of a less ordered phase in the XRD pattern.

Static tensile tests were conducted to determine the mechanical properties of the completed samples. The tensile strength values obtained for P1, P1HT, P2, and P2HT are measured as 50.93 MPa, 52.95 MPa, 67.63 MPa, and 73.4 MPa, respectively. When the results are evaluated based on the change in nozzle diameter, an increase in nozzle diameter is observed to result in a decrease in the tensile strength of the material. Specifically, in the examination of samples produced with a 0.2 mm nozzle, it is observed that the mechanically treated sample has better mechanical properties. However, for samples produced with a 0.4 mm nozzle, it is determined that heat treatment causes a 2.04 MPa change in mechanical properties, with no significant difference.

Using the data obtained from the static tensile test, stress-strain curves were generated for the samples. Through these data, the average modulus of elasticity was calculated for each sample. The modulus of elasticity values obtained for P1, P1HT, P2, and P2HT samples are as follows: 2.598 GPa, 2.549 GPa, 2.809 GPa, and 3.082 GPa, respectively.

This study aims to investigate the mechanical and physical behaviors of 3D-printed PEEK material under different parameters. In this context, a comparative evaluation was conducted by applying heat treatment to samples produced with different parameters. The comparison of mechanical properties was supported by surface images obtained with an optical microscope and profilometer. Additionally, XRD technique was used for crystal structure analysis.

The results reveal that samples with low nozzle diameter and high printing temperature show an increase in elastic modulus of up to 7.5%. Similarly, low nozzle diameter and high printing temperature result in an increase in tensile strength of up to 33%. Furthermore, it was observed that heat treatment provides an increase in tensile strength by up to 8% and elastic modulus by up to 9% for samples printed with a low nozzle diameter.

This study provides a valuable dataset for 3D PEEK designs that will be used in various medical and industrial applications in the future, contributing to research in this field.

4. Conclusions

The aim of this study is to investigate the mechanical and physical properties of 3D printed PEEK material using different printing parameters. To achieve this, a comparative evaluation was conducted by applying heat treatment to the samples produced with varying parameters. The mechanical properties were analyzed using optical microscopy and profilometry to obtain surface images, and XRD analysis was used to examine the crystal structure.

The results showed that samples produced with a low nozzle diameter and high printing temperature exhibited up to a 7.5% increase in elasticity modulus and up to a 33% increase in tensile strength. Additionally, heat treatment provided an increase of up to 8% in tensile strength and up to 9% in modulus of elasticity for samples printed with small diameter nozzles.

Overall, this study provides a valuable dataset for the design of 3D printed PEEK materials for use in various medical and industrial applications, and contributes to the advancement of research in this field.

Declaration of conflicting interests

The authors declare no competing interests.

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