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Research Article

Effect of Annealing on Tensile Strength of 3D Printed PLA with Material Extrusion

Pichai Janmanee and Pongpun Ratchapakdee*

Department of Industrial Engineering, Faculty of Engineering, Rajamangala University of Technology Krungthep, Bangkok, 10120, Thailand

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ABSTRACT

Polylactic acid (PLA) is a commonly used material in 3D printing processes. In the material extrusion (MEX) technique, the final 3D-printed parts have lower mechanical properties. The objective of this study was to investigate the tensile strength of 3D-printed PLA specimens that had undergone annealing. The variables considered were the annealing temperature and the annealing time, with three temperature levels: 70 °C, 90 °C, and 110 °C, and two annealing times: 60 and 90 minutes. The cooling rate is set at 10 °C per hour and cools in the furnace for 24 hours. The results showed annealing significantly affected the tensile strength, with annealed parts demonstrating a notable increase in tensile strength compared to non-annealed parts. Comparing the tensile strength values of pieces that did not undergo annealing, annealed pieces exhibited higher tensile strength. The elasticity modulus tends to decrease, and the workpiece size shrinks slightly in all directions. In the results of the annealing experiment on the ankle foot orthosis (AFO) for pediatric patients with foot drops, it was found that the ankle foot orthosis specimen that had been annealed shrank in all directions with relatively little change. When annealed workpieces are applied, there is no need to compensate for the workpiece size. The highest tensile strength was achieved when annealing was carried out at a temperature of 110 °C for a duration of 90 minutes. The annealed specimen showed an average 42% increase in tensile strength when compared with the printed specimen. The higher the temperature of this glass transition, the higher the calorific value, which will affect the arrangement of the chain and the crystallinity of the plastic and lead to changes in its physical properties. Moreover, the study findings indicate that the optimizing tensile strength of thermoplastic materials can be considerably increased by choosing the ideal process parameters and post-processing conditions.

1. Introduction

Three-dimensional printing (3D Printing) is a process of creating three-dimensional shapes, that follows a similar method to printing and is categorized as additive manufacturing because it is capable of layer-by-layer object creation with acceptable geometric accuracy [1]. Its primary purpose is to generate rapid prototypes [2]. Three-dimensional shapes are constructed from computer model data through software, which defines various parameters arranged in layer sequences. The creation of an object involves printing it layer by layer, starting from the base and progressing upwards [3]. This approach allows the fabrication of complex and intricate shapes without the need for molds, reducing costs significantly. This technology produces objects using the initial material for almost 100%, without the necessity for cutting, drilling, or

* **Corresponding author.**
E-mail address: *649041610035@mail.rmutk.ac.th*

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subtracting material, making it a highly efficient and cost-effective manufacturing method. The rapid development of 3D printing technology has led to the emergence of various innovative printing methods. This progress enables the production of complex shapes in a short amount of time. Currently, one of the most widely used 3D printing techniques is material extrusion (MEX). MEX is preferred due to its cost-effectiveness, ease of use and maintenance [4]. Its technology is widely used for rapid prototyping and has applications in various industries, including aerospace, automotive components, medical devices, and even the food industry. It is a 3D printing technology that was developed by Stratasys [5]. It operates by extruding thermoplastic polymer filaments to create 3D objects. The process begins by feeding polymer filaments into the 3D printer. These filaments are heated until they melt and become liquid, flowing out of the nozzle [6]. The nozzle moves along the X and Z axes, while the platform moves along the Y axis. The material is deposited layer by layer, starting from the base and building upward. The distance between the base and the nozzle, known as the layer thickness, determines the resolution and quality of the final object; see Fig. 1. As 3D-printed objects continue to find applications across various industries, especially in manufacturing machine components, automotive parts, medical equipment, and other fields requiring high mechanical properties, the demand for improved mechanical characteristics in 3D-printed objects has increased. Therefore, there has been the development of materials with improved mechanical properties [7], for example, the study of the mechanical properties of products obtained by reinforcement polymer or metal fibers such as carbon fiber (CFRP), brass (Cu), wood powder, glass fiber (GFRP), etc. [8, 9]. It was found that increasing the reinforcement of composites in different proportions produces products with high mechanical properties. However, these fibers are relatively expensive compared to regular fibers. Additionally, there has been a study on increasing the strength properties by annealing the mechanical properties of polyethylene terephthalate glycol (PETG) and carbon fiber with PETG (CFPETG) reinforcement materials [10]. From the previous studies, it was found that most of them are annealed in reinforced fiber materials such as carbon-reinforced PLA, CFPETG, and PEEK [11-14]. In this situation, annealing proves to be a useful technique for enhancing PLA's mechanical qualities. Consequently, improving PLA's mechanical qualities is a crucial prerequisite for MEX applications. Heating the component over its glass transition temperature and then gradually cooling it down is known as a thermal treatment [11]. The annealing process emerges as an effective method for improving PLA's mechanical properties. From the annealing study, it was found that the temperature and duration. Therefore, it is extremely important to achieve the best mechanical performance of PLA parts, and it has been found that annealing at temperatures above 110 °C results in part distortion [15]. There has been limited research on enhancing the mechanical properties of PLA materials through annealing in the 3D printing process. Therefore, enhancing the mechanical properties of PLA is an essential requirement for MEX applications. Therefore, this research focuses on studying the influence of annealing on the tensile strength of PLA material printed using the filament extrusion process, with the cooling rate set at 10 °C per hour and the material cooling in the furnace for 24 hours. The controlled variables include the printing method, which is the straight-line pattern, 100% infill density, a printing speed of 30 mm/s, and a layer thickness of 0.2 mm. The printed objects are oriented along the X-axis to compare the differences in tensile strength values. This information is crucial for enhancing the mechanical strength of 3D-printed objects in the future.

2. Material and Methods

2.1.Material

The plastic filament used for the experiment was PLA filament, especially from the E-Sun brand, with a diameter of 1.75 mm. It is widely used in MEX 3D printing, derived from renewable resources, and has excellent plasticity for long-term use.

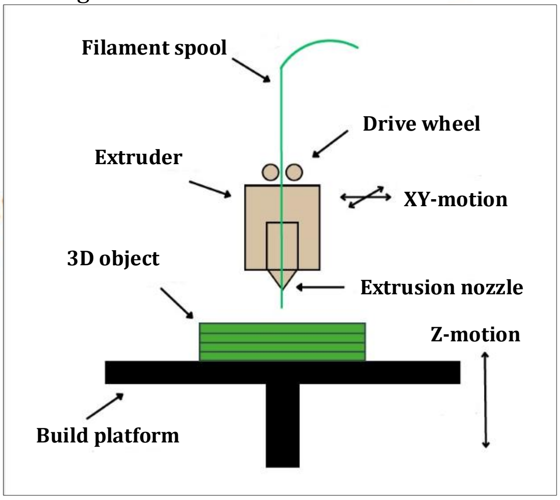

Fig. 1. MEX process

2.2.Preparing the Test Specimen

The test specimen was printed using a filament injection 3D printer with a 0.4 mm nozzle on an Ender 5 Pro 3D model. The printing parameters were set to a printing temperature of 220 °C, a base temperature of 60 °C, a printing speed of 30 mm/s, an infill density of 100%, a layer thickness of 0.2 mm, a linear printing pattern, and a print workpiece in the X-axis direction, as shown in Table 1.

Table 1. The printing parameters

Process parameters	Setting
Printing temperature	220 °C
Base temperature	60 °C
Printing speed	30 mm/s
Infill density	100%
layer thickness	0.2 mm
Printing pattern	linear
Print direction	X-axis
Raster angle	45°/-45°

2.3.Annealing of Test Specimen

The annealing of the test specimen was conducted using a hot air oven, especially a Mermmert oven, with a maximum temperature setting of 200 °C. The cooling rate is set at 10 °C per hour and cools in the furnace for 24 hours. The experimental factors included two variables: (1) Annealing temperature comprised three levels of 70, 90, and 110 °C; and (2) Annealing time had two levels: 60 and 90 minutes, determined by testing three times a total of 18 test specimens and analyzing the results with a statistical program, as shown in Table 2.

Table 2. Variables used in annealing experiments

Factors	Level		
Temperature (°C)	70	90	110
Time (min)	60	90	

2.4.Annealing of Test Specimen

The tensile strength testing was conducted on the test specimens according to ASTM D638 type 1 standards [16]. These specimens were designed using SolidWorks software and manufactured using a Creality 3D printer, as shown in Fig. 3. The specimens were then subjected to testing using a SHIMADZU Universal Testing Machine, specifically the AGS-X model with a 100 kN capacity, as depicted in Fig. 5. The test speed was performed at a controlled speed of 5 mm/min in accordance with the ASTM D638 standards.

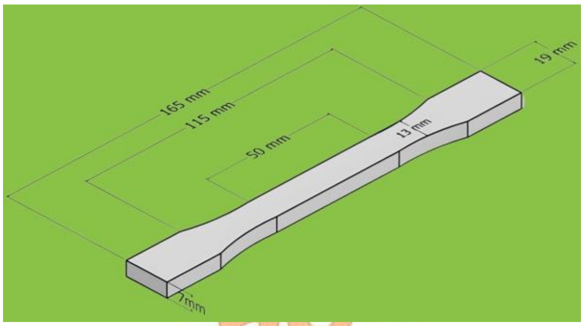

Fig. 2. Test specimen according to ASTM D638 type 1

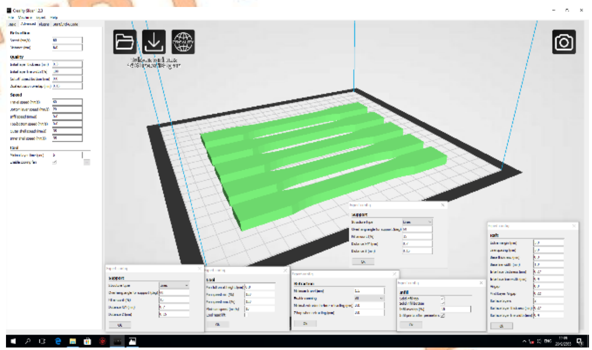

Fig. 3. Specimen printing settings

Fig. 4. Some of the test specimens printed with a 3D printer

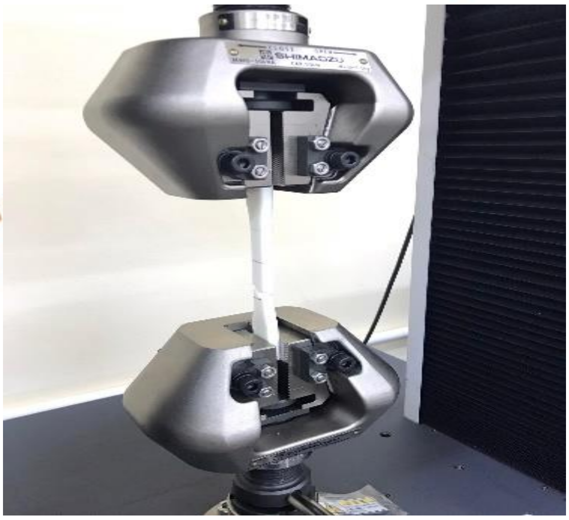

Fig. 5. Tensile test

3. Results

The analysis of variance (ANOVA) was conducted to examine the effects of two factors in an experimental design: (1) Annealing temperature consisted of three levels: 70 °C, 90 °C, and 110 °C; and (2) Annealing time was made up of two levels: 60 min and 90 min. The experiment was replicated three times, and the results were as follows:

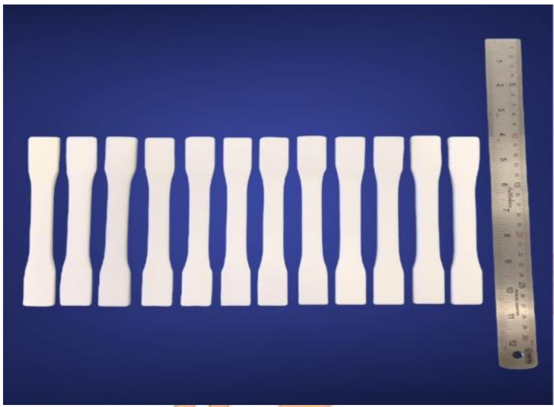

Fig. 5. Some of the testing samples after annealed

3.1. Tensile Strength Test Results

The analysis of the model's accuracy, including checking for the assumptions of normality, independence, and constant variables, was conducted using Minitab 18 software. The statistical data, as shown in Fig. 6, The normal probability plot indicates the data are normally distributed and the variables are influencing the response. Outliers don't exist in the data. Residuals versus fitted values indicate the variance is constant and a non-linear relationship exists. Histogram proves the data are not skewed and no outliers exist. Residuals versus order of the data indicate that there are systematic effects in the data due to time or data collection order, which confirmed that the analysis met these assumptions.

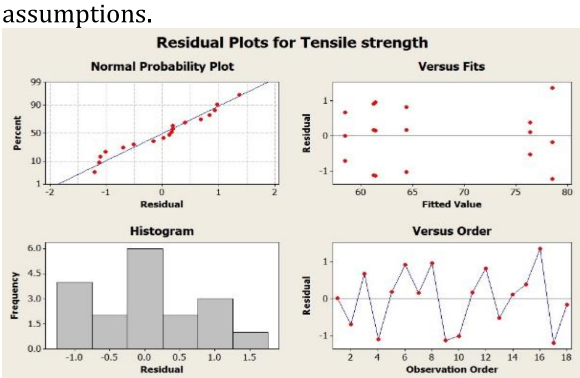

Fig. 6. Analysis of the accuracy of the experiment model

3.2.Impact of Main Factors

As shown in Table 3, the hypothesis testing for the influence of the annealing temperature on tensile strength, as well as the influence of the time spent in annealing, was conducted using an analysis of variance (ANOVA). Due to the fact that the p-values obtained were less than the predetermined significance level of 0.05, there is statistical evidence to support the assertion that the annealing temperature significantly affects tensile strength at a 95% confidence level. Similarly, when testing the hypothesis of the time spent in annealing, as indicated in Table 2, for the two-way ANOVA analysis, the p-value was found to be less than 0.05. This provides statistical support for the claim that the time spent in annealing significantly affects tensile strength at a 95% confidence level. This can be further explained in Fig. 7, which illustrates that both annealing temperature and time spent annealing are factors that have an influence on tensile strength. As observed, an increase in annealing temperature leads to a rise in tensile strength. This finding is in accordance with previous research [17, 18]. Additionally, regarding the time spent in annealing, the result shows that a longer annealing time brings about higher tensile strength values.

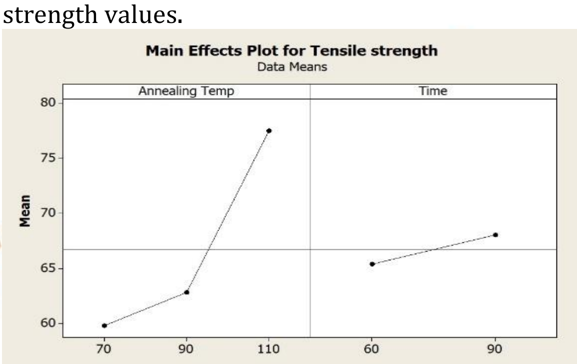

Fig. 7. Relationship of main factors to tensile strength

Table 3. Results of analysis of variance

Source	DF	Adj SS	Adj MS	F	p-value
Annealing Temp.	2	1067.17	533.9	592.41	0.000
Time	1	31.28	31.28	34.73	0.000
Temp * Time	2	0.62	0.31	0.35	0.715
Error	12	10.81	0.90		
Total	17				

S=0.949056 R-sq=99.03% R-sq(adj)=98.6%

3.3.Interaction Effect

Hypothesis testing for the interaction effect between annealing temperature and time spent in annealing, based on the data presented in Table 2 and the results from the analysis of variance (ANOVA) as shown in the variance analysis table, indicated that the p-value was greater than the predetermined significance level of 0.05. As a result, there is no statistical evidence to support the claim that the combined influence of annealing temperature and time significantly affects tensile strength at a 95% confidence level.

This can be further illustrated in Fig. 8, which demonstrates that increasing the values of both annealing temperature and time spent in annealing has only a negligible impact on tensile strength.

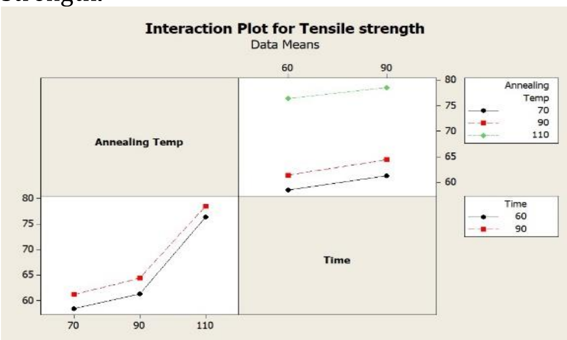

Fig. 8. Relationship of total factors to tensile strength

From Fig. 8, it can be observed that both the annealing temperature and the time spent in annealing result in a slight increase in tensile strength.

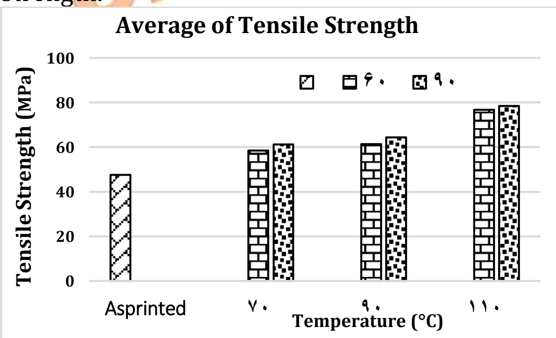

Fig. 9. Comparison graph of average tensile strength.

As displayed in Fig. 9, it is evident that when comparing the average tensile strength values, workpieces that have not undergone annealing have an average tensile strength of 47.55 MPa. For workpieces annealed at 70 °C with annealing times of 60 and 90 minutes, the average tensile strengths are 58.44 and 61.19 MPa, respectively. Workpieces annealed at 90 °C with annealing times of 60 and 90 minutes have average tensile strengths of 61.36 and 64.38 MPa, respectively. Workpieces annealed at 110 °C with annealing times of 60 and 90 minutes exhibit average tensile strengths of 76.79 and 78.53 MPa, respectively. The data reveal that the tensile strength tends to increase as the annealing temperature increases from 70 °C to 110 °C. Additionally, increasing the annealing time from 60 to 90 minutes has a minor effect, resulting in slightly higher tensile strength. However, it is important to note that while the tensile strength increases, the modulus of elasticity shows a decreasing trend as both temperature and annealing time rise, as depicted in Fig. 10. The highest increase in tensile strength was 78.53 MPa from annealing at 110 °C for 90 minutes.

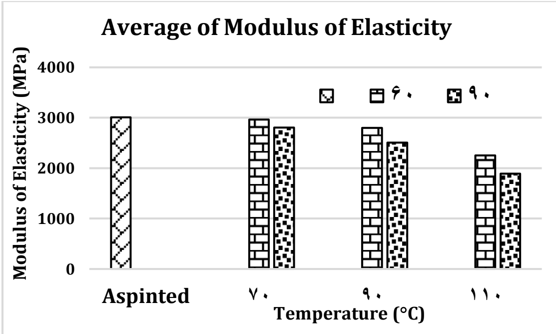

Fig. 10. Graph comparing the average modulus of elasticity.

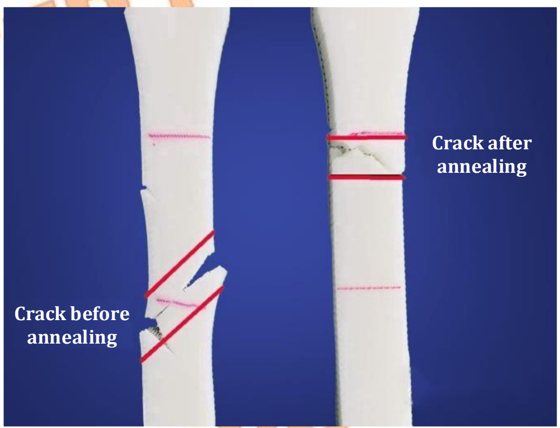

Fig. 11. Crack typology before and after annealing.

In Fig. 11, when considering a comparison of the characteristics of cracks in the test specimens before and after annealing, it was found that the cracks in the test specimens that underwent annealing exhibited straight-line fractures and had very little flexibility. This is because, after annealing, the annealing temperature increases, and the bonding between filaments becomes more homogenous, leading to a smoother fracture surface, resulting in test specimens having increased tensile strength due to the rearrangement of their structure, allowing them to withstand more force [19]. However, these specimens are more brittle compared to the test specimens that did not undergo annealing. In comparison, the parts of the test specimens that did not go annealed had cracks that were inclined in the direction of printing. It shows that the failure occurred between layers. (inter-layer) first, in the form of layer separation, the adhesion between layers weakens as the printing temperature decreases rapidly with each layer. but had greater flexibility.

3.4.Annealing Effect on Dimension Variation

The results of the annealing experiment on the ankle foot orthosis (AFO) for pediatric patients with foot drop aimed at examining the effect on dimension variation of the workpiece after annealing at 90 °C for 60 minutes by measuring the changes in size along the X, Y, and Z axes.

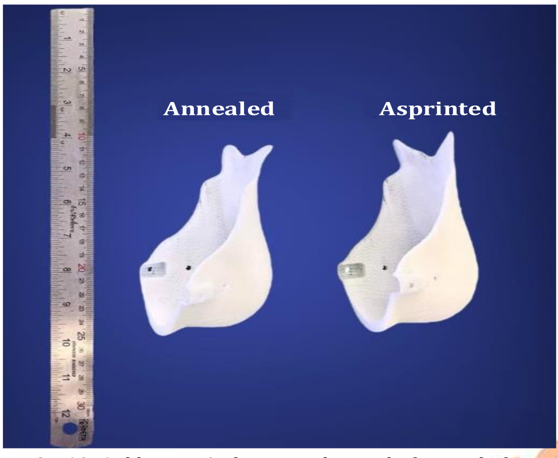

Fig. 12. Ankle Foot Orthosis workpiece before and after annealing

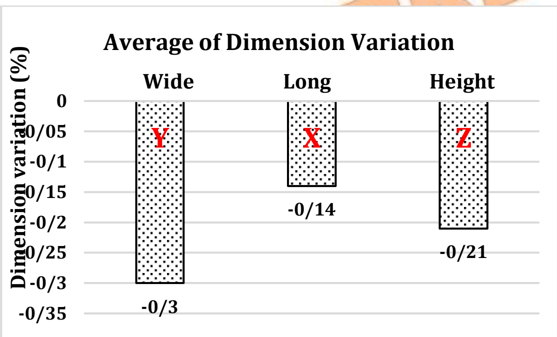

Fig. 13. Comparison graph of changing size values

As displayed in Fig. 13, comparing the size dimension, it was found that the ankle-foot orthosis specimen that had been annealed shrank in all directions with relatively little change, with the Y-axis (-0.30%), X-axis (-0.14%), and Z-axis (-0.21%) values in agreement with the results of prior research [20].

3.5.Microstructure Investigations

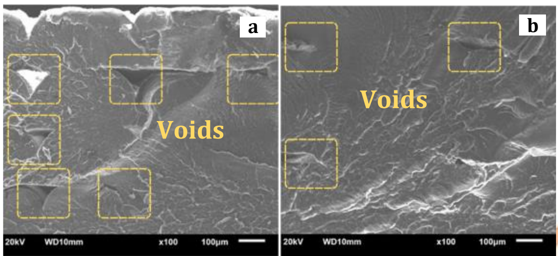

Fig. 14. (a) PLA tensile test fracture surface without annealing (b) PLA tensile test fracture surface with annealing.

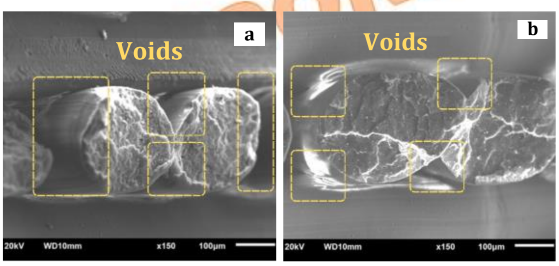

Fig. 15. (a) Cross-section of parts without annealing (b) Cross-section of parts with annealing.

From Fig. 14, when examining the surface microstructure of the test specimen after tensile testing, both before and after annealing, it was observed that the annealed specimen exhibited a reduction in voids or gaps between layers compared to the printed specimen, similar to the analysis of cross-sectional fiber arrangement before and after annealing. In Fig. 15, it is found that the annealed workpiece When it gets hot, the fibers in each layer have more fusion. This causes the fibers to stick together more in each layer. As a result, the voids between the printing layers are reduced and more homogeneity exists in the filament bonding. As a result, the tensile strength of the workpiece increases.

4. Discussion

The results of the annealing experiment on the three-dimensional printed workpiece reveal trends in tensile strength when subjected to varying annealing temperatures and times. Tensile strength tends to increase with higher annealing temperatures. However, the growth in annealing time has a relatively minor effect on increasing tensile strength. The highest increase in tensile strength, reaching 78.53 MPa, was observed when annealing at a temperature of 110°C for 90 minutes. These findings can be attributed to the 3D printing process, which results in different temperature gradients within each layer of the workpiece. Additionally, rapid cooling of the workpiece leads to internal stress, reducing its mechanical properties. This aligns with previous research [21, 22]. Upon reheating the workpiece, a rearrangement of the internal structure occurs, resulting in a more organized structure and a reduction in internal stress. Therefore, heating is higher than the glass transition temperature (Tg) because at this glass transition temperature, plastics with a disorderly arrangement of molecular chains, including semicrystalline characteristics, have both crystalline and amorphous parts mixed together, which will change the new structural state, and the molecular chains are arranged better than the traditional ones, allowing the workpiece to have a slow cooling rate. The higher the temperature of this glass transition, the higher the calorific value will affect the arrangement of the chain and the crystallinity of the plastic and lead to changes in physical properties. This leads to improved mechanical properties, which are consistent with other research studies [23-25]. In contrast, the modulus of elasticity exhibits a decreasing trend as both annealing temperature and time increase [26].

5. Conclusions

The study examined the influence of annealing on PLA material that had undergone three-dimensional printing and its effect on tensile strength properties. In this study, annealing temperature was considered at three levels: 70, 90, and 110 °C, and annealing time at two levels: 60 and 90 minutes. Tensile strength measurements were conducted, and the results revealed significant variations. Comparing the tensile strength values between the workpieces that had not undergone annealing and those that had, it was observed that the annealed workpieces exhibited higher tensile strength, the elasticity modulus tended to decrease, and the workpiece size shrunk slightly in all directions. Therefore, when annealed workpieces are applications, there is no need to compensate for the workpiece size. This increase in tensile strength can be attributed to the annealing process, which reduces internal stresses within the workpiece. The varying temperature gradients experienced by each layer during 3D printing contribute to this phenomenon. The annealed specimen showed an average 42% increase in tensile strength when compared with the printed. The highest increase in tensile strength was reached after applying the annealing process at a temperature of 110 °C for 90 minutes. Moreover, the study findings indicate that the optimizing tensile strength of thermoplastic materials can be considerably increased by choosing the ideal process parameters and post-processing conditions.

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