THE EFFECT OF THERMAL ANNEALING BETWEEN GLASS TRANSITION TEMPERATURE AND CRYSTALLIZATION TEMPERATURE ON DIMENSIONAL ACCURACY OF 3D PRINTED PART

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Jaya Suteja
Universitas Surabaya

Sunardi Tjandra
Universitas Surabaya

THE EFFECT OF THERMAL ANNEALING PROCESS BETWEEN GLASS TRANSITION TEMPERATURE AND CRYSTALLIZATION TEMPERATURE ON DIMENSIONAL ACCURACY OF 3D PRINTED PART

The Jaya Suteja
Universitas Surabaya
Email: jayasuteja@staff.ubaya.ac.id

Michael Evan
Universitas Surabaya
Email: mike99evan@gmail.com

Sunardi Tjandra
Universitas Surabaya
Email: s_tjandra@staff.ubaya.ac.id

ABSTRAK

Akurasi dimensi dari hasil *3D printing* mempengaruhi fungsi, kekuatan, dan proses perakitan dari komponen tersebut. Penelitian ini bertujuan untuk mengetahui pengaruh implementasi proses *thermal annealing* terhadap akurasi dimensi komponen *Polylactic Acid* yang dicetak menggunakan *Fused Filament Fabrication*. Penelitian ini menggunakan metodologi 3^2 desain faktorial dengan dua pengulangan. Dua parameter dari proses *thermal annealing* yang diselidiki adalah temperatur *annealing* dan waktu penahanan. Temperatur *annealing* diatur antara temperatur transisi gelas dan temperatur kristalisasi, yaitu 65 °C, 75 °C, dan 85 °C. Waktu penahanan yang digunakan adalah 45 menit, 60 menit, dan 75 menit. Respon dari penelitian ini adalah penyimpangan dimensi pada arah X, Y, dan Z. Berdasarkan hasil percobaan, ditemukan bahwa penggunaan temperatur *annealing* yang lebih tinggi meningkatkan penyimpangan arah X, Y, dan Z. Hasil analisis mengindikasikan bahwa penggunaan temperatur *annealing* sedikit di atas suhu transisi gelas menghasilkan derajat kristalisasi yang rendah tetapi menghasilkan akurasi dimensi yang lebih tinggi daripada temperatur *annealing* di bawah suhu kristalisasi. Sedangkan penggunaan waktu penahanan 60 menit meningkatkan penyimpangan pada arah X dan Z tetapi menurunkan penyimpangan pada arah Y.

Kata kunci: *Thermal annealing*, transisi gelas, kristalisasi, akurasi dimensi, *3D printing*.

ABSTRACT

The dimensional accuracy of a 3D printed part affects the function, strength, and assembly process of the part. This study aims to determine the effect of the implementation of thermal annealing process on the dimensional accuracy of Polylactic Acid parts printed by Fused Filament Fabrication. This research uses 3^2 *factorial design methodology with two replications. Two investigated parameters of the thermal annealing process are the annealing temperature and the holding time. The annealing temperature is set between the glass transition temperature and the crystallization temperature, which are 65 °C, 75 °C, and 85 °C. The holding times used are 45 minutes, 60 minutes, and 75 minutes. The response of this* *research is the dimensional deviation in the X, Y, and Z directions. Based on the experiment result, it was found that the use of higher annealing temperature increases the X, Y, and Z-direction deviations. The analysis result indicates that the use of annealing temperature slightly above the glass transition temperature results in a low degree of crystallization but produces higher dimensional accuracy than annealing temperature below the crystallization temperature. While the use of a holding time of 60 minutes increases the deviation in the X and Z directions but decreases the deviation in the Y direction.*

Keywords: *Thermal annealing, glass transition, crystallization, dimensional accuracy, 3D printing.*

1. INTRODUCTION

Fused Filament Fabrication (FFF) is a 3D Printing technology that is widely used for manufacturing prototypes. The printing process using FFF uses raw materials in the form of filaments with a certain diameter, which are then heated between glass transition temperature (Tg) and melting temperature (Tm). The glass transition temperature is the temperature at which the material begins to change from a solid to an elastic rubber-like form. The melting temperature is the temperature at which the material changes from a solid to a liquid state. After being heated, this filament is filled into one or more nozzles and then extruded raster by raster and layer by layer to form a certain geometry. Due to the characteristics of the FFF process, the printed part has a low strength.

There are several methods that can be conducted to increase the strength of the printed part, such as optimizing process parameters [1], adding reinforcement material to the filament [2], or conducting a post-processing step such as the thermal annealing process [3]. Each method has advantages and disadvantages. The most commonly used method is optimizing process parameters. However, this method cannot significantly increase the strength of the printed part because it is very dependent on the printed material.

The addition of reinforcement material can increase the strength of the printed part significantly because it has a reinforcement material that has a much higher strength than the virgin material. However, the addition of this reinforcement material will require a more complex extrusion process because the reinforcing material needs to be extruded through the nozzle. Meanwhile, the implementation of the post-processing step can increase the strength of the printed part and may improve the surface quality of the printed part. However, this post-processing step requires additional time and process. In addition, the post-processing involving heating and cooling such as thermal annealing process can cause dimensional deviation of the printed part.

Dimensional accuracy of the printed part that has been thermal annealed needs to be investigated because it affects the function, strength, and assembly process of the printed part. This study aims to investigate the effect of the thermal annealing process on the dimensional accuracy of the FFF printed part made of Polylactic Acid (PLA). The thermal annealing process for polymers is carried out by heating the material at a certain temperature and time and then cooling it until it reaches room temperature

Before an experiment is conducted, several research papers were reviewed to identify the important factors affecting the dimensional accuracy and define the hypothesis. Based on the literature study, the dimensional accuracy of the printed part after undergoing the thermal annealing process was influenced by its process parameters. Second, dimensional accuracy is also affected by the characteristics of the FFF printed part obtained before the thermal annealing process. Last, it is influenced by the properties of the material especially its glass transition temperature.

The important thermal annealing process parameters are the annealing temperature, the length of time the material is held at the annealing temperature, and the cooling rate of the material to reach room temperature. The use of high annealing temperature causes a significant temperature difference between the part temperature and the room temperature. The temperature difference reduces the dimensional accuracy of the printed part due to the development of internal stresses [3]. Meanwhile, the implementation of a short holding time causes the heat energy received by the material to be not enough to be able to create a uniform temperature of the printed part. The non-uniform temperature increases internal stresses and causes dimensional deviations [3]. Lastly, the cooling rate of the material also affects the dimensional accuracy. The use of a high cooling rate causes the internal stress to increase and reduces the dimensional accuracy of the printed part [4].

The relationship between the thermal annealing process parameters also affects the dimensional accuracy of the printed part. Low temperature but with a high cooling rate causes a decrease in dimensional accuracy. The printed part that is annealed at a low temperature and then cooled rapidly produces low and non-uniform crystallization. Low and non-uniform crystallization cause low order crystal structure. This will cause geometric distortion and reduce dimensional accuracy [5].

Next, the dimensional accuracy of the annealed part is influenced by the characteristics of the FFF printed part obtained before the thermal annealing process. In printed part that has a large empty space or void between layers, the thermal annealing process reduces the dimensional accuracy of the printed part. When this printed part is thermal annealed, the void will shrink and produce inaccurate part dimensions. This void is affected by several factors, such as raster thickness and layer thickness. In addition, the density of the printed part also affects the dimensional accuracy. The density value of the printed part is influenced by the use of a certain infill pattern and the density of the infill pattern. When the density is getting smaller, the dimensions will be inaccurate due to the shrinkage of the void between the raster [4].

Last, the glass transition temperature of different materials also affects the dimensional accuracy of the thermal annealed part. By performing the thermal annealing process using an annealing temperature lower than the glass transition temperature of a material, the crystallization process will be more difficult to occur. The lower crystallization degree causes the dimensional accuracy to be lower due to the low order crystal arrangement [6]. To be able to reach the fastest crystallization rate, the annealing temperature used must reach the crystallization temperature (Tc) of the material.

The hypothesis taken in this research is the use of annealing temperature below the crystallization temperature may increase the dimensional accuracy as long as the temperature is above the glass transition temperature. Therefore, this research is focused to investigate the effect of the annealing temperature, which is higher than the glass transition temperature but below the crystallization temperature on the dimensional accuracy of the printed part. The annealing temperature is set so that the temperature difference between the printed part and the room temperature is not too large. In this study, the annealing temperature variations used are 65 °C, 75 °C, and 85 °C because the glass transition temperature of PLA is between 55 °C and 60 °C and the crystallization temperature of PLA is between 95 °C and 115 °C [7].

This research also investigates the effect of the holding time used in the thermal annealing process because the combination of temperature and holding time can affect the tensile strength of the PLA printed part. This study uses variations in holding times of 45 minutes, 60 minutes, and 75 minutes because the highest tensile strength can be obtained at an annealing temperature of 80 °C or 75 °C with a holding time of 60 minutes [8, 9]. The effect of the variation of the holding time can be used as a preliminary study to optimize the dimensional deviation and the tensile strength.

2. METHODOLOGY

This study implements 3^2 factorial design methodology with two replications so it requires 18 (eighteen) parts printed by FFF. The printed part is designed to resemble a tensile test specimen that meets the ASTM D638 standard [10]. Anet® A8 FFF printer made by Shenzhen Anet Technology Co., Ltd. is used to print the specimen. This FFF printer has a work table with a size of 220 mm x 220 mm x 240 mm and an extruder nozzle with a diameter of 0.4 mm. Before being printed, the PLA material is in the form of a 1.75 mm diameter filament, made by CCTREE 3D Printing. The FFF process parameters used to make the specimens can be seen in Table 1. For the slicing process, this research uses the Prusa Slicer 2.3® software developed by Prusa Research and the Repetier-Host 2.2.2® software developed by Hot-World GmbH & Co. KG.

Table 1. Process Parameters of FFF

Parameters	Value
Nozzle Diameter (mm)	0.4
Bed Temperature (°C)	55
Nozzle Temperature (°C)	210
Print Rate (mm/s)	50
Layer Thickness (mm)	0.15
Number of Shell	3
Infill Density (%)	100
Raster Angle (°)	45
Infill Pattern	Rectilinear
Build Orientation	X-Y

After printing the specimen, the dimensions of the specimen are measured using a Mitutoyo® caliper with an accuracy of 0.01 mm and a measuring capacity of 150 mm. There are three dimensions that are measured, which are the dimensions in the X, Y, and Z directions. The dimensions measured in this study are shown in Figure 1 below.

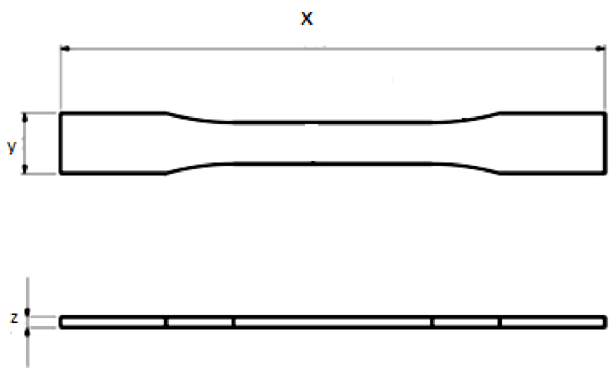

Figure 1. Measured Specimen Dimensions

Then, the specimen is annealed using several combination values of temperature and holding time, as shown in Table 2. The annealing temperature and holding time are the investigated parameters of the experiment. The thermal annealing process was carried out by heating the specimen in an Ofenbau Hofmann® furnace. Variations in temperature and holding time values used followed the 3^2 factorial design methodology with two replications. After the process is heated and held, the specimen is removed from the furnace and cooled at room temperature until it reaches room temperature.

Table 2. Thermal Annealing Parameters

Parameters	Value		
	Low	Medium	High
Annealing Temperature (°C)	65	75	85
Holding Time (minutes)	45	60	75

Finally, the specimens were re-measured using a Mitutoyo® caliper with an accuracy of 0.01 mm and a measuring capacity of 150 mm. The final dimensions after the thermal annealing process were recorded and compared with the dimensions before the process. Then the dimensional difference of each direction is recorded as deviation. The deviations, which are X, Y, and Z deviations, are used as the response of the experiment.

3. RESULTS AND DISCUSSION

Based on the experiment results, it was found that the thermal annealing process causes a dimensions reduction in the X and Y directions but causes an increase in the Z direction. Deviation in the X, Y, and Z directions as a result of the use of annealing temperature and holding time is shown in Table 3. Figure 2 shows the average deviation in the X, Y, and Z directions as a function of annealing temperature and holding time. From Figure 2(a) it can be seen that the use of higher annealing temperature increases the X direction deviation from 3.1 mm to 5.3 mm. While the holding time increases and then decreases as the holding time changes from 45 minutes to 60 minutes, and then from 60 minutes to 75 minutes. The X direction deviation will be maximum when using 60 minutes of holding time.

Increasing the temperature also increases the Y direction deviation as shown in Figure 2(b). The Y direction deviation increased from 0.23 mm to 0.43 mm with increasing temperature from 65 °C to 85 °C. The holding time decreases and then increases as the holding time changes from 45 minutes to 60 minutes, and then from 60 minutes to 75 minutes. The maximum deviation occurs when using a holding time of 75 minutes.

Same as X and Y direction deviation, Z direction deviation also increases with increasing temperature as shown in Figure 2(c). The minimum deviation of 0.13 mm occurs when using a temperature of 65 °C. While the use of a temperature of 85 °C cause the maximum deviation of 0.23 mm. The holding time increases and then decreases as the holding time changes from 45 minutes to 60 minutes and then from 60 minutes to 75 minutes. The maximum deviation in the Z direction of 0.19 mm occurred when using 60 minutes of holding time.

Table 3. Experiment Results

Temperature (°C)	Holding Time (minutes)	Deviation (mm)		
		X-Axis	Y-Axis	Z-Axis
65	45	3.45	0.30	0.15
65	45	3.15	0.30	0.10
65	60	3.05	0.00	0.10
65	60	3.00	0.10	0.15
65	75	2.95	0.55	0.10
65	75	3.00	0.10	0.15
75	45	4.15	0.30	0.20
75	45	4.10	0.45	0.20
75	60	5.10	0.20	0.20
75	60	5.00	0.50	0.15
75	75	3.90	0.65	0.10
75	75	4.40	0.35	0.30
85	45	5.80	0.45	0.20
85	45	5.00	0.25	0.20
85	60	5.20	0.60	0.25
85	60	5.60	0.35	0.30
85	75	5.20	0.45	0.20
85	75	5.25	0.45	0.20

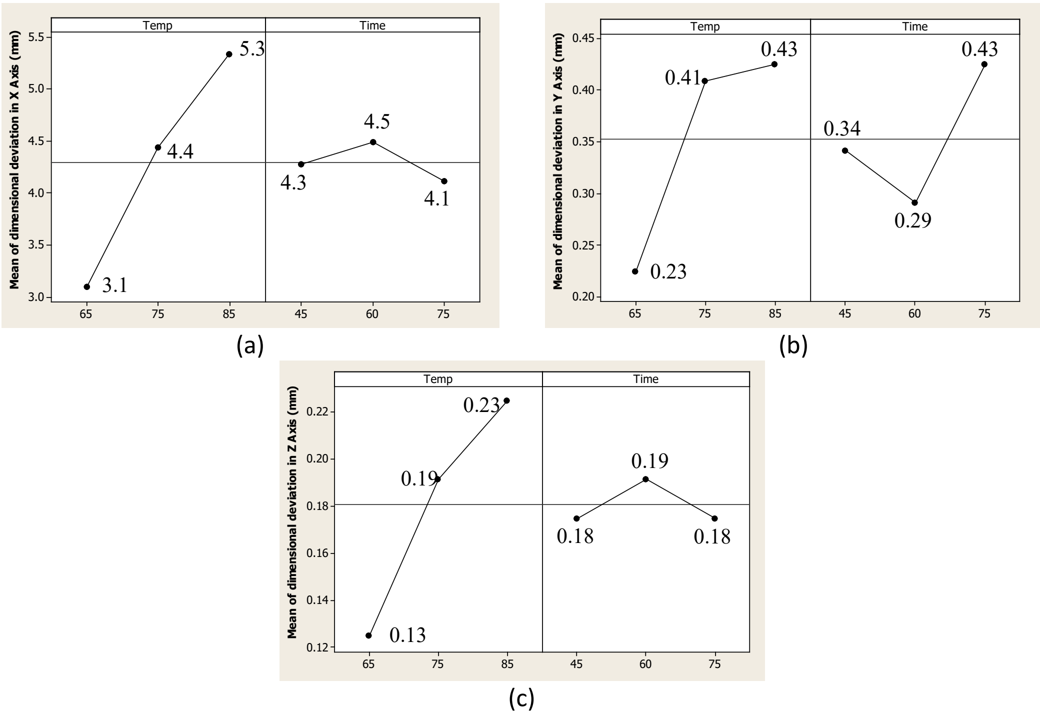

Figure 2. Mean of dimensional deviation in (a) X-axis; (b) Y-Axis; (c) Z-Axis as a function of annealing temperature and holding time

It can be concluded that increasing annealing temperature causes dimensional deviation to increase. By conducting an analysis of variance with a 95% confidence level, it is found that the annealing temperature has a significant effect on the dimensional deviations of the X and Z directions. However, the annealing temperature does not significantly affect the Y direction deviation.

While the use of a holding time of 60 minutes increases the deviation in the X and Z directions but decreases the deviation in the Y direction. To achieve the minimum deviation in the X and Z directions, the holding time used must be 75 minutes. However, from the analysis of variance, it is found that the holding time has no significant effect on dimensional deviation in all the X, Y, and Z directions.

The results of this study show that the thermal annealing process performed at an annealing temperature slightly higher than the glass transition temperature results in more accurate dimensions compare to annealing temperature slightly below the crystallization temperature. The implementation of temperature slightly higher than the glass transition temperature causes lower order crystal arrangement. In addition, the implementation of temperature slightly below the crystallization temperature causes low crystallization rate. However, the use of higher annealing temperature causes a significant temperature difference between the part temperature and the room temperature and decreases the dimensional accuracy of the thermal annealed part. It indicates that the use of annealing temperature slightly above the glass transition temperature creates a lower degree of crystallization but produces a higher dimensional accuracy printed part than annealing temperature slightly below the crystallization temperature.

4. CONCLUSION

This study aims to investigate the effect of the thermal annealing process on the dimensional accuracy of the PLA part printed by using FFF. Based on the experimental results, it can be seen that the thermal annealing process causes a reduction in the X and Y directions of dimensions but can increase in the Z direction. It can be observed that the use of high annealing temperature increases dimensional deviation. While the use of a holding time of 60 minutes increases the deviation in the X and Z directions but decreases the deviation in the Y direction. The analysis result indicates that the use of annealing temperature slightly above the glass transition temperature results in a low degree of crystallization but produces higher dimensional accuracy than annealing temperature slightly below the crystallization temperature. The results of this research can be used to determine the lowest annealing temperature that can be implemented to produce printed part with accurate dimensions and sufficient tensile strength as a functional component.

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