

EXPERIMENTAL ANALYSIS OF TEMPERATURE RESISTANCE OF 3D PRINTED PLA COMPONENTS

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PLA is one of the most widely used materials in additive technology, especially in the Fused Filament Fabrication method. However, its use is limited by its relatively low-temperature resistance. The glass transition temperature of PLA is around 60 °C. Some studies already performed on the annealing of 3D printed plastics have shown their increased heat deflection temperature. However, the interpretation of some of these studies' results does not correspond to our practical experience. For example, one of those works reports that annealed PLA samples soften at much higher temperatures than the original nonannealed ones. This paper intends to better understand the behaviour of annealed printed PLA samples at higher temperatures and extends previously performed studies to determine glass transition temperatures using TGA. The samples are tested in two devices to determine the temperature resistance. The first is based on the standard for determining the heat deflection temperature according to ISO 75. In the second device, the sample is subjected to tensile stress at a specific temperature under the simultaneous action of stress induced from the screw connection. The results show that the annealed samples' glass transition temperature does not differ significantly compared to the nonannealed samples. Furthermore, tests have shown that annealed samples deform under relatively small load at higher temperatures than nonannealed samples, but this deformation depends on its magnitude. If the load is high enough, the annealed specimens deform at the same temperature as the nonannealed specimens.

KEYWORDS

Annealing, Fused Filament Fabrication, Glass Transition Temperature, Heat Deflection Temperature, Additive Manufacturing, Polylactic Acid

1 INTRODUCTION

3D printing currently plays a key role in manufacturing and design [Beniak 2019]. It owes this mainly due to its large range of possible applications and favourable price, especially in piece production. Therefore, it is maximally used in the design of prototypes, such as robotic snake's cover [Virgala 2021], topologically optimized parts [Paska 2020, Jancar 2020], robot jaws [Vocetka 2020] or mechanical parts of racing formulas [Mesicek 2019]. One of the most common methods of 3D printing is Fused Filament Fabrication (FFF), mainly for its simplicity and low price [Beniak 2020]. One of the most widely used materials for this technology is polylactic acid (PLA) [Van der Walt 2019].

When designing products, the designer needs to know the properties of the material well. PLA material is characterized by very good mechanical properties [Zakaria 2019]. However, its

use is limited by its relatively low-temperature resistance. The glass transition temperature of PLA is around 60 °C [Sodergard 2002], which means that it starts to soften from this temperature. The material may deform appreciably in the soft state due to even a small force or even deform due to its own weight. The softening temperature of PLA parts at 60 °C can be a problem in some typical examples of the use of printed parts, such as motor flanges [GrabCAD 2021]. During operation, the motor may become so hot that the PLA printed part softens at the screw connection, which may loosen the screw connection [Thingiverse 2016].

On the topic of PLA parts' behaviour at higher temperatures, some studies have already been performed, which describe mainly the increase of heat deflection temperature after annealing [Rigid 2017, Koci 2019, Feeney 2019]. PLA annealing is a known heat treatment process in which the internal stress of the material caused by production on a 3D printer is minimized, and thus higher strength of printed parts is achieved [Suder 2020]. However, our own experience with these materials in the annealing process contradicts some claims published in temperature resistance studies of annealed plastics [Feeney 2019], where it is stated that the material begins to soften due to annealing about 170 °C. According to our practical experience, the annealing of PLA plastics increases their heat deflection temperature. However, it begins to soften at the same temperature as in nonannealed samples. This means that the annealed material begins to deform at a higher temperature only if the load is small enough. Otherwise, if the load is high enough, the annealed samples start to deform at the same temperature as the nonannealed ones. That means the load influences the heat deflection temperature. In some cases, for example, by the screw connection, the annealed samples will behave the same as to nonannealed ones.

This work aims to elucidate the behaviour of PLA printed parts at higher temperatures by determining their glass transition temperature using thermogravimetric analysis (TGA) and performing heat deflection temperature. These tests consist of a classical heat deflection temperature test, as performed in studies [Rigid 2017, Koci 2019, Feeney 2019], based on the standard for determining heat deflection temperature of plastics ISO 75 [ISO 75 2020]. Furthermore, tests of heat deflection temperature under tensile stress are performed, while in this test, the sample is also loaded by the stress caused by the screw connection. This is to clarify the behaviour if both the place with the higher stress (the area of contact of the screw connection) and the place with the lower stress (only a small tensile force act) deform at the same temperature. This work does not aim to determine the exact values of deformations depending on stress and temperature.

2 SAMPLE PREPARATION

All samples were printed by Fused Filament Fabrication. Some of them were then heat-treated by annealing.

2.1 Production of samples

Three most used different types of PLA material were used for this testing. Classic simple PLA [Prusament 2020], PLA-HD [Fiberlogy 2020] (this type is specially designed for annealing), and PLA-PLUS [Gembird 2020] (compared to the classic, it has added additives to improve some of its properties).

All samples were printed by Fused Filament Fabrication on an ORIGINAL PRUSA i3 MK3S printer [Prusa 2020]. The software PrusaSlic n2.2.0 was used to generate the G-code needed for this printer. [Prusa3D 2020]. The default setting of printing parameters for PLA materials was used, except the infill

parameter, which was set on the value of 100 %. This value prints solid material so that the object does not deform inside due to annealing. The selection of the most important printing parameters is shown in Table 1.

Parameter	Value
Nozzle width	0.4 mm
Layer height	0.2 mm
Infill	100 %
Nozzle temperature	215 °C
Bed temperature	60 °C
Number of perimeters	2
Perimeter speed	45 mm/s
Speed for infill	80 mm/s
Speed for first layer	20 mm/s
Speed for top layer	40 mm/s

Table 1. The used basic printing parameters

The shape of the samples and its dimensions are shown in Figure 1. The cross-section of the samples corresponds to the required dimensions for testing the heat deflection temperature of plastics according to ISO 75 [ISO 75 2020].

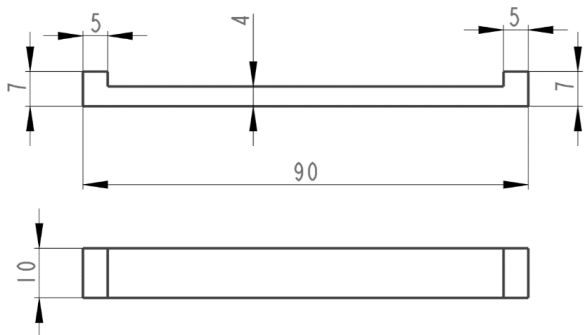


Figure 1. Dimensions of the sample

For each individual test, 10 samples were printed from each material. Thus, a total of 360 samples were printed.

2.2 Annealing

Samples annealing was performed in a Memmert Universal Oven UN30 industrial dryer [Mettler 2020]. The annealed samples were two sets, one annealed at 80 °C and the other at 100 °C. These temperatures were selected according to the work [Suder 2020], which dealt with the annealing of these materials. The temperature was maintained for 30 minutes, and then the samples were removed from the oven, where they were slowly cooled to room temperature.

3 DESCRIPTION OF EXPERIMENTS

The samples were tested for TGA to determine the glass transition temperature, and then other samples were tested for heat deflection temperature under bending stress and then in a heat deflection temperature device for tensile stress under simultaneous stress induced by a screw joint.

3.1 Glass transition temperature testing

Glass transition testing was performed on a TGA / DSC 2 STARE System [Mettler 2021]. Because the printed samples are too large for this analysis, approximately 10 grams of material was taken from them and tested. Temperature analyzes ranged from room temperature to a maximum temperature of 250 °C. This

temperature is sufficient for the measurement, as about 240 °C the material begins to melt.



Figure 2. Machine TGA / DSC 2 STARE System

3.2 Heat Deflection Temperature

Two measurement methods were tested for measuring Heat Deflection Temperature. The first is based on ISO 75 [ISO 75 2020], which tests the heat deflection temperature to bending stress. The second method measures the heat deflection temperature under tensile stress, while in this test, the sample is also loaded by the stress caused by the screw connection.

3.1.1 Heat deflection temperature resistance test in bending stress

Figure 3 shows the principle of the heat deflection temperature test, which is based on ISO 75 [ISO 75 2020]. The test specimen is placed on two supports, subjected to a force in the middle. The whole device is gradually heated to 50 °C. The temperature is then maintained for 10 minutes to warm the entire device, including the sample. It is then checked for sample deflection. If there is no deflection of the sample, the temperature is increased by 5 °C, and the whole process is repeated until it is visibly bent (approx. 1 mm). Samples were tested at a load of 15 g (weight of nut M10) and 100 g (4 screwed-in nuts M10 on a screw M10x45). Due to the slight differences in weights of the purchased nuts, the nuts were weighed, and only those nuts were selected for testing that weighed 15 g to the nearest 0.01 g. A weight of 15 grams will cause bending stress of approximately 0.1 MPa in the sample and a weight of 100 grams will cause bending stress of approximately 0.7 MPa.

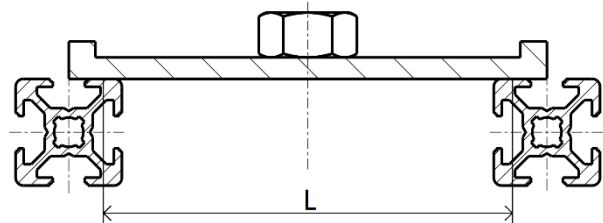


Figure 3. Heat deflection temperature resistance test in bending stress

3.1.2 Heat deflection temperature test under tensile stress

Since ISO 75 [ISO 75 2020] only describes bending stresses, we were interested in how the specimens will behave under tensile stress under the simultaneous action of stress caused by a screw joint. Figure 4 shows the principle of the tensile stress resistance test.

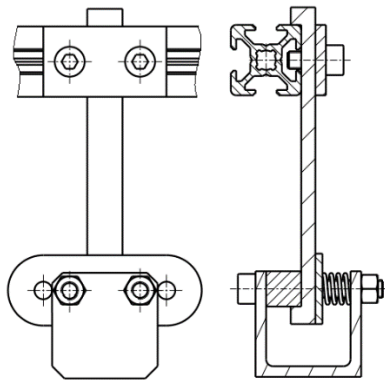


Figure 4. Principle of heat deflection temperature test under tensile stress

The test specimen is bolted to the upper frame. The sample is provided with protrusions that prevent it from falling out of the frame in the event of a broken screw connection caused by softening the material and loss of prestress. A weight of 50 g is suspended from the bottom of the sample, which causes tensile stress of approximately 0.012 MPa in the cross-section of the sample. The whole device is gradually heated to 50 °C. The temperature is then maintained for 10 minutes to warm the entire device, including the sample. It is then checked that the sample has not been stretched and that the screw connection has not become loose. If the sample does not stretch, the temperature is increased by 5 °C, and the whole process is repeated until it is stretched (by about 1 mm). If the screw connection loosens, the sample is still attached to the device by its shape, and the measurement continues.

4 RESULTS

The results consist of three different sections, namely TGA, to determine the glass transition temperature and two heat deflection temperature tests.

4.1 Glass transition temperature

Using TGA performed on a TGA / DSC 2 STARe System [Mettler 2021], three graphs were plotted for all three tests material types. Figure 5 shows a graph from TGA for PLA material.

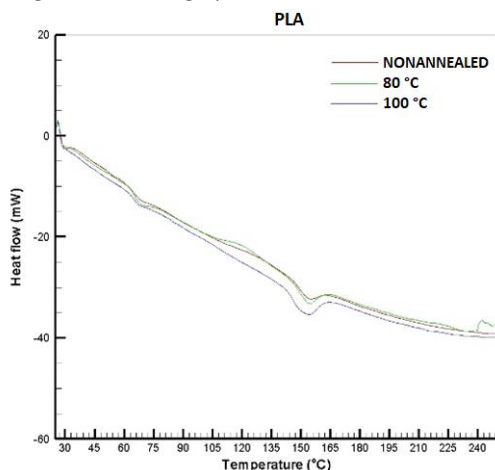


Figure 5. TGA of PLA material

It is clear from the figure that the material's behaviour did not differ significantly between annealed and nonannealed samples. Near 60 °C, the glass transition is visible in all PLA samples.

Figure 6 shows a graph from TGA for PLA-PLUS material.

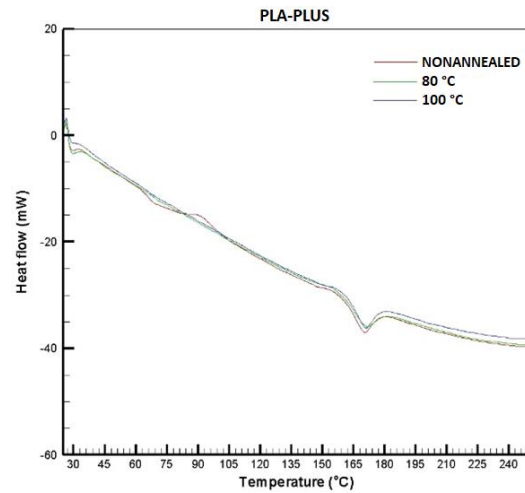


Figure 6. TGA of PLA-PLUS material

In the case of PLA-PLUS, the glass transition area is around 60 °C. By the nonannealed samples is the area good visible, while in the case of annealed samples, this area is less obvious.

Figure 7 shows a graph from TGA for PLA-HD material.

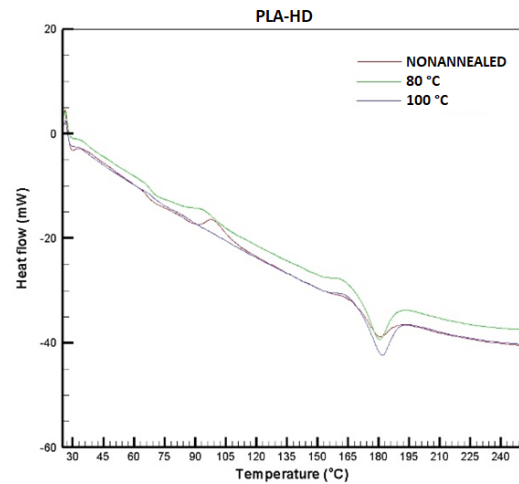


Figure 7. TGA of PLA-HD material

Also, with PLA-HD, a glass transition area is visible around 60 °C, and as with all samples tested above, annealing did not change this area.

Glass transition's temperatures exact values were read from the graphs shown above and entered in Table 4. The values indicate that the glass transition temperature did not change significantly with annealing, which means that all samples, regardless of the annealing process, begin to soften at approximately the similar temperature.

Annealing Temperature	Glass transition temperature (°C)		
	PLA	PLA-PLUS	PLA-HD
Non-annealed	59.6 ±0.5	62.6 ±0.3	63.8 ±0.4
80 °C	61.2 ±0.4	62.9 ±0.4	65.8 ±0.5
100 °C	62.4 ±0.5	62.8 ±0.4	66.8 ±0.3

Table 2. Glass transition temperature

4.2 Heat deflection temperature resistance test in bending stress

Additional specimens were tested for the heat deflection temperature test, which bends the test specimen. All samples from the individual groups were deformed at the same

temperature, which was measured for the used method with an accuracy of 5 °C. The results of this test are shown in Table 3.

Annealing Temperature	Heat deflection temperature (°C)					
	PLA		PLA-PLUS		PLA-HD	
	Load (g)		Load (g)		Load (g)	
	15	100	15	100	15	100
Non-annealed	60	60	60	60	60	60
80 °C	125	75	135	85	140	90
100 °C	125	75	135	85	140	90

Table 3. Results of heat deflection temperature under bending stress

It is evident from the measured values that all nonannealed samples deflect at 60 °C, which corresponds to their softening temperature. The dependence between load and temperature resistance is also observed from the results. At higher loads, the temperature resistance is lower. The highest temperature was maintained by PLA-HD material when annealing at both temperatures. The annealing temperature did not affect the resulting temperature resistance for either material. There was no difference in the samples' results annealed at 80 °C and 100 °C.

Figure 8 shows this test at 80 °C, where it is visible how the classical PLA (black samples) show deformation, while PLA-PLUS (white samples) still retain their shape.



Figure 8. Heat deflection temperature resistance test in bending stress at 80 °C

4.3 Heat deflection temperature test under tensile stress

Finally, the samples were tested by a heat deflection temperature test under tensile stress. All samples from the individual groups were deformed at the same temperature, which was measured for the used method with an accuracy of 5 °C. The results of this test are shown in Table 4.

Annealing Temperature	Heat deflection temperature (°C)		
	PLA	PLA-PLUS	PLA-HD
Non-annealed	60	60	60
80 °C	150	160	165
100 °C	150	160	165

Table 4. Results of heat deflection temperature test under tensile stress

It is clear from the table that the nonannealed samples stretch at a glass transition temperature of about 60 °C. Heat deflection temperature is higher for annealed samples. The highest

temperature was maintained by PLA-HD. The annealing temperature did not affect the resulting temperature resistance for any material. There was no observed difference in the samples' results annealed at 80 °C and 100 °C.

Simultaneously, when monitoring the stretching, the experiment was focused on monitoring the loosening of the screw connection. From 60 °C, all samples tested deformed at the area where the sample was screwed to the device's upper frame, as shown in Figure 9.

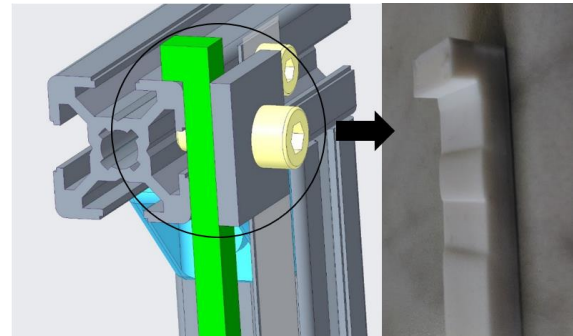


Figure 9. Deformation of the material at the area of application of stress from the screw connection

Due to this plastic deformation, the stress in the bolted joint decreased (its exact size was not the subject of this investigation) and the bolted joint cannot be considered reliable.

5 CONCLUSIONS

Testing annealed and nonannealed samples for the glass transition temperature and heat deflection temperature yielded several notable results.

It was measured by TGA that the annealing of the printed PLA samples did not affect their glass transition temperature. None of the three tested PLA materials did the glass transition temperature after annealing differ significantly from the nonannealed samples. As a result, the samples begin to soften at a temperature of about 60 °C, whether or not they have undergone an annealing process. This information is very essential for designers. Some articles may confuse them with their claims when they state that materials begin to soften due to annealing at much higher temperatures [Feeny 2019]. Their claims are based only on temperature resistance tests, which is not the right method to determine the material's softening temperature.

Heat deflection tests have shown increased temperature resistance of annealed samples. It should be noted here that this is only a matter of keeping the shape at a higher temperature, not the fact that the material is already softening. When testing the heat deflection temperature to bending at a relatively low load, the annealed samples, compared to nonannealed ones, retain their shape even at higher temperatures. However, the magnitude of this temperature depends on the magnitude of the load, the higher the load, the lower the temperature it can resist before it begins to deform. At a load of 15 grams, the PLA-HD material withstood the highest temperature of 140 °C, while at a load of 100 grams, the temperature resistance dropped to 90 °C. A weight of 15 grams will cause bending stress of approximately 0.1 MPa in the sample and a weight of 100 grams will cause bending stress of approximately 0.7 MPa. Samples made of classic PLA material withstood the lowest temperatures, which with their load of 100 grams withstood their shape up to a temperature of 75 °C.

Finally, the heat deflection temperature test under tensile stress was performed, where an additional screw connection was added. A weight of 50 g is suspended from the bottom of the sample, which causes tensile stress of approximately 0.012 MPa in the cross-section of the sample. This testing, similar to the bending test, showed that annealing increases dimensional stability at higher temperatures. The highest temperature resistance of 165 °C were reached for PLA-HD material and the lowest 150 °C for conventional PLA. However, in the case of a screw connection, plastic deformation occurred in all three tested materials at 60 °C in the area of the screw connection contact. This behaviour confirms the TGA results that the material softens at approximately the same temperature regardless of the annealing process. The reason is that the temperature resistance depends on the magnitude of the stress. The loads in the test methods produced much lower stress values than in contact with the screw connection joint.

The results showed that annealing increases the temperature resistance, which proves dimensional stability at higher temperatures but depends on the applied stress's magnitude. If the stress is large enough, as in the case of a screw connection, the annealed material deforms at the same temperature as the nonannealed material, i.e., at a temperature around the glass transition temperature. We recommend choosing another material with a higher glass transition temperature in real situations where a higher operating temperature occurs to loosen screw connection joints in PLA printed materials (such as printed motor flanges).

The results of this work can serve as a proposal for further more complex testing of temperature resistance. The effect of stress on temperature resistance could be determined or at which magnitude of stress the annealed samples do not have higher temperature resistance to nonannealed.

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