The effect of annealing on deformation and mechanical strength of tough PLA and its application in 3D printed prosthetic sockets

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Abstract

Purpose – Fused filament fabrication (FFF) using tough poly lactic acid (PLA) was determined to be the most suited method to achieve low-cost prosthetic sockets. However, improvement in the material properties is desirable to strengthen these sockets. This study aims to evaluate annealing as a potential method to improve material properties by a heat treatment of the object after 3D printing.

Design/methodology/approach – Four different annealing methods and a control group were tested according to ISO standard 527–1 and ISO standard 527–2. The four annealing methods included: oven; sand; water; and glycerol annealing. Tests were performed on longitudinal and transversal 3D printed samples. Deformation was determined on 3D printed test rings.

Findings – Annealing using an oven, sand and water resulted in a significant increase in tensile strength in longitudinally 3D printed tensile test samples. However, the tensile strength was decreased in the transversally 3D printed tensile test samples. The tensile modulus had no significant increase in the longitudinally and transversally printed samples. Sand annealing resulted in the least deformation, with a shrinkage of 2.04% of inner diameter and an increase in height of 1.99% for the horizontally annealed test rings.

Research limitations/implications – The annealing of prosthetic sockets is not recommended as a decrease in tensile strength in transversally printed tensile test samples was observed. More research is needed towards the strengthening of tough PLA in both print directions.

Originality/value – This paper fulfils the need for understanding the impact of annealing on 3D printed items intended for daily use, such as a prosthetic socket.

Keywords Mechanical properties, Deformation, 3D-printing, Annealing, Tough PLA

Paper type Research paper

1. Introduction

Fused filament fabrication (FFF) is a print technique that can built objects layer by layer using thermoplastic material. However, the main concern with FFF printed objects is the problem of delamination. As in any manufacturing process using layering, the strength of the FFF socket depends on how well one layer of material is bonded to another. The amount of inter-filament bonding between the printed layers and high internal stresses due to different heating and cooling rates makes objects weaker (Lam, 2017; Butt and Bhaskar, 2020).

From 2018, we started a project in which we research low-cost prostheses for people in low and middle income countries with the use of computer-aided design (CAD) and computer-aided manufacturing (CAM). FFF using tough poly lactic acid (PLA) was determined to be the most suitable material due to its high tensile strength and rigid properties (van der Stelt *et al.*, 2020;

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Tensile tests were conducted on a Hydraulic MTS at the Department of Orthopedics at the Radboudumc, Nijmegen, the Netherlands. The authors are grateful that they were able to use this facility.

Authorship confirmation statement.

All authors had substantial contributions to the concept and design of the work. Final approval to publish this version is given by all authors. They also agreed to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

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Ultimaker, 2018a, 2018b). However, research into how material properties can be further improved is desirable.

Annealing is a potential method to improve material properties. With annealing a heat treatment alters the microstructure of a material causing changes in properties such as strength, hardness and ductility. The process of heating an object above the glass transition, but below the melting temperature, increases the crystallinity of the material and redistributes the stress within the material (Butt and Bhaskar, 2020; Wang *et al.*, 2019). The amount of crystallinity determines the performance of the material, improving stiffness, strength, heat deflection temperature and chemical resistance (Srithep *et al.*, 2012). However, a disadvantage of annealing is that the 3D-printed object might deform, this can affect its function (Butt and Bhaskar, 2020; Aydın and Çağlar Okur, 2018).

The effect of annealing has only been researched on normal PLA. Generally, the degree of crystallinity depended on the annealing time and temperature (Lam, 2017; Butt and Bhaskar, 2020; Wang *et al.*, 2019; Srithep *et al.*, 2012; Chen *et al.*, 2020). However, its efficacy on the exact increase in tensile strength, tensile modulus and the amount of deformation ranged greatly across the studies (Lam, 2017; Butt and Bhaskar, 2020; Wang *et al.*, 2019; Srithep *et al.*, 2012; Chen *et al.*, 2020). As tough PLA consists of an unknown mixture of PLA and acrylate polymers, the results might also not be translatable to the annealing of tough PLA (Ultimaker, 2019a, 2019b). Moreover, annealing can be performed using different methods, such as heated air or using a water-bath, but no comparison was made between different annealing methods (Lam, 2017; Butt and Bhaskar, 2020; Srithep *et al.*, 2012; Chen *et al.*, 2020). Thus, the exact increase in tensile strength, tensile modulus and the amount of deformity might also highly depend on the annealing method.

In this study, the influence of annealing on the material properties of tough PLA is investigated according to ISO standard 527–1 and 527–2 (International Organization for Standardization, 2012a, 2012b).

2. Methods

Tensile test were performed using ISO standard 527–1 and 527–2 to determine the influence of annealing on the tensile strength and the tensile modulus of Tough PLA (ISO, 2012a, 2012b). Four different methods of annealing were investigated: oven, sand, water and glycerol annealing. In addition, test rings were used to determine the influence of annealing on the deformation of tough PLA, in both vertical and horizontal annealing orientation.

2.1 Manufacturing tensile test samples

The tensile test samples were manufactured according to the type 1B test specimen dimensions mentioned in ISO standard 527–2 (ISO, 2012b). Six tensile test samples were used to evaluate each annealing method and the control group, three printed in longitudinal direction and three printed in transversal direction. The tensile test samples were printed with black Tough PLA filament and 3D-printed with the Ultimaker S5 (Ultimaker, 2018a, 2020a). Cura 4.6.1 was used as slicing software package (Ultimaker, 2020b). Print settings are shown in Table 1. An example of how tensile test samples are printed, in longitudinal and transversal direction, is shown in Figure 1.

Six test rings were produced to evaluate the deformation of each annealing method. The test rings were designed based on the prosthetic socket designed by van der Stelt *et al.* (2020). The dimensions of the rings are shown in Figure 2. During annealing, three test rings were placed vertically, and three rings were placed horizontally to evaluate the deformation of the rings in each orientation. The test rings were 3D printed in transversal direction, according to the print settings shown in Table 1. An example of how a test ring is printed is visualised with blue lines in Figure 2.

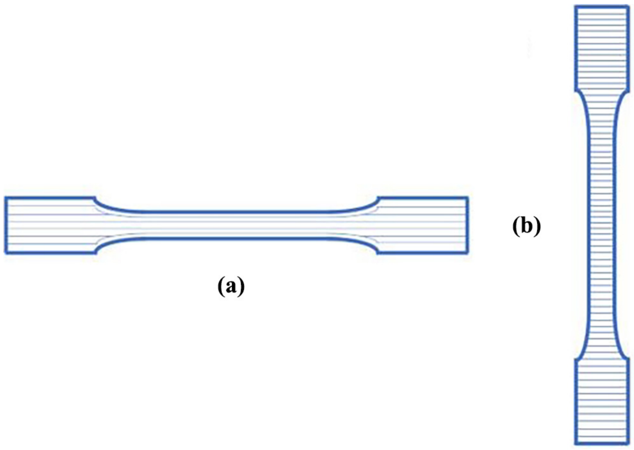
2.2 Methods of annealing

Four different methods of annealing were used: oven; sand; water; and glycerol annealing. These methods were chosen as they are widely available and easily applicable in low and middle income countries (Sarcevic, 2019). For the oven annealing, a Siemens HB331.4 convection oven with fan (Siemens, 2021) was used. The tensile test samples were placed on a baking tray, lying flat.

Table 1 Parameters used for 3D-printing of the tensile test samples in longitudinal and transversal direction

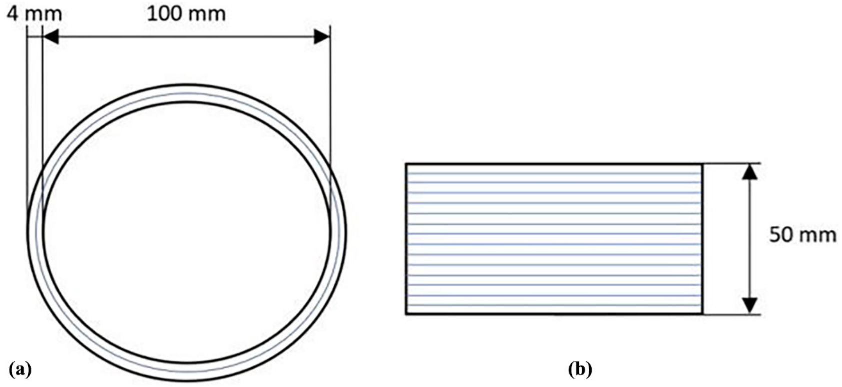
	Longitudinal direction	Transversal direction
Nozzle diameter	0.8 mm	0.8 mm
Print temperature	210 °C	210 °C
Bed temperature	60 °C	60 °C
Layer thickness	0.2 mm	0.2 mm
Print speed	45 mm/s	45 mm/s
Print orientation	Lying flat	Standing
Infill percentage	100%	100%
Infill pattern	Lines	Lines
Infill direction	Lengthwise	Circular
Flow rate	1	1

Figure 1 Images of the longitudinally and transversally printed tensile test samples with printing layers



Notes: (a) longitudinally printed; (b) transversally printed

Figure 2 Dimensions of the test rings. Views from above and from the side re shown. Printings layers are displayed with blue lines



(a) (b)

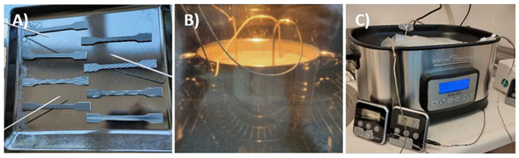
Notes: (a) above view; (b) side view

The second method was sand annealing. The sand is used to prevent bending and warping of the tensile test samples by supporting them and to achieve a more even temperature around the tensile test samples. The sand was used in combination with the Siemens HB331.4 oven (Siemens, 2021). The tensile test samples were placed in a container filled with sand, which was placed in the oven.

For the hot water annealing, the ProfiCook Sous-Vide cooker PC-SV 1112 (ProfiCook, 2021) was used. The tensile test samples were placed in a vacuum plastic bag to prevent any absorption of water into the tough PLA (Elsawy *et al.*, 2017; Kim *et al.*, 2016). The plastic bag was weighed down with weights to ensure the tensile test samples remained fully submerged.

Glycerol annealing is a method to prevent bending and warping of the tensile test samples as the densities of glycerol and PLA are similar (Ultimaker, 2019a; Takamura *et al.*, 2012). For glycerol annealing, the water in the Sous-Vide cooker was replaced with Glycerol 99.5% (Mystic Moments UK, Fordingbridge, UK). A visualisation of each method in shown in Figure 3.

Figure 3 Visualisation of oven, sand, water and glycerol annealing



Notes: (a) oven annealing; (b) sand annealing; (c) water and glycerol annealing

All annealing methods took place at a temperature of 80°C for 30 min. The annealing temperature was controlled with three separate Westmark digital thermometers (Westmark GmbH, Lennestadt, Germanty). These thermometers were placed at an equal distance from each other, either above the baking tray, in the sand, in the water or in the glycerol. The temperature of each thermometer was noted every minute to evaluate whether the tensile test samples were annealed at the right temperature. When 30 min had passed, the tensile test samples and test rings were allowed to cool down to a room temperature of 20°C in the oven or liquid. Gradual cooling down prevents the build-up of internal stresses due to uneven cooling and reduces deformation of the tensile test samples (Lam, 2017; Koci, 2019). The heating, annealing and cooling time per annealing method are shown in Table 2. The control group did not undergo any processing after printing.

2.3 Tensile testing

Tensile testing of the tensile test samples was performed according to ISO standard 527–1 and ISO standard 527–2 (ISO, 2012a, 2012b). Tensile tests were conducted on a Hydraulic MTS (15 kN) (MTS, Eden Prairie, MN, USA) with an axial torsion load cell, type 2816 in the orthopaedic research lab at the Radboudumc, Nijmegen, The Netherlands. The tensile test samples were tested at a speed of 1 mm/min to determine the tensile strength and the tensile modulus (ISO, 2012b). The tensile test samples were elongated until break.

2.4 Dimensional analysis

Before and after annealing, the dimensions of the test rings were measured using a Vernier calliper (Spurtar, Guangzhou,

Table 2 Heating, annealing and cooling time per annealing method (when 30 min of annealing time had passed, the tensile test samples and test rings were cooled to room temperature in the oven or liquid)

	Heating time (min)	Annealing time (min)	Cooling time (min)
Oven	15	30	200
Sand	60	30	270
Water	50	30	180
Glycerol	70	30	220

precision: 0.02 mm). Measurement was performed on the inner diameter, the wall thickness and the height of the 3D-printed rings. Two measurements were performed, the minimal and maximal value of the inner diameter, the wall thickness and the height were reported.

2.5 Statistics

The data was processed in MATLAB R2020a. Mean values and standard deviations are presented for the tensile strength, tensile modulus and dimensional measures. Differences between these values were tested by unpaired t-tests. Values of $p < 0.05$ were considered statistical significant.

3. Results

3.1 Annealing temperature

The average temperatures per annealing method are shown in Table 3.

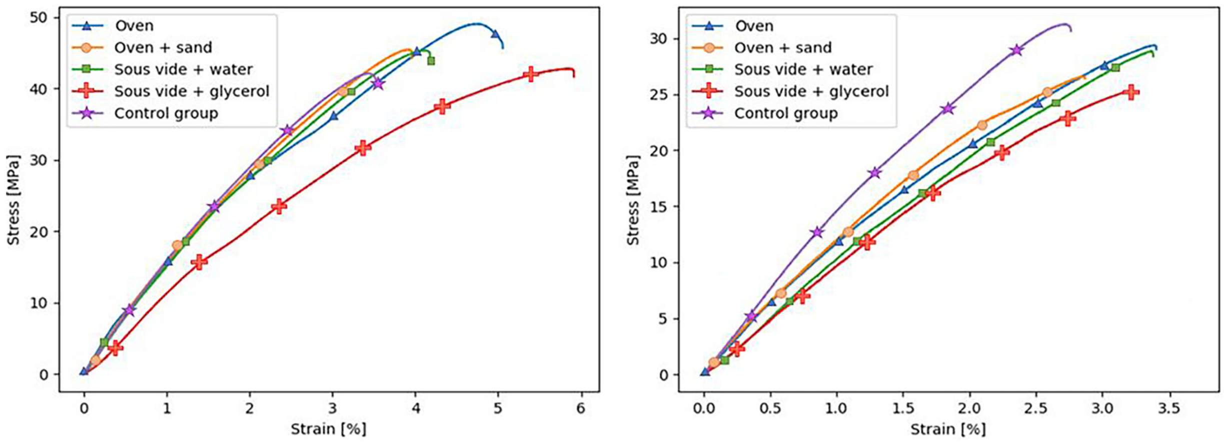
3.2 Tensile testing

Representative engineering stress-strain curves calculated from these tests are shown in Figure 4 for the longitudinally and transversally printed tensile test samples. The mean tensile strength and tensile modulus, their standard deviations and *p*-values are shown in Tables 4 and 5 for each annealing method and the control group.

Table 3 Average temperature and standard deviations per annealing method. Average temperature is shown for annealing of the tensile test samples and the test rings. The temperature was measured using three thermometers of equal distance and was noted every minute. Mean and standard deviations (SD) are based on 90 measurements. When 30 min of annealing time had passed, the tensile test samples and test rings were cooled to room temperature in the oven or liquid

	Temperature tensile test samples (°C)		Temperature test rings (°C)	
	Mean	SD	Mean	SD
Oven	81.0	4.24	81.7	2.80
Sand	82.4	5.85	80.1	5.76
Water	79.9	0.70	80.0	0.94
Glycerol	80.2	0.82	79.9	0.56

Figure 4 Representative engineering stress-strain curves of the longitudinally and vertically printed tensile test samples, for each annealing method and the control group



(a) (b)

Notes: (a) Longitudinally printed samples; (b) transversally printed samples

Table 4 Average tensile strength and standard deviations for each annealing method performed on tensile test samples printed in longitudinal and transversal printing directions (*p*-values are shown for the difference between the control group and the annealing method)

	Longitudinal printing direction			Transversal printing direction		
Tensile strength (MPa)	Mean	SD	*p*-value	Mean	SD	*p*-value
Control group	42.8	0.35		29.7	1.26	
Oven	47.7	1.26	<0.001	27.7	1.44	0.12
Sand	44.6	0.77	0.003	28.1	1.88	0.23
Water	44.8	0.67	0.004	28.5	1.39	0.36
Glycerol	43.1	1.30	0.39	26.2	0.78	0.019

Table 5 Average tensile modulus and standard deviations for each annealing method performed on tensile test samples printed in longitudinal and transversal printing directions (*p*-values are shown for the difference between the control group and the annealing method)

	Longitudinal printing direction			Transversal printing direction		
Tensile modulus (MPa)	**Mean**	**SD**	**p-value**	**Mean**	**SD**	**p-value**
Control group	1600.8	37.9		1402.1	128.4	
Oven	1567.2	44.7	0.33	1142.1	145.0	0.053
Sand	1531.4	135.4	0.36	1182.3	86.4	0.052
Water	1489.7	207.3	0.33	1106.7	14.8	0.012
Glycerol	1224.5	189.9	0.011	1029.1	105.5	0.010

Annealing using an oven, sand and water resulted in a significant increase of 11.4%, 4.2% and 4.7% in tensile strength in longitudinally printed tensile test samples. However, the tensile strength was decreased in all transversally printed tensile test samples, of which only Glycerol annealing had a significant decrease of 11.8%. The tensile modulus was decreased in all longitudinally and transversally printed samples. A significant decrease of 23.5% for glycerol annealing in de longitudinal print direction and a significant decrease for water and glycerol annealing of 21.0% and 26.6% in the transversal print direction.

3.3 Dimensional analysis

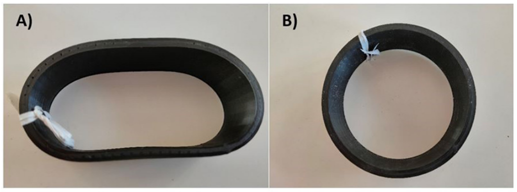
The average minimal and maximal inner diameter, wall thickness and height are shown in Figure 6, before annealing (control) and after each annealing method. Results are shown separately for test rings annealed in horizontal and vertical orientation. Tables with the average values and standard deviations of the dimensional analysis are shown in Appendix (Table A1 and A2). Differences in inner diameter and height for each annealing method had a p-value <0.05 for both the minimum and maximum measurement and for both the horizontal and vertical annealing orientation. Differences in wall thickness all had a p-value >0.05.

Sand annealing resulted in the least deformation for each dimension, with a shrinkage of 2.04% of inner diameter, an increase in wall thickness of 0.46% and an increase in height of 1.99% for the horizontally annealed test rings. This annealing method also showed the least difference in minimum and maximal deformation. This in contrast with the oven annealing of the vertically placed test rings, which resulted in high deformations for each dimension. An example of such a test ring is shown in Figure 5.

4. Discussion

In this study, four different annealing methods were performed on 3D printed tough PLA samples. Tensile tests and dimensional analysis were performed to research the dimensions and changes in strength. Annealing resulted in an increase in tensile strength in longitudinally printed tensile test samples and a decrease in the transversally printed tensile test samples. The tensile modulus had no significant increase in the longitudinally and transversally printed samples. Sand annealing resulted in the least deformation, with a shrinkage of 2.04% of inner diameter, and an increase in height of 1.99% for the horizontally annealed test rings.

Figure 5 Vertically annealed test ring resulting from oven annealing as opposed to a vertically annealed test ring resulting from oven with sand annealing



Notes: (a) oven annealing; (b) oven with sand annealing

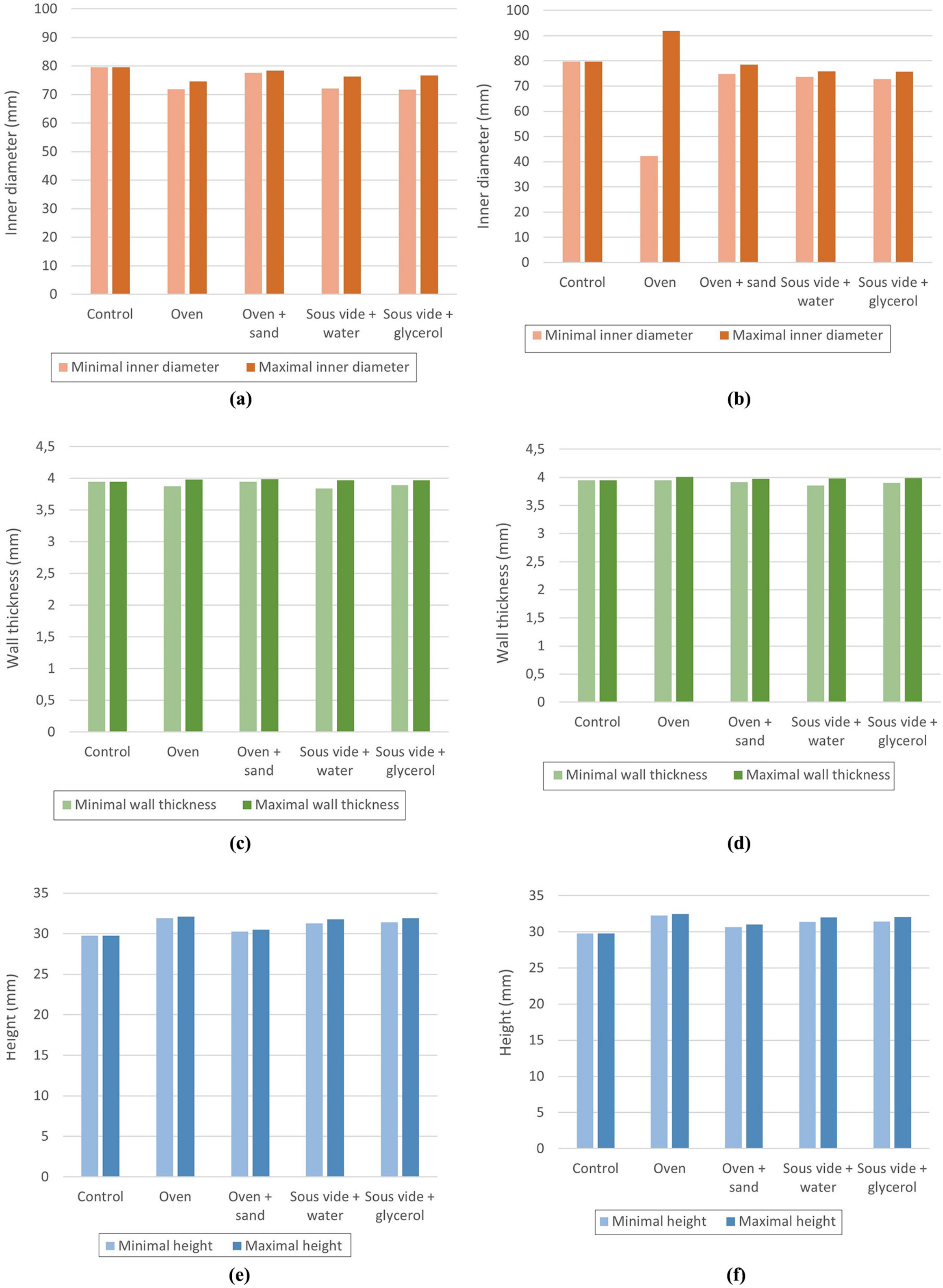
Regarding the different annealing methods, oven annealing resulted in the biggest increase in tensile strength in longitudinally printed PLA tensile test samples. This is possibly caused by a lower heat transfer to the tensile test samples when using air, than when using sand, water or glycerol (de Vree, 2021). As the heat transfer coefficient of air is lowest, the samples will heat up and cool down slower, ensuring an even distribution of the heat through the tensile test samples (de Vree, 2021). Sand and water annealing resulted in a similar increase in tensile strength, while glycerol annealing did not result in a significant increase in tensile strength. The non-significant increase in tensile strength when using glycerol annealing might be caused by deformations of the tensile test samples as mentioned in subsection 4.2. Sand annealing resulted in the least deformation as the tensile test samples had the lowest degree of freedom using this method. This in contrast with oven, water and glycerol annealing, where glycerol annealing failed in preventing warping and bending of the tensile test samples.

4.1 Comparison earlier studies

To the best knowledge of the authors, no previous literature is available on testing annealing on tensile test samples printed in transversal direction.

The decrease in tensile modulus for longitudinally printed tensile test samples is inconsistent with the results found by Wang *et al.* (2019). This study showed a decrease in tensile modulus when annealing PLA for 30 min at 80°C (Wang *et al.*, 2019). However, at different annealing times and temperatures, Wang *et al.* found that the tensile modulus did increase. Therefore, the decrease in tensile modulus might be an effect more coherent with tough PLA than PLA. This insinuates that the decrease in tensile modulus might be caused

Figure 6 Dimensional analysis. Average minimal and maximal inner diameter, wall thickness and height for horizontally annealed test and vertically annealed test rings



Notes: (a) Inner diameter hirizontally annealet test rings; (b) inner diameter vertically annealet test rings; (c) wall thickness hirizontally annealet test rings; (d) wall thickness vertically annealet test rings; (e) height hirizontally annealet test rings; (f) height vertically annealet test rings by heating of the acrylic polymers present in tough PLA, which are not present in PLA.

Butt and Bhaskar (2020) found a 11% increase in tensile strength for longitudinally printed when annealing PLA for one hour at 80°C in a convection oven, this is comparable with the 12.1% increase found in the current study. However, this is opposed to Wang *et al.* (2019), who did not find a significant increase in tensile strength after annealing PLA for 30 min at 80°C. Only Butt and Bhaskar (2020) used comparable tensile test samples and print settings; therefore, it seems that results also highly depend on the design of the tensile test samples.

4.2 Limitations

A limitation of the current study is that the tensile test samples resulting from the sous vide annealing methods deformed slightly. Although the tensile test samples were weighted down with weights, they were not lying flat on the bottom of the sous vide cooker unlike the case with oven or oven with sand annealing. This most likely gave the tensile test samples a higher degree of freedom, causing them to deform. Therefore, the influence of sous vide annealing using water or glycerol on the tensile strength and the tensile modulus might be underestimated. Furthermore, the deformations complicated clamping of the tensile test samples during tensile testing. In two cases, this caused a slip of the tensile test sample in the clamps, as can be seen in Figure 4.

In this study, tests were performed on tough PLA. The effect is not expected to be very different compared to normal PLA; however, this will need to be investigated first. In addition, in this study annealing was performed at 80 degrees and for a period of 30 min. Literature has shown that these are the most ideal temperature and time to anneal PLA (Lam, 2017; Butt and Bhaskar, 2020; Wang *et al.*, 2019; Srithep *et al.*, 2012; Chen *et al.*, 2020). However, the effect of using different temperatures and annealing time might influence the result. We have not done any further research on this, because we believe it will provide minimal improvement in strength in the transversal direction, as annealing shows a reduction in strength instead of an improvement.

4.3 Future research

In future research, annealed tensile test samples could be analysed microstructurally to evaluate differences before and after the annealing process. This has not been done before in previous research and, therefore, could increase the understanding of the influence of annealing on tough PLA. Performing both the annealing methods, tensile tests and microstructural analysis on PLA and Though PLA would enable a comparison of both materials, leading to a more comprehensive understanding of differences between them.

5. Conclusion

Oven, sand and water annealing of longitudinally printed Tough PLA tensile test samples resulted in a significant increase in tensile strength. However, annealing of tough PLA FFF-printed prosthetic sockets is not recommended as this effect could not be seen in the transversally printed tough PLA tensile test samples. An increase in tensile strength in transversal direction is desired as the printed prosthetic socket receives most load in this direction when used. More research is needed towards the strengthening of tough PLA in both print directions.

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Appendix

Table A1 Dimensional analysis. Averages and standard deviations of minimal (min) and maximal (max) inner diameter, wall thickness and height for horizontally annealed test rings

	Inner diameter (mm)				Wall thickness (mm)				Height (mm)			
	Min		Max		Min		Max		Min		Max	
Horizontal	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
Control	79.63	0.26	79.74	0.24	3.95	0.06	3.96	0.07	29.77	0.08	29.80	0.08
Oven	71.83	0.69	74.64	3.15	3.87	0.03	3.98	0.02	31.94	0.33	32.11	0.39
Sand	77.64	0.40	78.37	0.41	3.95	0.01	3.99	0.01	30.24	0.28	30.49	0.19
Water	72.07	0.84	76.37	3.84	3.84	0.02	3.97	0.03	31.25	0.06	31.76	0.17
Glycerol	71.71	0.93	76.65	1.37	3.89	0.03	3.97	0.01	31.39	0.14	31.92	0.16

Table A2 Dimensional analysis. Averages and standard deviations of minimal (min) and maximal (max) inner diameter, wall thickness and height for vertically annealed test rings

	Inner diameter (mm)				Wall thickness (mm)				Height (mm)			
	Min		Max		Min		Max		Min		Max	
Vertical	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
Control	79.63	0.26	79.74	0.24	3.95	0.06	3.96	0.07	29.77	0.08	29.80	0.08
Oven	42.31	1.61	91.90	2.35	3.95	0.02	4.01	0.01	32.25	0.03	32.47	0.08
Sand	74.79	0.53	78.58	0.24	3.91	0.06	3.97	0.04	30.65	0.09	31.01	0.04
Water	73.70	1.22	75.90	0.72	3.85	0.03	3.98	0.02	31.39	0.12	31.97	0.16
Glycerol	72.77	0.65	75.75	0.82	3.90	0.02	3.99	0.01	31.42	0.59	32.03	0.23

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