Preliminary study on the impact of thermal processing on the performances of parts obtained by fused deposition modeling (FDM)

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Abstract. Additive Manufacturing (AM) allows to build complex geometries while generating less waste than conventional processes such as machining. However, in AM processes such as Material Extrusion (MEX), workpieces tend to have low mechanical properties, low dimensional accuracy as well as rough surfaces. Indeed, several pre-processing and post-processing techniques exist to attempt to control these concerns. One post-processing method, namely thermal annealing, is already widely used with metallic parts. However, the influence of this technique has yet to be tested on mechanical properties such as tensile properties, dimensions, and surface roughness for polymer parts obtained by MEX and, particularly, by Fused Deposition Modelling (FDM). This paper aims to determine the relevance of using thermal annealing as a post-process to enhance FDM-obtained parts tensile properties while keeping in mind their dimensional and surface roughness aspects.

Introduction

Context. In the last few years, additive manufacturing (AM) processes have been developed quickly in response to the increasing demand for personalized items [1]. One of the most well-known AM technologies is Material Extrusion (MEX) and, particularly, Fused Deposition Modeling (FDM) because of its low cost and ease of use for industries and private users. This process consists of building a part by successively adding layers of melted material (including polymer, composite, ceramics in thermoplastic matrix). Therefore, some geometries impossible to obtain by conventional manufacturing processes can be achieved thanks to this approach [1].

However, some limitations remain despite the wide use of this technology. Indeed, parts obtained by FDM usually exhibit low mechanical properties, low dimensional accuracy (around 0.1 mm in optimal conditions) as well as rough surfaces [1]. In addition, mechanical properties may vary widely from one part to another because of their dependence on printing parameters such as the printing temperature, build speed, and infill pattern and percentage [2]. If many studies have been seeking to optimize these parameters [2–4], research now tends to explore post-processing solutions to enhance the mechanical properties of polymer parts obtained by AM [5].

One of the possibilities currently studied in terms of post-processing is the thermal annealing of the parts [5]. This technique which is commonly used for metal workpieces consists of raising the specimen temperature after manufacturing before bringing the temperature back to ambient temperature. However, studies regarding thermal annealing applied to AM parts are only starting to emerge.

Literature review. Investigations about thermal annealing of Polylactic Acid (PLA) are still in their early stages. Annealing temperatures for PLA used in literature vary from 60°C in Behzadnasab et al. [6] to 160°C in Jo et al. [7] and in Hart et al. [8]. In fact, lower temperature ranges allow to scan effects of temperatures around the PLA glass transition temperature ($T_g \approx 60$°C). On the other hand, the highest temperatures tested provide information on effects of temperatures reaching above the melting temperature ($T_m \approx 150$°C).

Regarding annealing duration for PLA, intervals of values tested in literature are also quite broad. In Wach et al. [9], samples were annealed for as few as 15 seconds. On the other side of the spectrum, the maximum annealing time (2 hours) was reported in Hart et al. [8] and in Slavkovic et al. [10].

Overall, results provided by the different studies are mitigated. Slavkovic et al. [10] saw an increase of 19% in Young's Modulus and an increase of 30.25% in Ultimate Tensile Strength (UTS). On the contrary, Behzadnasab et al. [6] reported no significant impact on Young's Modulus for the same ranges in terms of annealing temperature and duration.

Others explore the effect of thermal annealing with other types of mechanical tests such as flexural [8, 9, 11] and torsion tests [12]. According to Hart et al. [8], semi-crystalline materials like PLA will remain in an amorphous state if they are cooled quickly. On the other hand, if they are cooled slowly (10°C/min or slower), a crystalline domain will form, leading to an increase in toughness. In Wach et al. [9], the main topic discussed is the annealing time with respect to the annealing temperature. For instance, it is demonstrated that the crystallinity degree does not change at a temperature too close to the glass transition temperature T_g (in the study, at 60 and 70°C). Therefore, it leads to very few effects on the mechanical properties.

An interesting point to note is that devices are used in a couple of studies to hold the samples during annealing. For instance, a mold is used in Rane et al. [13] to avoid distortions caused by thermal processing. Similarly, the use of a custom fixture is mentioned in Hart et al. [8]. In a previous study, Hart et al. attempted to anneal one ABS sample for 18 hours at 175°C without using the annealing fixture [14]. As a result, the sample was distorted and became unusable for flexural testing. According to their analysis, the deformation resulted from the release of residual stresses from the initial manufacturing process and the polymer's own weight. The hypothesis of residual stresses is also mentioned by Butt et al. [15] and Behzadnesab et al. [6] in their respective research although they do not mention holding their samples during annealing. The custom fixtures used are different depending on the studies since they were made specifically by the researchers.

Goal and motivations of the study. This study aims to determine the relevance of using thermal annealing as a post-process to enhance FDM-obtained parts tensile properties while keeping in mind their dimensional and surface roughness aspects that have not been studied so far.

Material and Method

Part printing. The parts were printed from 2.85 mm-diameter filaments of blue Ultimaker Polylactic Acid (PLA). The printer used was an Ultimaker S3 with AA 0.4 mm print cores. A light mist of 3DLAC was applied on the build plate before each print to avoid warping. Printing parameters were chosen according to the manufacturer recommendations, given in the Cura 4.13.1 software for the "Fine" resolution. The initial layer height was modified from the default parameters (0.2 mm) to avoid creating a shell effect. The infill pattern was changed to "Lines" and oriented in the tensile test traction direction. Other printing parameters are given in Table 1.

Table 1 - Printing parameters

Layer height (including Initial layer height)	0.1 mm
Infill density	95%
Infill Line Directions (including Top/Bottom Lines)	Traction direction
Build orientation	Flat (XY)
Nozzle temperature	200°C
Build plate temperature	85°C
Printing speed	70 mm/s

Surface roughness and dimensions assessment. A Wurth caliper was used to measure specimens with a scale of 0.05 mm. All dimension measurements were evaluated three times and the average value of the three measurements was kept as data. A Carl Zeiss Handysurf E-35A was employed to evaluate the arithmetic surface roughness R_a of the specimens top and bottom faces (corresponding respectively to the last and first layers printed). These values were measured following the guidance of ISO 4288 [16]. Measurements of surface roughness and specimen dimensions were assessed before and after thermal annealing.

Thermal annealing. Annealing was conducted using a Binder FD115 E2 Proofer. The aim was to assess the influence of annealing temperature and duration on tensile properties, sample dimensions, and surface roughness. Therefore, a total of 10 conditions were employed. 9 conditions resulted from the combination of 3 levels of annealing temperature and duration while the last one consisted of leaving the samples unannealed as a control condition. Each condition was repeated over 5 samples. Intervals of temperature and dimensions were chosen according to literature. The thermal annealing parameters used in this study are given in Table 2. The proofer was preheated at the given temperature before placing the samples for annealing.

Table 2 - Thermal annealing parameters

Control specimens: Category 0			
	100°C	120°C	140°C
10 min	Category 1	Category 2	Category 3
35 min	Category 4	Category 5	Category 6
60 min	Category 7	Category 8	Category 9

At the end of the annealing time, the specimens were left in the fixture until they reached room temperature, which lasted around an hour. Indeed, according to Hart et al. [8], for semi-crystalline materials like PLA a slow cooling rate (10°C/min or slower) has a beneficial impact on the crystallinity and therefore on mechanical properties of the material.

Tensile testing. A Zwick Roell Z2.5 bench was employed with an Xforce 2.5 kN load cell and Type 8297 pneumatic grips from the same manufacturer. Test speed was set to 3 mm/s with respect to the ASTM D638 standard [17]. Type IV of this standard geometry was employed (Figure 1). testXpert II was the software used to retrieve data in terms of stress σ [MPa] and strain ε [%]. Data was later processed to compute Young's Modulus *E* [MPa], Ultimate tensile strength UTS [MPa] and the elongation at break *A* [%]. Tensile tests were performed on 5 samples per thermal annealing category. Therefore, mean values and standard deviation of the different tensile properties cited were also computed.

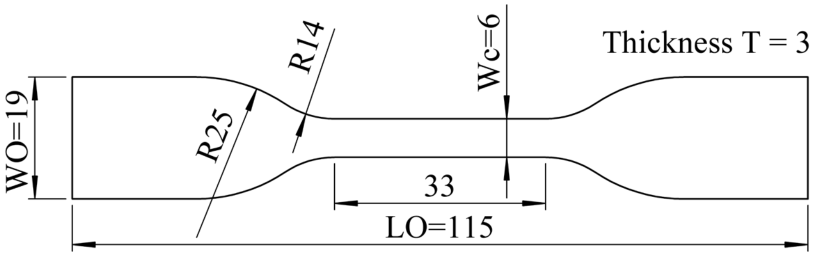

Figure 1 - Specimen dimensions in mm from ASTM D638 Type IV [17]

Results and Discussion

Thermal annealing fixture. As mentioned in the literature review, some kind of mold or fixture may be employed to hold specimens during annealing. Indeed, studies [13, 14] reported geometry distortions because of the heating process. Consequently, samples were first annealed without any kind of device to hold them to assess the distortion scope in this case. Two batches of 5 specimens were annealed without fixture and with two different placements. The first batch was placed in its build orientation (first layer printed at the bottom, last layer printed at the top, Figure 2). The second batch was placed upside-down compared to its build direction (first layer printed at the top, last layer printed at the bottom, Figure 3).

The specimens did not keep their original shape as expected from literature. Indeed, no matter the annealing orientation of the samples, the latter curled towards the first layer printed (Figure 2 and Figure 3). This phenomenon may be due to residual stresses caused by the printing process. Particularly, it can be induced by the temperature gradient between the bottom layers kept heated by the build plate and the top layers being printed.

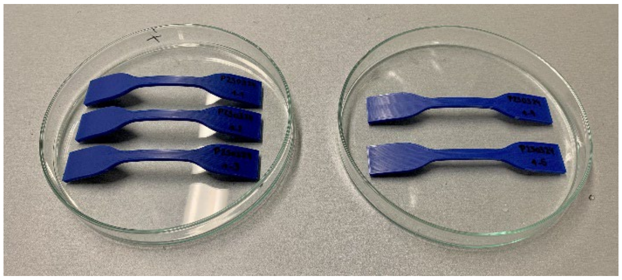

Figure 2 - Samples annealed on their build plate side

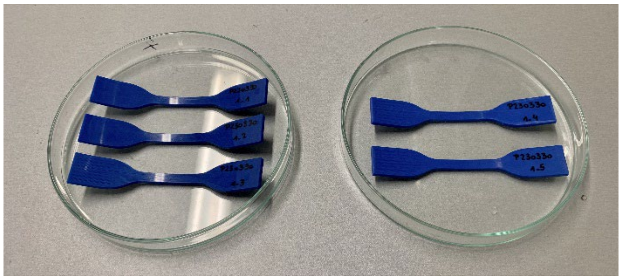

Figure 3 - Samples annealed on their top side

Other than the curvature making the length of the samples not measurable, the specimen dimensions, such as the width of the ends (WO, Figure 1) and narrow part (Wc, Figure 1) as well as the thickness (T, Figure 1), remained within the tolerance interval allowed by the tensile test standard ASTM D638 [17].

Following these observations, a fixture (Figure 4) was designed to maintain the samples flat during annealing. Compared to fixtures showed in literature [13, 14], the design used for this study also intends to maximize the heat exchange by using thin metal plates ensuring good conductivity as well as the largest contact surface possible. As a result, this fixture is made from two aluminum Al 6082 T6 plates of 5 mm of thickness where screws were evenly distributed (spaced by 44 mm) so that each sample was held in similar conditions. Therefore, this fixture was used to perform thermal annealing for the whole study. The nuts were screwed by hand, therefore the torque applied on them was not specific. A perspective for this study could be to perform it again using a consistent torque on the nuts.

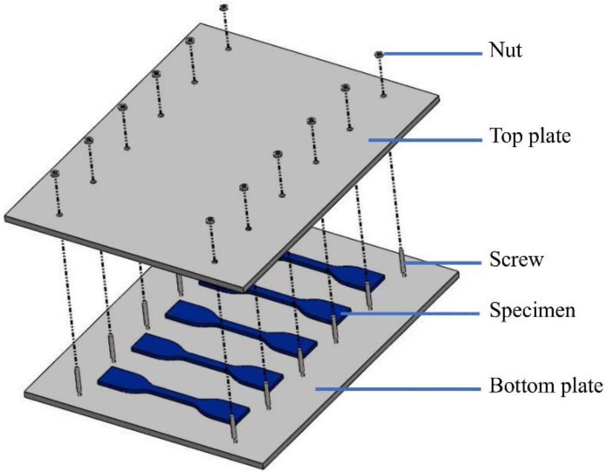

Figure 4 - Fixture used to anneal the specimens

Thermal annealing. In terms of Young's Modulus, specimens annealed for 10 minutes (Category 1 through 3) show a decrease in property values. Overall, a trend seems to exist as Young's Modulus increases with annealing temperature for the same annealing duration (Figure 5). These results need to be put into perspective as the maximum increase in Young's Modulus is only about 6.5%.

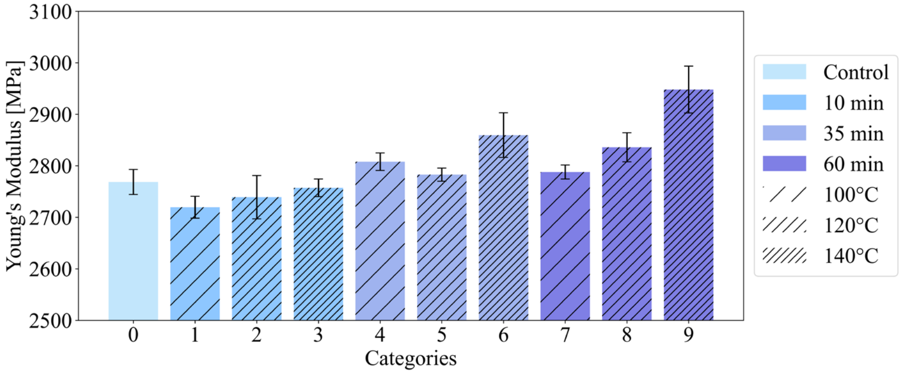

Figure 5 - Young's Modulus comparison between categories

Regarding the UTS, it does not seem to follow any specific trend (Figure 6). In addition, all the standard deviation intervals of annealed samples overlap the standard deviation of the control specimens.

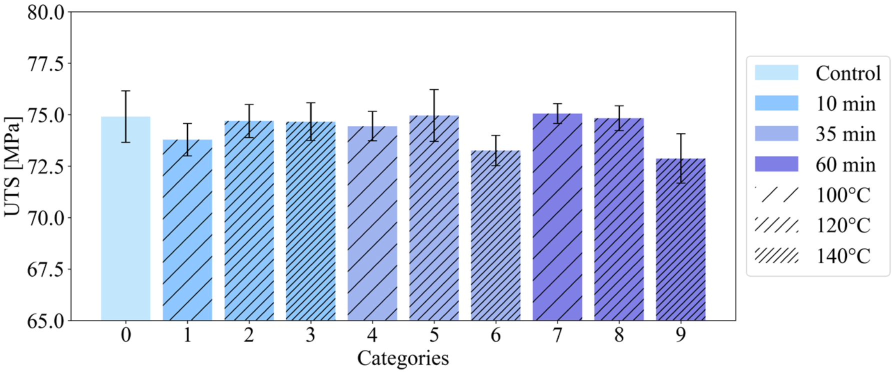

Figure 6 - UTS comparison between categories

As for the elongation at break, it appears to follow the opposite trend to that of Young's Modulus (Figure 7). Indeed, 10 minutes of thermal annealing seem to have a positive effect on the elongation at break. For instance, it leads to a 34.7% increase. The graph also exhibits that the elongation at break is decreasing with the annealing temperature for the same annealing duration. This correlation between elongation at break and Young's Modulus is not surprising. Indeed, materials generally tend to have a more brittle behavior when their plastic properties increase.

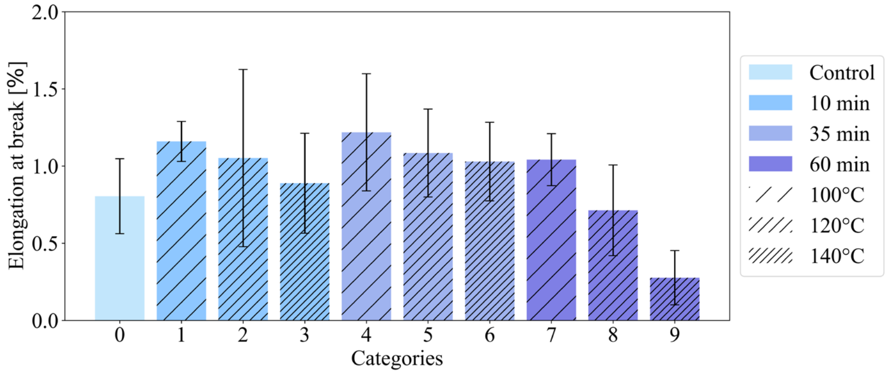

Figure 7 - Elongation at break comparison between categories

Dimensions assessment. Dimensions were compared batch per batch to avoid the influence of any disparities between batches. As a reminder, each batch contained five specimens and was subjected to the categories number from 1 to 9 (Table 2).

For each dimension type (LO, WO, Wc and T, Figure 1), the difference between the mean value for one batch before annealing and the theoretical value was compared to the mean measured value for one batch after annealing and the theoretical dimension. These differences were also compared with the ISO 2768-1 tolerances classes [18]. As a reminder, the intervals allowed by tolerances classes depend on nominal linear dimensions. Consequently, the class intervals differ for LO and WO compared to Wc and T.

Overall, the mean LO, WO and Wc were decreased by annealing according to the graphs (Figure 8) although they tend to remain in the same tolerance class due to the great standard deviation. In terms of thickness T, no specific trend was established although most samples appear to keep their initial thickness. However, the lower deviation regarding thickness could be linked to the fixture use since it does not allow samples to expand in this direction. As a reminder, samples needed to be maintained in a fixture to avoid warping and large distortions. In fact, this step is necessary to obtain usable samples with respect to the ASTM D638 tensile test standard [17]. It should be noted that the overall decrease in specimen dimensions could be linked to the decrease in the initial voids between layers thanks to thermal annealing. In fact, samples were printed with an infill density of 95% (Table 1) making them porous. Reduction of porosity is, indeed, generally a way of increasing mechanical properties.

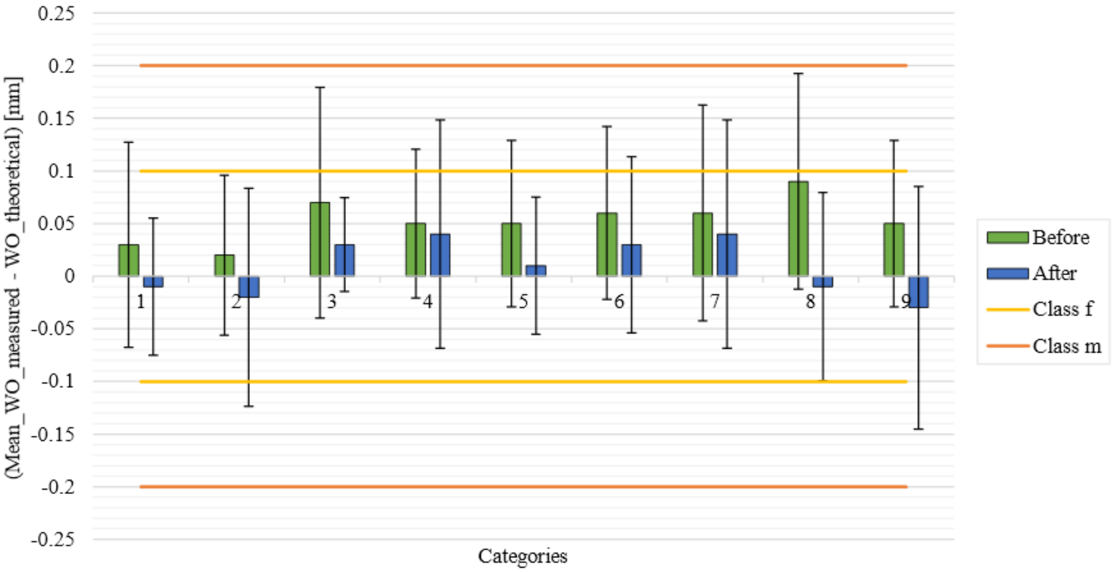

Figure 8 - Example of dimensions comparison graph before and after thermal annealing, here for WO

In terms of dimensions tolerances after annealing, the samples exhibit dimensions that fall within the medium (class m, for WO and T) or coarse (class c, for LO and Wc) tolerance classes of ISO 2768-1. This should be considered in addition to the printing tolerances although measured values stay in the same order of magnitude before and after annealing.

As for the relative standard deviations, trends were not observed for LO and WO. On the contrary, thermal annealing occurs to have a negative impact on the relative standard deviation of Wc which is enlarged at least by 6.5% and up to 23.9%.

Part surface roughness. The arithmetic surface roughness R_a was considered. Values of R_a measured on samples were compared to classes based on the intervals of R_a mentioned in ISO 1302 [19]. For each batch of 5 samples, the mean R_a over the batch was compared before and after annealing. In Figure 9, the graph highlights the fact that thermal annealing tends to decrease the surface roughness of samples' top face. For instance, the general trend is for the mean surface roughness to go down a class after annealing. The only case for which the conclusion cannot be applied is samples belonging to Category 1 which corresponds to the shortest annealing time and lowest annealing temperature.

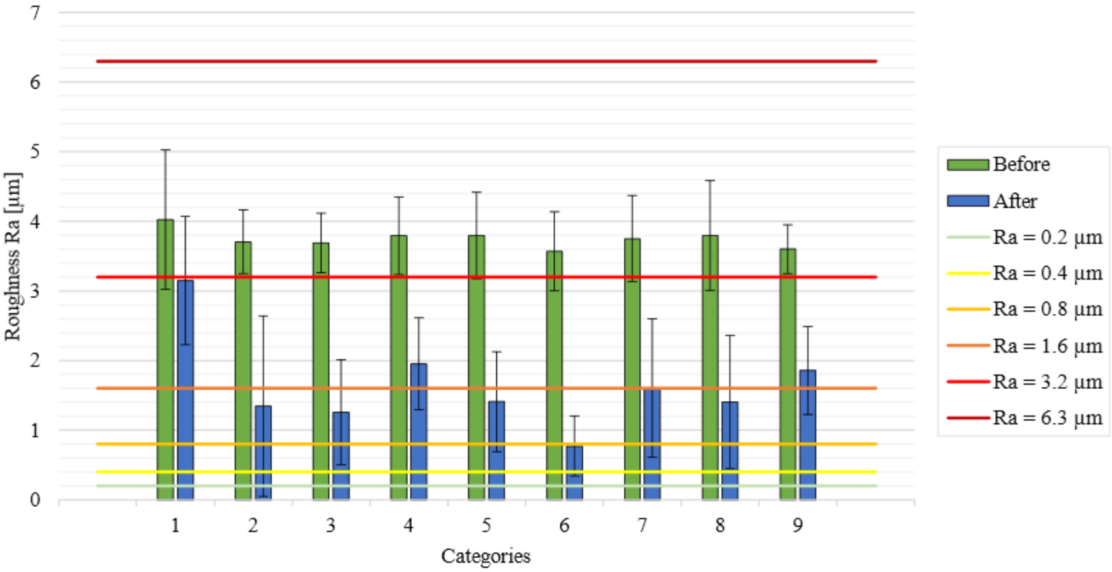

Figure 9 - Comparison of the top face roughness before and after thermal annealing

For the bottom surface, the effects of thermal annealing on surface roughness are inconsistent. For some categories, surface roughness decreases while, for others, it increases marginally or significantly. However, even for the most important surface roughness changes, values for each batch stay in the same order of magnitude and therefore in the same class before and after thermal annealing.

Summary

Conclusion. The main findings are:

- Distortions appear if the parts are annealed without being held. Therefore, a fixture needs to be designed and used. This need for a fixture during thermal annealing could limit the use of this post-process. Indeed, one of the main advantages of AM is to allow the manufacture of complex shaped parts for which it is not possible to create a mold.
- Annealing tends to increase Young’s Modulus of the PLA if the annealing temperature is above its crystallization temperature, with a maximum increase of 6.5% compared to unannealed parts.
- For the elongation at break, the progression is opposite to the one showed by Young’s Modulus with a maximal increase in elongation at break of 34.7%.
- In terms of dimensions, overall, the length LO and widths (of the ends Wo and narrow parts Wc of the specimens) appear to be decreasing while the thickness T remains unchanged in most cases.
- As for the surface roughness of the parts, the top and bottom surface roughness need to be addressed separately. The surface roughness of the samples’ top face occurs to be reduced by annealing although staying in the same surface roughness class, while no significant change appears on the bottom face.

Perspectives. These are the main perspectives of the work:

- Finding optimized annealing parameters based on the most promising parameters found in this study.
- Research could be lead on ways to avoid the build-up of residual stresses during printing such as specific printing parameters or conditions to avoid distortions during thermal annealing.

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