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Effect of Normalizing on the Tensile Strength, Shrinkage and Surface Roughness of PLA Plastic

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Abstract

The two major methods of altering the properties of a material include alloying and heat treatment processes. Out of the several heat treatment processes, normalizing is one in which air is used as a cooling media after the material has reached its recrystallization temperature. This project explores the effects of normalizing on the tensile strength of a chosen material, in this case as PLA (Polylactic Acid) plastic. The research also extended to investigate the effects of normalizing on surface roughness and shrinkage. The investigation follows a strategic and systematic approach whereby the methodology used to conduct the experiments gave a quantitative output. In general it was found that the heat treatment makes significant impact on the chosen property.

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Keywords: Normalizing; Fused Deposition Modeling; Tensile Strength; Shrinkage; Surface Roughess

1. Introduction

1.1. Normalizing

Normalizing is a three phase heat treatment process in which the material is firstly heated above its temperature of recrystallization / critical temperature (usually in a furnace) after which the material is soaked in the temperature

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over a sufficient period for transformation to occur and finally it is taken out from the furnace and allowed to cool in atmosphere / room temperature. The objectives of normalizing include relieving internal stresses, improving toughness and refining the grain size.

When dealing with semi-crystalline thermoplastics such as PLA plastic, instead of using the recrystallization temperature, the glass transition temperature of the thermoplastic is used [1]. The glass transition temperature is a temperature above which many polymers and inorganic glasses no longer behave as brittle materials. They gain a considerable amount of ductility above the glass transition temperature [2]. In present investigation, the normalization process is carried out above the glass transition temperature range. Another similar heat treatment process which uses the glass transition temperature of thermoplastics is called annealing, which is performed at a temperature slightly below the glass transition temperature [3]. The purpose of annealing plastic polymers is to create a higher degree of crystallinity [4].

The degree of crystallinity is essentially a function of the heat treatment temperature and duration of the process. It was reported elsewhere that an experiment was conducted to investigate the degree of crystallinity in injection-moulded and annealed PLA specimens under different annealing times and temperatures [5]. It was found that at annealed temperature of 65 $^{\circ}C$, work material reached 43% crystallinity in 31 hours, however when annealed at 80 $^{\circ}C$, the work material reached its highest crystallinity of around 49% in 30 minutes. The maximum crystallization of the PLA samples occurred around 48% - 49%. The investigation also found that the cold crystallization peaks had decreased with an increase in annealing time. This meant an enhanced PLA crystallization. The same degree of crystallization can be achieved by either annealing for a very long period of time or annealing at a higher temperature. It is noted from the literature that not much work had been explored using normalizing was the driving factor to take up the present investigation.

1.2. Fused deposition modelling (FDM)

FDM is a 3D printing procedure that deals with the use of thermoplastic materials. The printing of the molten material is done layer by layer, on top of the previous layer as it fuses almost instantaneously after the thermoplastic has left the nozzle. Once a layer is fully printed, the printer platform is lowered by a fraction. The process is a computer controlled through which variables such as printing temperature or the speed of the head can also be controlled to form an interrupted plane without stringing between intersections [6].

1.3. Mechanical characterization

Surface roughness is a physical property of a material which can be found by measuring the deviations in the direction of the vector normal to the real surface in its ideal form. Large deviations mean high surface roughness whereas low deviations indicate a smoother surface. High roughness is often undesirable in manufacturing firms. As for 3D printing techniques such as FDM, the surface roughness is quite difficult to control. There are various factors responsible for surface roughness in FDM. Some include the distortion effect of successive filaments due to melting, surface angle, stacking and overlapping between layers [7]. Surface roughness testers, also known as profilometers are used to find out the exterior roughness of materials such as metal, wood or plastic.

Tensile tests are mandatory to understand the characteristics of different materials and their behaviour under tensile load. It is one of the most fundamental tests done by engineers as it helps in attaining important information about properties associated with a material. Such properties can aid in design of engineering structures, as well as developing alloys better suited to a certain application. In the tensile testing procedure, samples are exposed to a controlled tension until failure occurs. A common phenomenon that occurs in tensile tests is necking. Necking is when one region deforms more than others and a large local decrease in the cross-sectional area occurs. In a tensile test, “the true strain reaches its maximum value at the smallest cross-section in the necked region” [8]. The properties which can be directly measured include the breaking strength, ultimate tensile strength, reduction in cross sectional area and maximum elongation. The properties aforementioned can be used to calculate Young’s modulus, yield strength, Poisson’s ratio and strain hardening characteristics.

1.4. Effects of process parameters in rapid prototyping

Huang et. al., studied the effect of shrinkage in stereolithography 3D printing technique. It explains the modeling and predicting the part shrinkage technique thereby deriving a shrinkage compensation plan to achieve dimensional accuracy [9]. Ahn et. al., discussed a new approach for formulation surface roughness of FDM parts. A greater detailed approach was developed by studying key variables (such as surface angle) that influenced the surface roughness. Parts were fabricated through FDM to verify the proposed surface roughness expression. Through comparison of the measured data and computed data, the validity was proved [7]. Hwang et. al, studied the viscosity of ABS material that found to be decreasing by increasing the printing temperature. To add on, at different printing temperatures, the viscosity of ABS followed a relationship between the print temperature and the tensile strength. Additionally, it was assured that the tensile properties of the end product was affected by the viscosity of ABS whilst extrusion; thus upon decreasing the viscosity of ABS, the tensile strength and strain were found to be reduced [10]. Takayama et. al., found that the softening heat treatment process improves mechanical properties, such as bending modulus and strength. This is due to the crystallization of the PLA occurs while annealing that subsequently strengthens the structure. It was further explained that the embrittlement caused in PLA while annealing substantially decreases the type I fracture energy of work material [11]. Srithep et. al., analysed the mechanical performance and crystallinity of PLA plastic through injection molding with the influence of annealing temperature and time. Through the XRD technique and DSC methods it was ascertained in the study that effect of crystallinity greatly depends on the time and temperature; subsequently found that the mechanical strength and heat resistance can improve by improving the overall crystallinity [12]. Harris and Lee were also observed a similar relationship between mechanical performance of PLA and the material crystallinity. However, it was accomplished by "addition of nucleating agents, post-annealing of injection molded specimens, and direct enhancement of crystallinity by injection molding into a preheated mold". This gave an enhanced flexural stiffness, strength, and HDT ('heat deflection temperature', or 'heat distortion temperature') [13].

The present investigation was carried out because it shows potential for significant contribution especially in terms of practical applications. Examples of such applications include "PLA drainage material used in construction ground works to reduce or eliminate hydrostatic pressure". PLA drainage material has good workability for soft ground with sufficient permeability and tensile strength [14]. The effects of normalizing on surface roughness are relevant on the microscale level. This comprises of striations arising from microscale roughness inside the print nozzle [15]. As for the case of shrinkage, it is applicable to almost all processes which involve normalizing PLA filaments as it affects the desired dimensions of the part.

2. Methodology

2.1. 3D printing

The test samples were designed in SolidWorks computer aided design software (2014 version) and saved as a ".stl" file. The printing specifications were documented which defined the print temperature and % infill using the MakerBot software. The 3D printing process was accomplished using a MakerBot Replicator 3D printer, which is shown in Fig. 1. As each sample was printed, the print temperature was labelled on it with a marker. This is to differentiate between the samples printed temperature as it is almost indistinguishable visually. The PLA plastic chemical composition is explained elsewhere [16].

The tensile test 3D printing specifications are shown in Table 1, this test was based for plastic tensile test therefore a standard sample size was chosen to be experimented. The standard used for the experiment was ISO 527-1:2012 [17] and is shown in Fig. 2.

Table 1. Printing specifications for tensile test samples.

Printing specifications	Details
Material	PLA plastic
Percentage infill density	100%
Infill pattern	Diamond infill
Print temperatures	215 °C and 240 °C

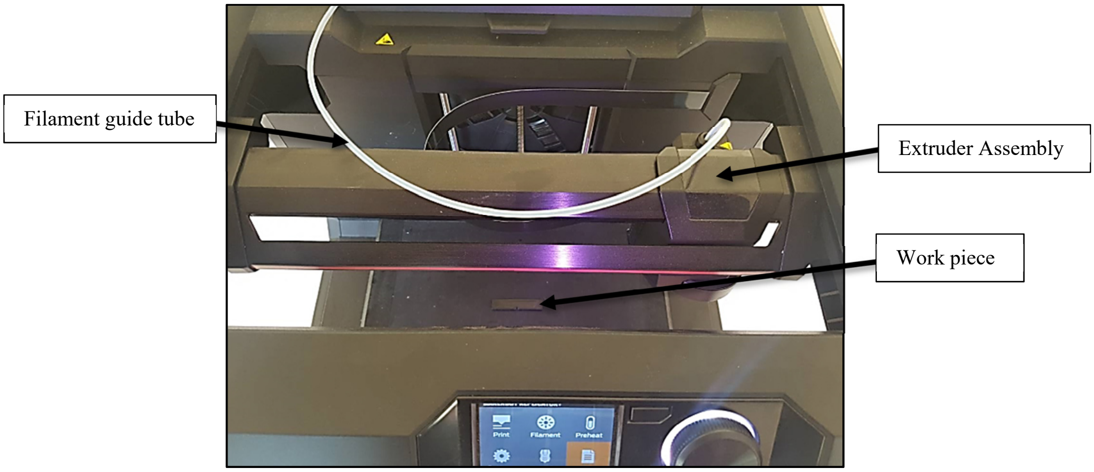

Fig. 1. FDM printing using MakerBot replicator (Model: PABH65).

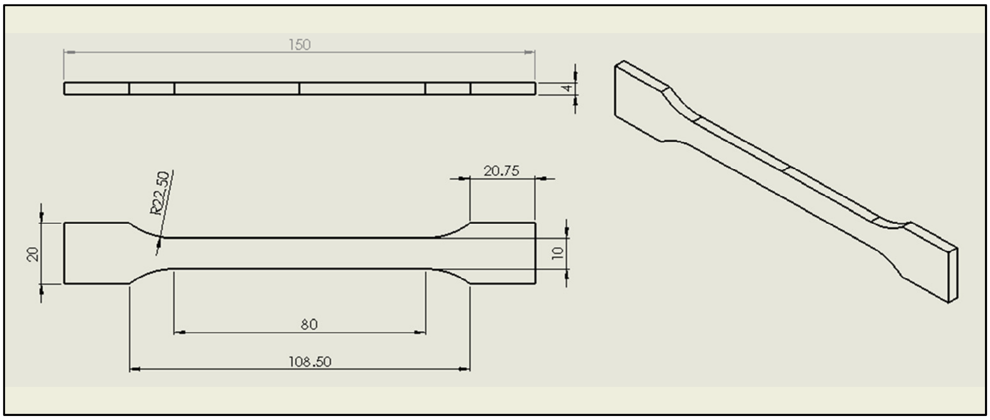

Fig. 2. Tensile test sample dimensions in millimetres.

2.2. Shrinkage test methodology

This test was carried out on the samples that are produced in two different temperatures that underwent with and without the heat treatment process. The measurements of all the three dimensions such as length, width and thickness are made using a digital Vernier Caliper with the dimensional accuracy of 0.01 *mm*. The objective was to see the % change in the dimensions after normalizing.

2.3. Surface roughness test methodology

The surface roughness test was carried out using a surface roughness tester (Mitutoyo SJ.201P/M) on a tensile sample itself. For which, a wooden base was made to support the sample and prevent it from moving while the surface roughness tester was being used on it. The complete set up is shown in Fig. 3. The base was a simple wooden structure with a screw inserted in the side which when tightened, would hold the sample tightly and prevent it from moving and interfering with the experiment. After this, the sample was placed in the support and the screw was tightened. The height placement of the surface roughness tester was adjusted by fine-tuning with the height adjusting screws. Once perfectly aligned the power supply was turned on and the surface roughness test was carried out. Three trails were taken and the average value was taken in the calculations.

Fig. 3. Surface roughness measurement set-up.

2.4. Tensile test methodology

The tensile test was carried out using the Griffin tensile testing machine. A total of four samples were tested. These included the samples at 215 °C and 240 °C which had not undergone normalized heat treatment. The remaining two were the 215 °C and 240 °C samples which went through normalized heat treatment process. The necessary dimensions of the specimen were noted. The dial and counter was calibrated before conducting the experiment. The lever that is used for applying load on the specimen is rotated in the intervals of two. This process was continued until fracture while the gauge deflection was noted at every two intervals. Similarly, this process was carried out on the other three samples.

2.5. Normalizing methodology

The normalizing process was carried out using an electric muffle furnace. The samples that were to be normalized were placed inside the furnace. From here, the temperature was set to 80 °C, which is higher than the glass transition temperature of the PLA plastic that is 60 °C [18]. The samples were heated for one hour after which the samples were taken out to be slowly cooled down (room temperature cooling) for 40 minutes.

3. Results and discussion

3.1. Shrinkage

The shrinkage is measured in terms of overall change in dimensions. The tensile samples are considered to observe the percentage of shrinkage. Fig. 4 indicates the effect of normalizing on the test specimens at two different temperatures at which the samples are printed. The parameter shown in Fig. 4., is the length of the test samples. The actual dimension set in SolidWorks was 150 mm, the experiment shows that for the sample printed at 215 *°C* undergone 7.15% decrease in length after normalizing process. Whereas the test specimen printed at 240 *°C* undergone 7.32 % decrease in length after the heat treatment process. The calculation for percentage of change in dimension is made using the Eq. (1).

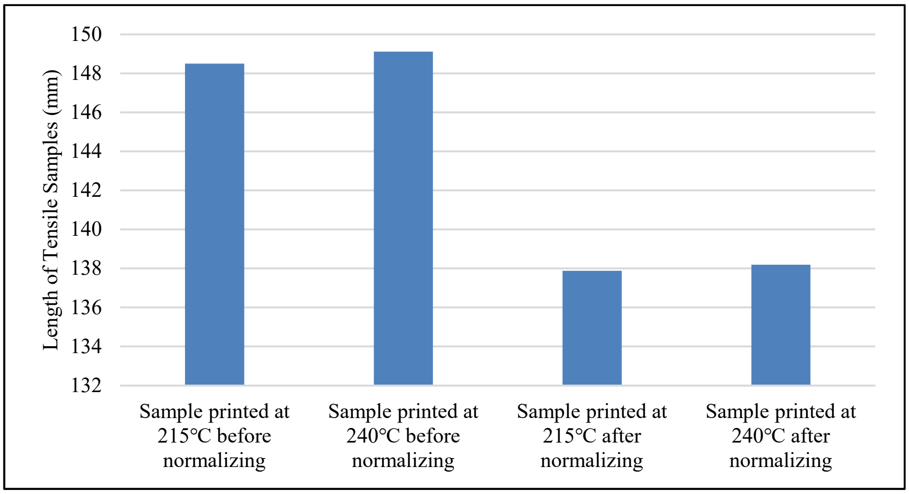

Fig. 4. Effects of normalizing on the length of the test samples.

$$\%\Delta L = \frac{Y - X}{X} \tag{1}$$

Where Y is the length after normalizing, X is the length before normalizing process and ΔL represents the change in length after the normalizing process. Sample calculation for % change in length for the test sample is shown for sample printed at 215 °C.

$$\frac{148.5 - 137.88}{148.5} \times 100 = 7.15\%$$

Similarly the changes in dimension on width of the test samples were observed, which is portrayed in Fig. 5. It is interesting to note that the in place of shrinkage was expected, the expansion took place. As it can be observed from the Fig. 5., the percentage of expansion after heat treatment for samples printed at low temperature is 0.307% whereas for high temperature is 0.88%. As compared to the length wise shrinkage, the expansion of width is much lower in amount. This is because the pattern used for producing the test sample is diamond infill. This means that there the presence of deliberate voids in the structure, upon thermal relaxation particles fuse together. It is also observed that the thickness measurement of the sample before heat treatment is 4 mm and after the heat treatment the measurements shown almost nil variation irrespective of the temperature in which it is printed. The original test samples before and after the heat treatment process is shown in Fig. 6, in which the samples are marked by mistake as annealing however the process carried out is normalizing.

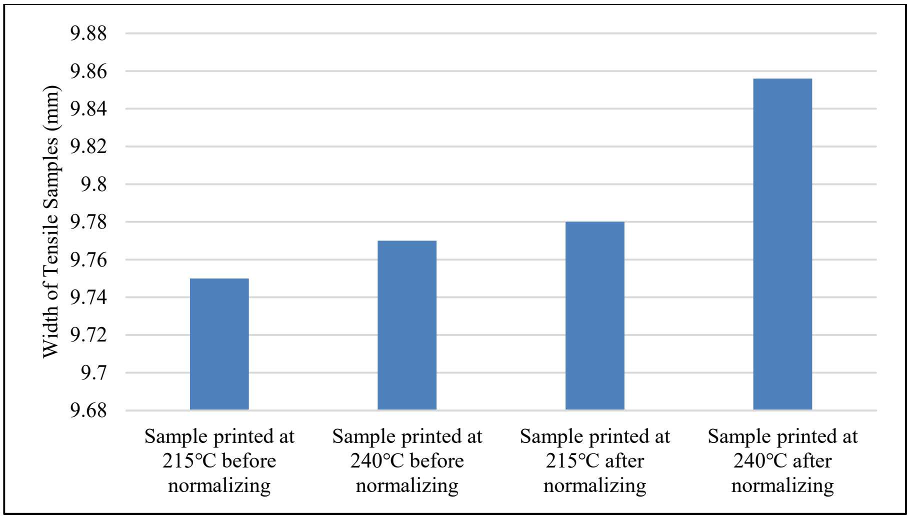

Fig. 5. Effects of normalizing on the width of the test samples.

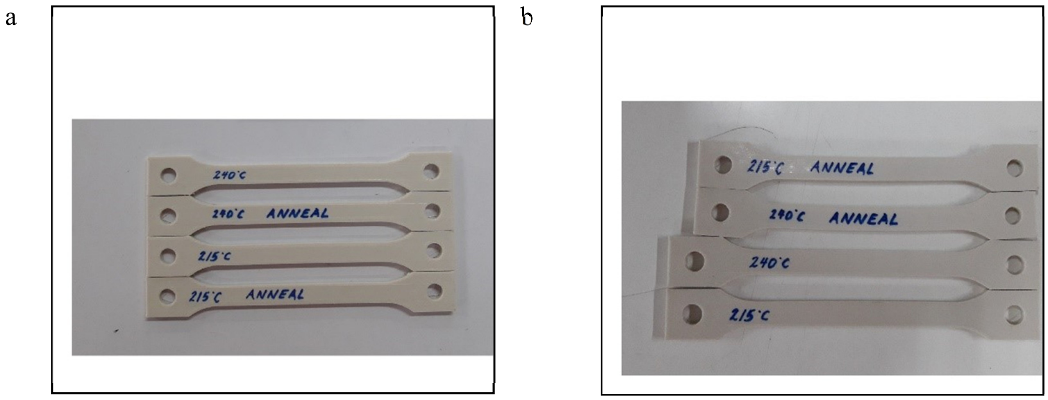

Fig. 6. (a) Samples before heat treatment; (b) after heat treatment.

The results obtained for length, width and thickness of the test samples are utilized to calculate the overall change in dimension and it is noted to have shrinkage after the heat treatment process. The percentage shrinkage for the sample printed at low temperature is 6.86 and for the high temperature print is 6.51%. Therefore it can be concluded that percentage shrinkage for the low temperature print out is higher than the high temperature print. There are two major possible causes for the shrinkage. Firstly, the heat treatment induces the internal structure to better fuse together / molecular relaxation [19]. The second factor is while heat treatment the voids present in the structure gets infilled. Therefore for the applications that involve bit elevated temperature the solid infill print should be chosen in place of diamond or honeycomb structure, although the later structure preserves enormous amount of material for producing product.

3.2. Surface roughness

The sample printed at 215°C had 95.09% increase in surface roughness after normalizing heat treatment process while the sample printed at 240°C had a 75.97% increase in surface roughness, the results can be referred in Fig. 7. Based on the results, it was found that there was a significant increase in surface roughness after normalizing. The major cause of this is set normalizing temperature. Similar to annealing, the normalizing temperature causes the grain structure to fuse together and to form new boundaries [20]. It can be said that the surface roughness properties and crystallization of the normalized samples are strongly dependent on the conditions set for normalizing at it was proven for other heat treatment process elsewhere [21].

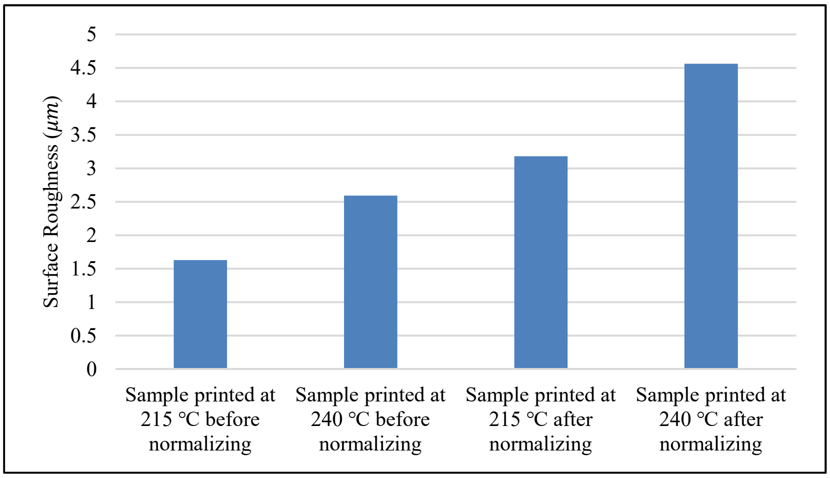

Fig. 7. Surface roughness before and after normalizing process.

3.3. Tensile strength

These results show that there was a 35.71% increase in tensile strength for the sample printed at 215°C after the normalizing process. This meant that it was able to withstand more load before failure. As for the sample printed at 240°C with normalizing, there was 11.25% increase in tensile strength. The results are shown in Fig. 8., and it is proved that the normalized samples had developed a greater internal resistance to applied load. Normalizing helps to improve the crystallinity of materials, eliminate coarse grain structure and relieve internal stresses. Furthermore, similar to annealing, normalizing also affects the grain boundaries [22]. Fundamentally, the existing structure (crystals) of the PLA plastic used in the experiment, after experiencing normalizing heat treatment process, fused together better to form stronger crystalline structure. This in return gave a greater resistance to the applied external load.

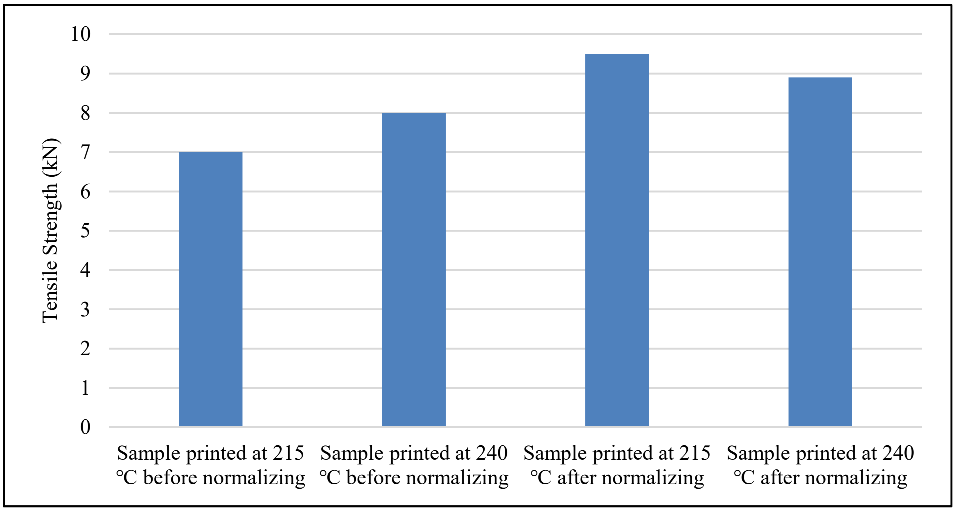

Fig. 8. Tensile strength of the samples before and after normalizing.

4. Conclusions

The standard test specimens are produced to conduct tensile strength, shrinkage property and surface roughness characteristics on PLA plastics that are produced through fused deposition modelling, which had undergone a heat treatment process. The major findings from the present investigations are:

- The tensile samples were analysed for shrinkage analysis, it is observed that length undergone substantial reduction, whereas the width of the specimen undergone a very minor expansion; however the thickness shows no changes in dimensions literally. Thus overall it was found to undergo shrinkage; in particular the specimen produced under low temperature underwent higher shrinkage than the high temperature specimens.
- The heat treatment particularly the normalizing process is found to increase the tensile strength of the specimens irrespective of the printing condition, however the low temperature sample shows relatively higher tensile strength after heat treatment process as compared to high temperature specimen.
- Almost a similar observation is found that the heat treatment process did affect the surface roughness substantially however the impact was less for the sample that produced under low temperature.

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