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# Heat Treatment and Its Effect on Tensile Strength of Fused Deposition Modeling 3D-Printed Titanium-Polylactic Acid (PLA)

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***Abstract*** **-** Titanium is a biocompatible metal commonly applied in biomedical fields such as bone and dental implants. Recently, the produced titanium-Polylactic Acid (PLA) filament for 3D printing Fused Deposition Modeling (FDM) technique is easier to operate and affordable. This filament contains less than 20% PLA, which is also biocompatible but hydrophobic and capable of producing inflammation of the surrounding artificial living tissue. Therefore, a heat treatment is needed to reduce or even eliminate PLA. The research aimed to optimize the mechanical properties and biocompatibility of titanium-PLA filaments through heat treatment, demonstrating significant advancements in 3D printing applications for biocompatible materials. A Thermogravimetric Analysis (TGA) was carried out to find out the right temperature for reducing PLA levels. Specimens were heat treated with four temperatures at 100°C, 160°C, 190°C, and 543°C, and two holding times of 60 and 120 minutes. The mass of the specimens was weighed before and after heat treatment to determine the mass reduction and tested for tensile, micrograph, and fractography observation. The result is a meagre mass reduction. The highest tensile strength of the heat-treated specimen with a heat treatment temperature of 160°C and a holding time of 60 minutes is 18.310 MPa. However, it is still below the strength of the non-heat treated specimen, 19.890 MPa. Specimens with low tensile strength have a microstructure that shows an uneven distribution of titanium particles. Last, fractography shows porosity in the specimens with the lowest tensile strength.

***Keywords*****:** heat treatment, tensile strength, Fused Deposition Modeling (FDM), 3D printed titanium-PLA

## I. INTRODUCTION

3D printing, also known as additive manufacturing, involves creating three-dimensional objects under computer control. Designs are initially created using Computer-Aided Design (CAD) software, converted into STL files, processed in slicer software, and finally printed on a 3D printer (Husin et al., 2024). Among the various 3D printing techniques, Fused Deposition Modeling (FDM) is particularly popular. This method is accessible to home users due to its simple working principle and minimal equipment requirements, making it more cost-effective compared to other additive manufacturing methods (Çevik & Kam, 2020).

FDM uses filament as the printing material, which can be plastic or metal (Minh et al., 2022). Metals commonly used in FDM 3D printing include titanium (Grygier et al., 2022; Darsin et al., 2024), aluminum (Sukindar et al., 2022), stainless steel

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(Darsin et al., 2023; Sakthivel et al., 2020; Mogan et al., 2024), copper (Ambruș et al., 2021; Darsin et al., 2022), and bronze (Lu et al., 2020). In the research, the filament used is a mixture of titanium and Polylactic Acid (PLA), with a composition of approximately 80% titanium and less than 20% PLA.

Titanium and its alloys are highly compatible metals, making them the top choice for biomedical applications such as orthopedic implants (Kaur & Singh, 2019). PLA is a thermoplastic polymer that is both biocompatible and biodegradable (Singhvi et al., 2019). It provides plasticity, which facilitates the filament manufacturing process. The combination of titanium and PLA in the filament aims to leverage the mechanical strength of titanium and the biocompatibility of PLA, making it suitable for medical applications.

There has been extensive research on 3D-printed titanium, particularly focusing on its applications and properties. For instance, Kim et al. (2020) explored the mechanophysical and biological characteristics of 3D-printed titanium alloys specifically designed for dental applications. Their research highlighted the potential of these alloys in improving dental implant performance through enhanced mechanical strength and biocompatibility. Similarly, Kelly et al. (2019) investigated how surface topography and porosity affected the tensile fatigue of 3D-printed titanium alloys. Their research provided valuable insights into optimizing the surface characteristics to enhance the durability and performance of these materials under cyclic loading conditions. Both studies utilize the Selective Laser Melting (SLM) technique, which involves using a laser to fuse powdered material layer by layer to create the final product. This method allows for precise control over the microstructure and properties of the printed titanium alloys, making it a popular choice for producing high-performance components.

Previously, the researchers have published a paper discussing the tensile strength of titanium-PLA printed parts using Fused Deposition Modeling (FDM) (Darsin et al., 2024). This initial research aims to develop bone implants using titanium with the FDM technique. Additionally, there is previous research on the post-processing of 3D-printed FDM specimens with copper-PLA material by heating to reduce the PLA content. The PLA content in the specimen is significantly reduced, resulting in increased mechanical strength, specifically hardness (Ambruș et al., 2021).

In a previous study by Ambruș et al. (2021), the heat treatment of copper-PLA printed specimens has been conducted in two stages. Initially, the specimens are heated to 205°C for 30 minutes to remove the PLA content. This process is followed by a second heating stage at 983°C for one hour to facilitate diffusion between the copper particles. Remarkably, the specimens remain relatively stable or even exhibit an increase in tensile strength after this treatment. The specimens are placed in an alumina crucible and covered with a mixture of $Al_2O_3$, $SiO_3$, and $TiO_2$ powders before undergoing heat treatment to achieve those results. These powders likely play a crucial role in maintaining the specimen's shape and preventing destruction as the PLA evaporates.

The two-step heat treatment process effectively removed the PLA while promoting the diffusion of copper particles, enhancing the mechanical properties of the specimens. The use of the alumina crucible and the powder mixture provides a protective environment, ensuring the structural integrity of the specimens during the high-temperature treatment. This method demonstrates a promising approach for improving the tensile strength of copper-PLA composites by carefully controlling the heat treatment parameters and utilizing protective materials. Further research can explore the optimization of these parameters and the potential application of similar techniques to other metal-PLA composites (Ambruș et al., 2021; Shbanah et al., 2023).

Given that one of the applications of this 3D printing product is for implants in the body, it is crucial for PLA to meet biocompatibility requirements. However, PLA is hydrophobic and can cause inflammation in the surrounding living tissue due to its very low affinity for cells when used as an artificial tissue material (Khouri et al., 2024). Therefore, the researchers plan to apply heat treatment to 3D-printed FDM specimens made of titanium-PLA to reduce the PLA content in the specimens. Subsequently, tests will be conducted to determine the changes in the mechanical strength, particularly the tensile strength, of the specimens after heat treatment.

This approach aims to enhance the suitability of titanium-PLA composites for biomedical applications by improving their mechanical properties and reducing potential adverse reactions in the body. By optimizing the heat treatment process, the researchers hope to achieve a balance between maintaining structural integrity and ensuring biocompatibility, ultimately contributing to the development of more effective and reliable bone implants. Hence, the research purpose is to optimize the mechanical properties and biocompatibility of titanium-PLA filaments through heat treatment, specifically aiming to reduce the PLA content to enhance tensile strength. This study uniquely demonstrates the effectiveness of heat treatment in improving filament quality for 3D printing, marking a significant advancement in the application of biocompatible materials in medical fields.

## II. METHODS

The research involves several key steps, beginning with the design and printing phase. The design process was initially carried out using Inventor software. Then, the design file is processed using Prusa Slicer software to set the printing parameters and convert it into machine code (G-code). The 3D printing process uses a Cartesian-type 3D printer,

specifically the Ender 3 V2 until the part is fully completed.

The design, based on ASTM D638 Type V CD as depicted in Figure 1 (ASTM International, 2022), is inputted into Prusa Slicer software to set the printing parameters: bed temperature at 50°C, nozzle temperature at 225°C, print speed at 60 mm/s, infill density at 100%, and layer height at 0.1 mm. The physical appearance of the printer and the printed specimen is shown in Figure 2. Tensile test and micro-observation specimens are weighed using a digital balance before heat treatment.

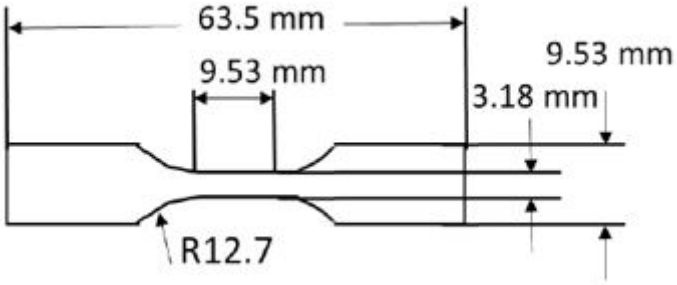



Figure 1 ASTM D638 Type V CD

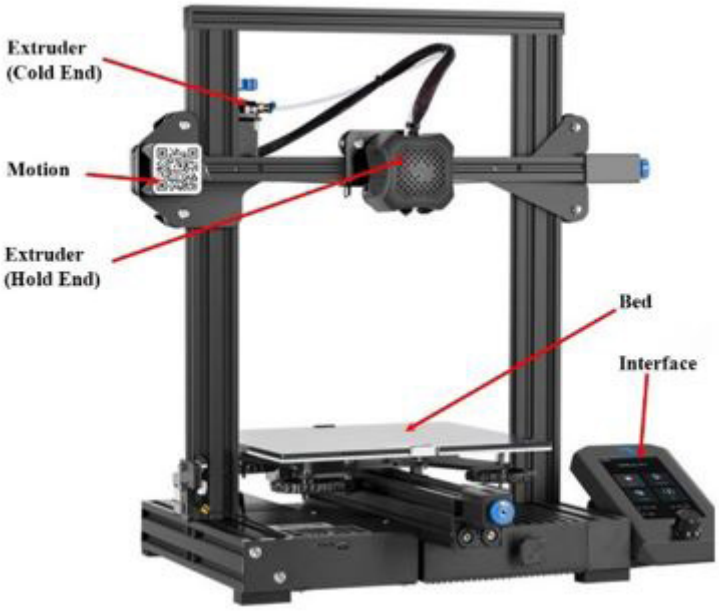



Figure 2 3D Printer Ender 3-V2

Thermogravimetric Analysis (TGA) is performed on a specimen in the form of a cylinder with a diameter of 4 mm and a height of 2 mm at the Integrated Laboratory, Universitas Diponegoro, using a NEXTA STA (Hitachi STA200RV with Real View Sample Observation) operating within a temperature range of 30–550°C. TGA measures the mass of a sample as it is heated to temperatures up to 1600°C under controlled gas flow, providing insights into the thermal stability and composition of materials.

The purpose of the heat treatment is to reduce the levels of PLA in the specimen. According to Rajan et al. (2011), key parameters influencing the heat treatment process include temperature, holding time, heating rate, and cooling rate. Heat treatment is conducted at four different temperatures: 100°C, 160°C, 190°C, and 543°C, each with holding times of 60 and 120 minutes, followed by air cooling. The selection of these variables is based on a thorough review of the literature (Guduru & Srinivasu, 2020; Jayanth et al., 2021) and preliminary studies, including TGA and initial heat treatment experiments. The equipment used is a Payun Tech NDT 5 muffle furnace with chamber dimensions of 200 mm × 200 mm × 130 mm. After heat treatment, tensile test specimens are weighed again and subjected to micrograph observations to assess changes.

Tensile strength measures a material's ability to withstand tensile forces until it undergoes plastic deformation and breaks. The research conducts tensile testing on both non-heat-treated and heat-treated specimens. It uses a universal testing machine with a capacity of 20 kN and a testing speed of 10 mm/min (ASTM International, 2022; Miller et al., 2019).

Then, microstructural observations are conducted using an Olympus BX41FX microscope. Specimens are prepared through mounting, grinding with four different grit sizes (P100, P400, P1500, P2000), and polishing with metal polish. Additionally, fracture observations are performed on specimens with the lowest tensile strength to enhance the analysis. It includes macro-fractography, which examines visible features of the naked eye or with a hand lens (Ameen, 1995). It is conducted using a digital microscope with a zoom range of 20-800×.

## III. RESULTS AND DISCUSSIONS

All specimens are printed using a 3D printer Ender 3-V2 with titanium-PLA material based on the same parameters between other layers height of 0.1 mm; filling pattern with 100%; printing speed with 60 mm/s; nozzle temperature with 225°C; and bed temperature with 60°C. The printed specimen has a visually textured surface but is smooth. The specimen results can be seen in Figure 3. It is a little hard to print a small size specimen with a 4 mm diameter and 2 mm height for the TGA due to some limitations of the printer used. In the initial printing process, the first layer of the specimen does not stick on the bed because the size is too small, so the loss of adhesion fails to print. This case can be anticipated by applying paper glue to the bed to help stick the first layer. A bit rougher surface is evident, as shown in Figure 3(a). During the process of printing bigger specimens for tensile test and micrograph observation, there is no significant problem encountered. Relatively textured surfaces and smooth side parts are obtained, as shown in Figures 3(b) and (c).

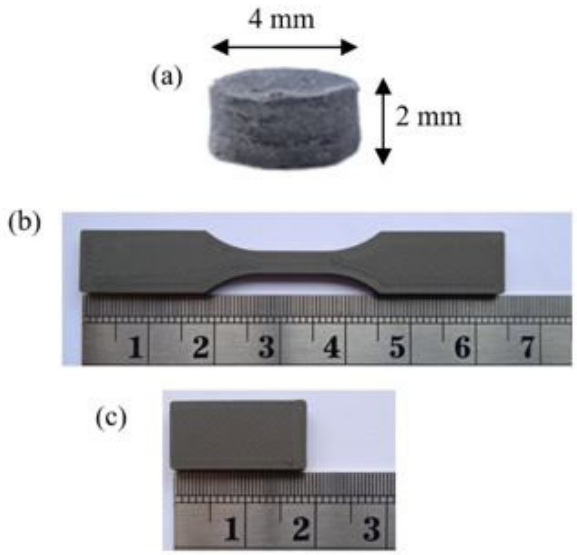



Figure 3 Specimens: (a) Thermogravimetric Analysis (TGA) Specimen, (b) Tensile Test Specimen, and (c) Micrograph Specimen

The result of the TGA (blue line) graph in Figure 4 shows that the mass of the specimen is stable up to a temperature of ± 300°C. After that, there is a drastic decrease in mass to a temperature of more than 500°C. Precisely at a temperature of 543°C, the specimen experiences the highest mass decrease, up to 22.18%. It may be related to the PLA, which burns out according to the composition of the filament, which is less than 20%. Then, until the end of heating, the specimen increases in mass by 1.2%. Sample (specimen) mass change can occur due to the presence of decomposition, evaporation, adsorption, or reaction with the atmosphere (gases) used (Saadatkhah et al., 2020).

Then, the graph with the red line is a derivative of TGA with a Derived Thermogravimetric Curve (DTG). It functions as a complement by showing the rate of change in the mass of the sample. Figure 4 shows that 3D printed titanium-PLA samples experience the highest rate of mass change at a temperature of more than 300°C, to be precise, 337°C. The value of the rate of mass change at a temperature of 337°C is 488.057 mg/minute. From the thermal analysis results, the temperature used for heat treatment is only one, namely 543°C, when the highest mass reduction occurs.

Next, the heat treatment specimens can be seen in Figure 5. At a heating temperature of 100°C, there is no change in color or shape. Slightly darker color changes and slightly curved shapes occur in specimens with heat treatment temperatures of 160°C, 190°C, and 543°C. Moreover, the heat treatment tensile test specimen at 543°C becomes crushed and out of shape. If it is touched with a fingertip, it will turn into powder.

Micrograph observation specimens are not heat treated with a temperature of 543°C. The holding time of either 60 minutes or 120 minutes is not printed because when the tensile test specimen is heated, the results are destroyed. So, the microstructure cannot be observed.

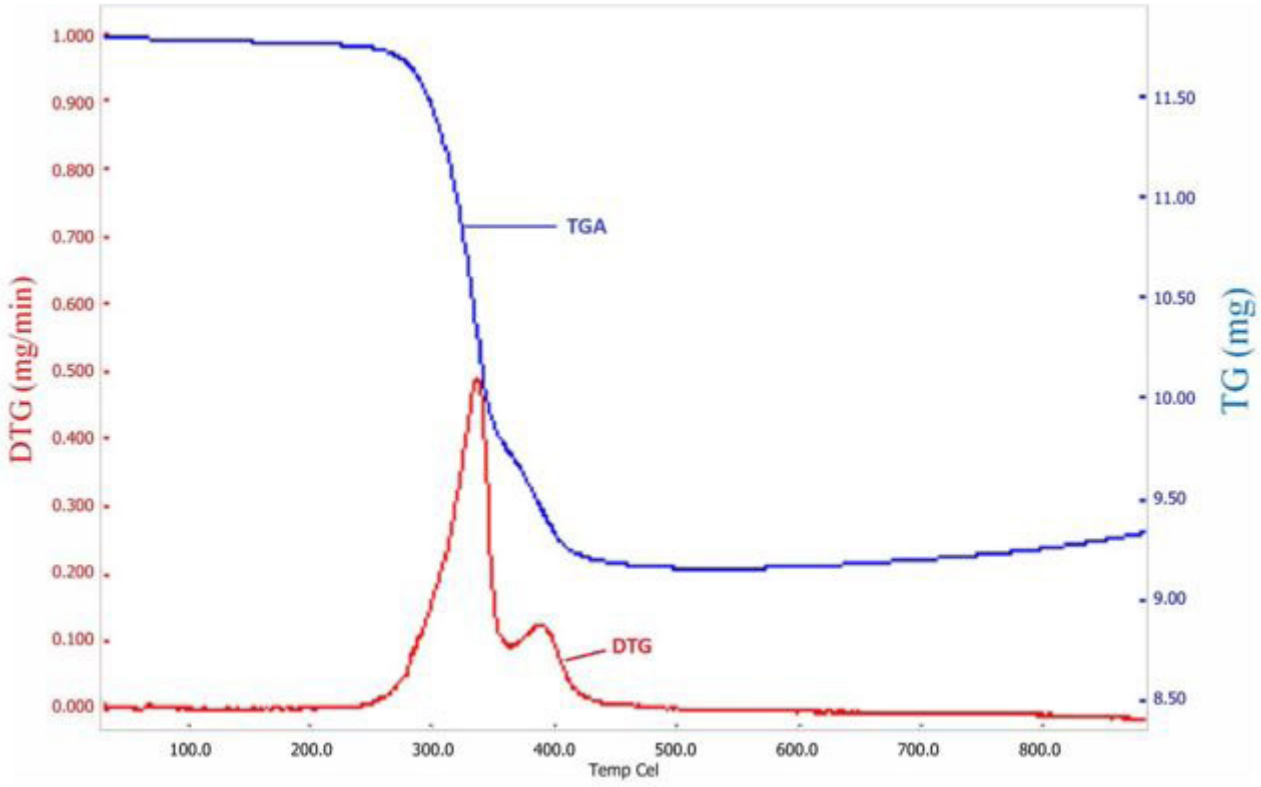



Figure 4 Thermogravimetric Analysis (TGA) Graph

The mass of the tensile test specimen, along with micrographic observations, is measured using a digital balance with an accuracy of 0.01 grams, both before and after the heat treatment. The balance is verified against another balance to ensure the accuracy of the measurements. This verified data serves as a reference for determining the mass reduction, specifically the PLA content, in the specimen after undergoing the heat treatment process.

In the presented data in Figure 6, tensile test specimens heated to 100°C show no mass change after 60 and 120 minutes of holding times. Conversely, specimens heated to 160°C experience mass reductions of 0.26% and 0.53% for the same holding times. Specimens heated to 190°C exhibit even greater mass reductions, with decreases of 0.84% and 3.27% for 60 and 120 minutes, respectively.

Similarly, micrographic observation specimens heated to 100°C also show no change in mass after 60 and 120 minutes. However, those heated to 160°C have mass reductions of 0.75% and 1.75% for the respective holding times. Finally, specimens heated to 190°C demonstrate the highest mass reductions, similar to the tensile test specimens, with decreases of 2.22% and 4.5% for 60 and 120 minutes, respectively.

The reduction in mass observed in some of these specimens cannot be definitively linked to a decrease in PLA content since the primary research objective has not yet been achieved. This is due to the lack of further analysis, such as SEM EDX testing, which is necessary to determine the specific changes in the composition of the specimens. However, it is hypothesized that the mass reduction is caused by the evaporation of certain chemicals present in the filament. Specifically, these chemicals include 2-Propenenitrile, with a boiling point of 78°C, and ethenylbenzene, with a boiling point of 138°C. The evaporation of these substances is likely contributing to the overall decrease in mass observed in the specimens.

The tensile test specimen is subjected to heating at 543°C. During this process, specimens held for 60 minutes and 120 minutes exhibit significant mass reductions of 22.44% and 22.20%, respectively. These values closely align with the TGA results, which show a mass loss of 22.18%. Upon heating to this temperature, the specimens become brittle and disintegrate into powder when pressed with a fingertip. The result suggests that approximately 20% of the PLA composition combusts and evaporates during the heating process. Further research using a Scanning Electron Microscope (SEM) is recommended to gain a deeper understanding of these changes.

Moreover, the tensile test results indicate that the highest tensile strength, measured at 19.890 MPa, is achieved by a specimen without heat treatment. Following this, a specimen subjected to heat treatment at 160°C for 60 minutes exhibits a tensile strength of 18.310 MPa. These findings suggest that heat treatment does not enhance the tensile strength of the specimens. In fact, two specimens fail the tensile test entirely, breaking before the test can be completed. These include the specimen with the lowest tensile strength value, treated at 190°C for 120 minutes, and another specimen treated at 190°C for 60 minutes. The results of the tensile tests are illustrated in Figure 7.

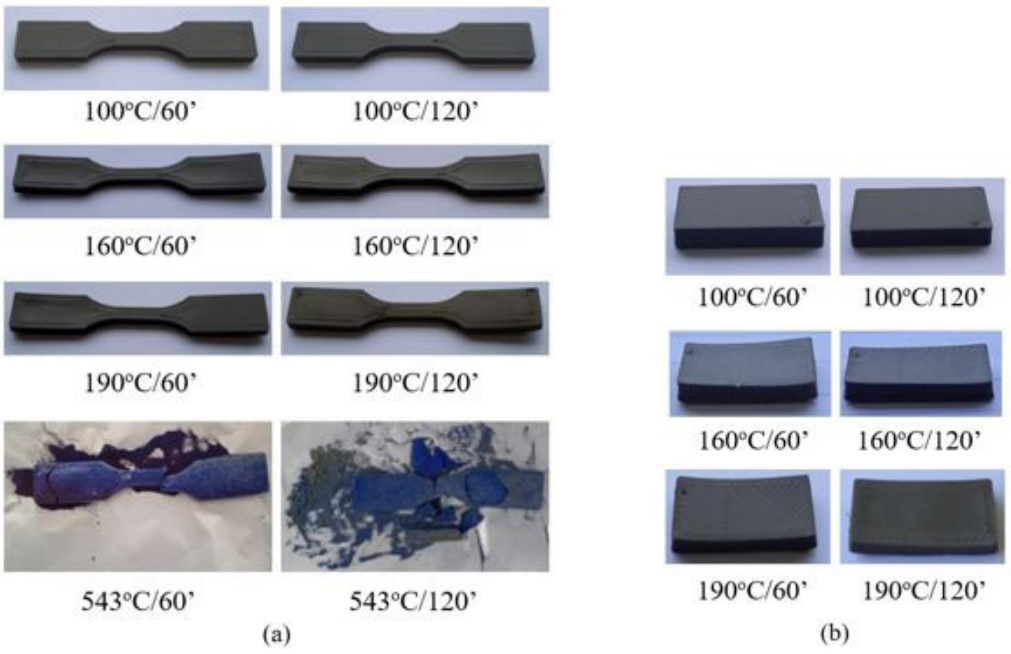
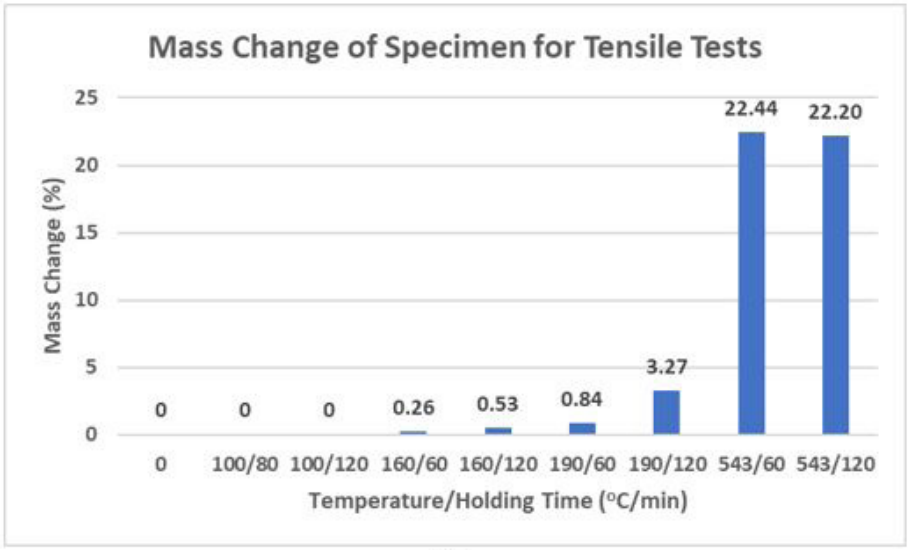



Figure 5 Heat Treated Specimens: (a) Tensile Test Specimen and (b) Micrograph Specimen

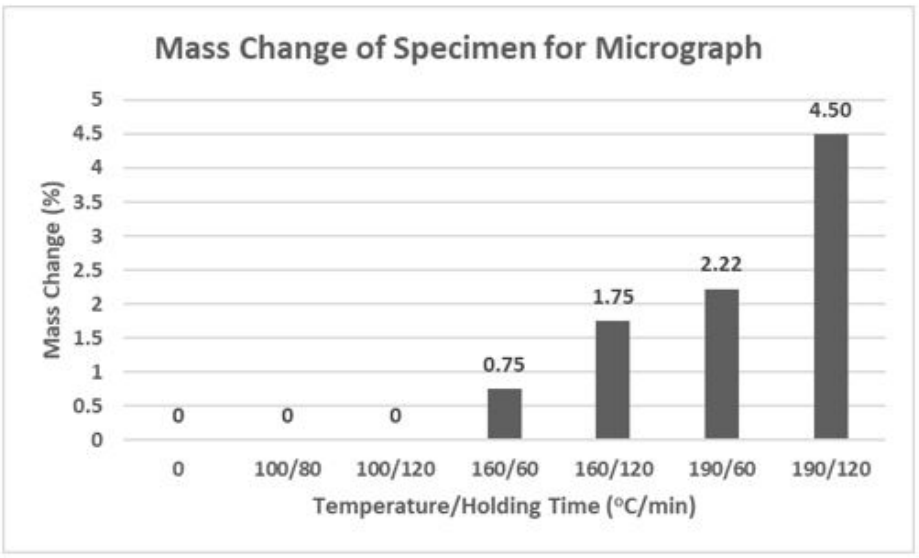
The data in Figure 7 highlight that while heat treatment at moderate temperatures (160°C) for shorter durations (60 minutes) results in a slight reduction in tensile strength, more extreme conditions (190°C) significantly compromise the structural integrity of the specimens. It indicates that the mechanical properties of PLA material are adversely affected by higher temperatures and longer exposure times, leading to brittleness and failure under stress. Further investigation into the microstructural changes occurring at these temperatures can provide deeper insight into the degradation mechanisms at play.

The microstructure of both non-heat treated and heat-treated specimens is thoroughly examined. Figure 8 illustrates micro-observation images of specimens that are not heat-treated and those that undergo heat treatment at 190°C for 120 minutes, representing the specimens with the highest and lowest tensile strengths, respectively. Figures 8(a) and (b) reveal that in the non-heat treated specimens, titanium particles are uniformly distributed within the PLA matrix. In contrast, the specimen subjected to 190°C for 120 minutes, which exhibits the lowest tensile strength, shows titanium particles clustering in several areas. This clustering indicates a significant drawback of the heat treatment process at this particular temperature and holding time. The formation of particle clusters can create weak points within the matrix, leading to a reduction in tensile strength. The clustered particles suggest inadequate dispersion during the heat treatment, which adversely affects the mechanical properties (Shrestha et al., 2020).

The fractography of the specimen subjected to 190°C for 120 minutes, which exhibits the lowest tensile strength, is shown in Figure 9. This detailed fractographic analysis provides insights into the




(a)




(b)

Figure 6 Mass Change Chart: (a) Tensile Test Specimen and (b) Micrograph Specimen

failure mechanisms of the specimen. The images reveal the microstructural changes and fracture patterns that occur due to prolonged exposure to high temperatures. These observations are crucial for understanding the material's behavior under thermal stress and can help identify the contributing factors to the significant reduction in tensile strength. Examining the fracture surfaces can give a better understanding of the degradation processes and the role of particle agglomeration in weakening the material. This information is valuable for optimizing heat treatment processes and improving the mechanical properties of PLA composites.

The fractographic analysis also reveals several voids or empty spaces in the center of the specimen, commonly referred to as porosity. This central porosity hinders the adhesion between layers, leading to premature failure of the specimens before testing. Even the specimens that withstand the testing process exhibit a significantly low tensile strength of approximately 1.4 MPa. These findings align with the observations made by Wang et al. (2022), noting that the presence of porosity defects compromises a material's ability to endure tensile loads.

The presence of porosity is a critical factor in the mechanical performance of the specimens. These voids act as stress concentrators, weakening the overall structure and making it more susceptible to fracture under tensile stress. The inability of the layers to bond effectively due to porosity results in reduced tensile strength and early failure. It highlights the importance of minimizing porosity during the manufacturing process to enhance the mechanical properties of the material. Further research and advanced imaging techniques can provide deeper insights into the formation of porosity and strategies to mitigate its impact on material performance.

Among the three tensile test specimens subjected to a heat treatment temperature of 190°C for 60 minutes, one specimen fails the test. Despite this, the other two specimens have successfully completed the test, resulting in an average tensile strength of 14.734 MPa. The fractographic analysis of these specimens is shown in Figure 10. In the case of pure 3D printed PLA, heat treatment actually reduces the porosities (Shbanah et al., 2023).

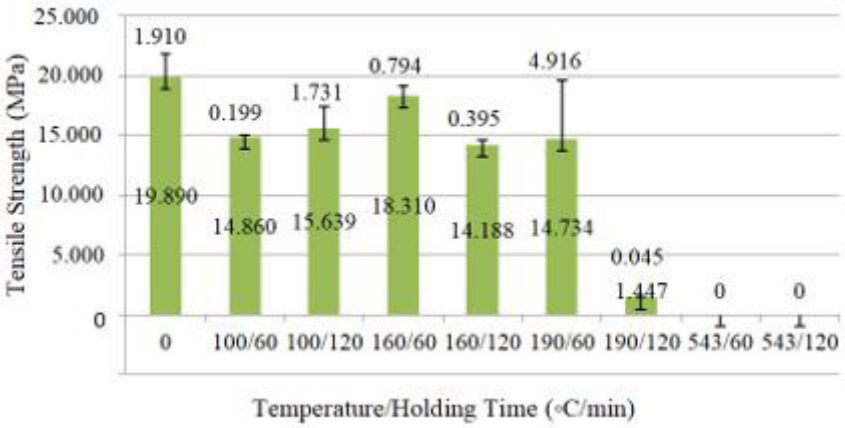



Figure 7 Tensile Test Chart

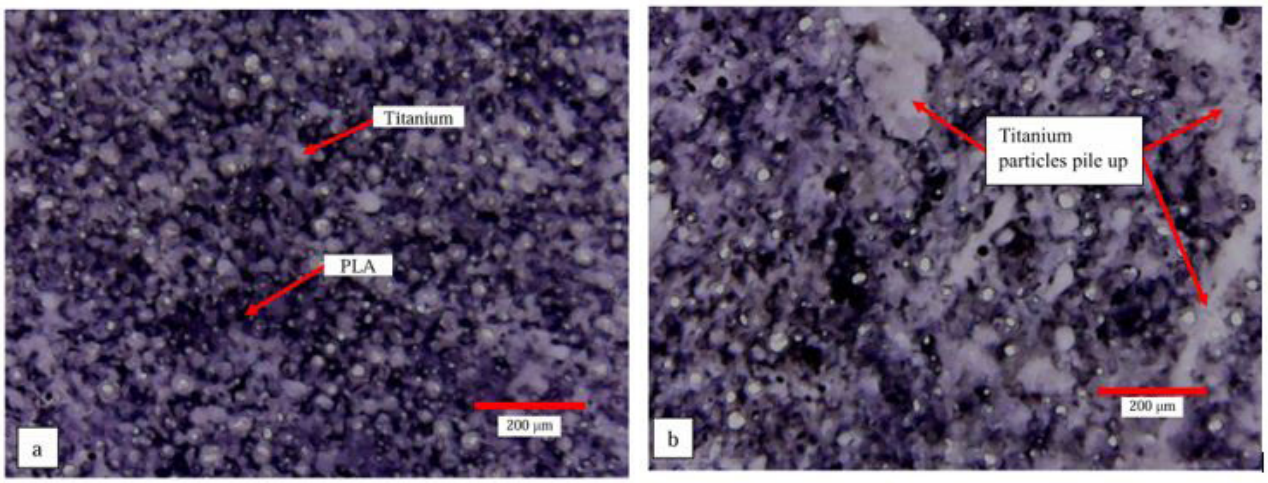



Figure 8 Microstructure: (a) Non-Heat Treated Specimen (b) Specimen Heat Treatment with 190°C/120'

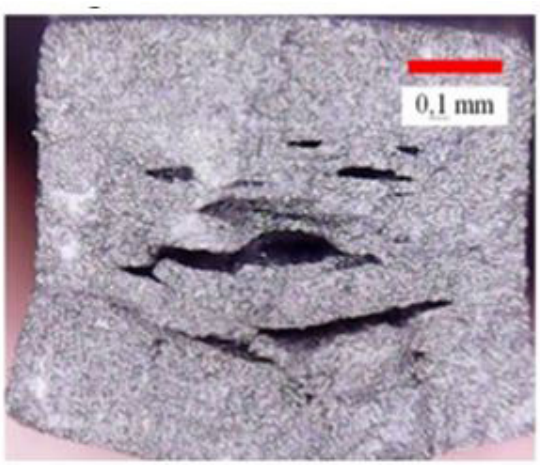



Figure 9 Fractograph of Specimen with 190°C/60’

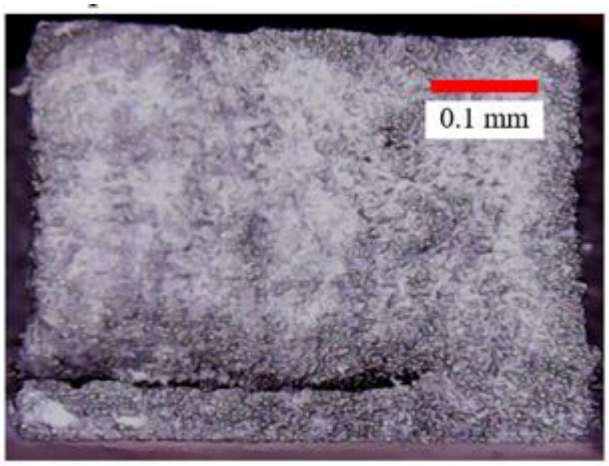



Figure 10 Fractograph of Specimen with 160°C/120’

The failure of one specimen highlights the variability in the material's response to heat treatment at this temperature and duration. While achieving average tensile strength, the successful specimens still indicate more reduced mechanical properties than non-heat treated specimens. Fractographic images provide valuable insights into the fracture mechanisms and microstructural changes that occur due to heat treatment. These observations can help to understand the contributing factors to the variability in tensile strength and guide future improvements in the heat treatment process to enhance the material’s performance.

A line-shaped cavity is observed, formed within the layer during the specimen printing process due to thermal runaway. Thermal runaway occurs when the machine temperature continuously rises uncontrollably, necessitating an immediate shutdown. However, the printing process can resume once the machine temperature stabilizes according to the set parameters. This interruption causes the specimen to be imperfect, with one layer not aligning properly, thus creating a cavity. Additionally, when the specimen is heated to 160°C for 120 minutes, this existing cavity can enlarge, leading to the specimen breaking before testing.

Thermal runaway is a critical issue in the 3D printing process because it disrupts the uniformity and structural integrity of the printed layers. The resulting cavities act as weak points within the material, compromising its mechanical properties. When subjected to further heat treatment, these cavities can expand, exacerbating the defects and causing premature failure. Understanding and mitigating thermal runaway is essential for ensuring the quality and reliability of 3D-printed specimens. Implementing better temperature control mechanisms and monitoring systems can help to prevent such occurrences, thereby improving the overall performance of the printed materials.

Several attempts have been made to reduce or eliminate the PLA from printed products made with titanium-PLA filament. However, reducing the PLA content significantly diminishes the tensile strength of the product because the titanium-PLA compound functions similarly to a composite material, where titanium acts as the reinforcement and PLA serves as the matrix. The PLA binds the titanium particles together. Without it, the titanium will simply be a collection of grains. When heated to a certain temperature, the PLA melts, causing the titanium to revert to its original granular form, as shown in Figure 8b. Insufficient PLA as a binding agent leads to a substantial decrease in tensile strength, as illustrated in Figure 7 (Shbanah et al., 2023).

The role of PLA in the titanium-PLA filament is crucial for maintaining the structural integrity of the printed product. PLA not only binds the titanium particles but also provides the necessary matrix to distribute stress and enhance mechanical properties. However, removing or reducing PLA disrupts this balance, resulting in a weaker, less cohesive material. It highlights the importance of optimizing the PLA content to ensure the desired mechanical performance while exploring alternative methods to enhance the properties of the composite without compromising its integrity.

Based on the discussion presented, the researchers’ analysis includes several key points. The observed mass decrease of 22.18% at a temperature of 543°C is indicated by TGA. It suggests that the PLA content is burned off and evaporates. This result aligns with the filament composition, which contains 20% PLA, with the remaining 2.18% considered as tolerance.

The changes in the shape of the specimen after heat treatment are attributed to the temperatures used, which are close to, slightly above, and well above the melting point of PLA (180°C) (Rahmayetty et al., 2018). These temperature variations cause the PLA to melt and alter the specimen’s structure, leading to significant changes in its mechanical properties.

Meanwhile, changes in mass during the TGA heating process can result from volatile evaporation, drying, desorption, and decomposition (Gabbott, 2008). The lower mass decrease observed in the specimen heated at 543°C for 120 minutes compared to the one heated for 60 minutes may be due to adsorption, where ions are retained, or metal oxidation occurs in air or oxygen in the 543°C/120 minutes specimen. The change in mass cannot be conclusively attributed to a decrease in PLA levels because further testing has not been conducted.

Fluctuations in the average tensile strength of all specimens are observed due to the varying accumulation of titanium powder particles, which is influenced by different heat treatment temperatures and holding times. These inconsistencies lead to variations in mechanical properties. The accumulation of titanium powder particles in certain areas and the presence of porosity contribute to the decrease in the tensile strength of the specimens. These factors create weak points within the material, reducing its overall mechanical performance. However, the case is different when heat treated with 3D-printed pure PLA. Heat treated specimen at 65°C for 5 hours increases the strength by 35% (Shbanah et al., 2023). It may be due to the different nature of materials, in which PLA is a binder in filaments made of PLA-titanium. Removing PLA may result in the loss of bonds between titanium grains.

Last, the analysis underscores the importance of precise temperature control and uniform particle distribution in maintaining the structural integrity and mechanical properties of PLA-based composites. Further research and advanced testing methods are necessary to fully understand the impact of these variables and optimize the heat treatment process for improved material performance.

## IV. CONCLUSIONS

Heat treatment is conducted using temperature parameters of 100°C, 160°C, 190°C, and 543°C, with holding times of 60 and 120 minutes. These treatments do not result in an increase in the tensile strength of the specimens compared to those that are not heat-treated. Additionally, most specimens exhibit an uneven distribution of titanium particles after heating. The optimal tensile strength for 3D-printed titanium specimens is achieved with a heat treatment temperature of 160°C and a holding time of 60 minutes.

The findings indicate that while heat treatment can influence the distribution of titanium particles within the PLA matrix, it does not necessarily enhance the tensile strength of the material. The uneven distribution of particles observed in most specimens suggests that higher temperatures and longer holding times may lead to agglomeration or other structural changes that weaken the material. Therefore, maintaining a moderate temperature of 160°C and a shorter holding time of 60 minutes appears to be the most effective approach for preserving the mechanical properties of 3D-printed titanium-PLA composites.

Further research can explore additional parameters and techniques to optimize the heat treatment process and improve the overall performance. A factor that may affect the result of heat treatment is the heating rate. A lower heating rate ensures the very slow melting of PLA. It can also ensure the intermetallic bonding between titanium occurs during heat treatment.

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