EFFECT OF HEAT TREATMENT ON THE MECHANICAL PROPERTIES AND MICROSTRUCTRURE OF ADDITIVE MANUFACTURED TI-6AL-4V ALLOY

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This research explores how heat treatment influences both the mechanical properties and microstructure of Ti6Al4V alloy produced through additive manufacturing. The samples were produced using the selective laser melting (SLM) technique and analysed using evaluation equipment, including digital optical microscope, scanning electron microscope, X-ray diffractometer, compression tester, and hardness tester. The findings indicate that notable differences in mechanical properties and microstructural characteristics due to the heat treatment process. The annealed samples exhibit a significant improvement in mechanical properties compared with the as-printed sample, which may be due to the changes in microstructure. Annealing at lower temperatures resulted in improved hardness and compressive strength; namely, the specimens that underwent annealing at 850 °C showed better mechanical strength than the specimens subjected to annealing at 950 °C and 1050 °C. The grain is finer at lower annealing temperatures, which may be the main reason for the changes in mechanical properties. Understanding the relationship between heat treatment and material characteristics is essential to advancing the application of Ti6Al4V in aerospace and medical implants.

KEYWORDS

ADDITIVE MANUFACTURE, TI-6AL-4V, HEAT-TREATMENT

1 INTRODUCTION

Ti-6Al-4V is recognized for its outstanding mechanical characteristics, resistance to corrosion, and biocompatibility, making it widely used in industries such as aerospace, biomedical implants, and automotive [Leyens 2003]. Its exceptional strength-to-weight ratio and superior mechanical properties make it an ideal material for applications such as jet engines, gas turbines, and various airframe components [Liu 2021; Rawal 2013; Gomez-Gallegos 2018; Peters 2003]. Ti-6Al-4V's low density, high strength, excellent corrosion resistance, and biocompatibility make it highly suitable for applications like bridges and implants [Haydar 2024; Fojt 2012; Yan 2015].

Conventional manufacturing techniques for Ti-6Al-4V products usually include processes such as forging, casting, and rolling of bulk material, followed by machining to obtain the desired shapes and dimensions. These conventional processes often lead to substantial material waste, high manufacturing costs, and extended production times. In contrast, additive manufacturing (AM) provides a more efficient alternative. Additive manufacturing (AM) creates components layer by layer directly from CAD designs, minimizing waste and allowing the creation of complex geometries without traditional processing steps.

Among the different additive manufacturing (AM) techniques, Selective Laser Melting (SLM) has emerged as a leading method for producing high-performance metal alloys, such as Ti-6Al-4V [Blaha 2023; Tanzli 2024]. SLM involves the selective melting and solidification of fine titanium powder using a high-energy laser, constructing the part layer-by-layer. This method produces a microstructure with fine cellular structures and columnar grains. However, the rapid solidification associated with SLM introduces significant challenges [Liu 2019; Thijs 2010; Yang 2016]. The process generates large temperature gradients and high cooling rates, leading to residual stresses, anisotropic mechanical properties, and potential defects such as porosity and micro-cracks. These factors can adversely impact the mechanical performance and fatigue resistance of the finished parts.

The microstructure of Ti-6Al-4V generally comprises two phases: the hexagonal close-packed (hcp) α phase and the body-centered cubic (bcc) β phase [Simonelli 2012; Sun 2019]. The composition, morphology, and proportion of these phases significantly influence the alloy's static properties. In the as-built condition, as a result of the rapid cooling during SLM, the microstructure primarily consists of needle-like α' martensite within the prior β grains. This acicular martensite α' phase, while strengthening the material, is also brittle and lacks ductility [Yang 2016]. Thus, addressing these issues through heat treatment is essential for optimizing the alloy's performance.

Post-processing techniques, including heat treatment, are essential for improving the mechanical properties of Ti-6Al-4V components produced through Selective Laser Melting (SLM). Heat treatment involves controlled heating, holding at specific temperatures, and cooling to alter the alloy's microstructure and mechanical properties

Heat treatment helps reduce residual stresses, homogenize the microstructure, and improve ductility [Khorasani 2018; Liu 2021]. During heat treatment, heating to elevated temperatures allows for phase transformations that affect the distribution of α and β phases and the formation of secondary phases. Subsequent cooling rates influence microstructural refinement by controlling grain growth and precipitation, therefore influencing mechanical properties like strength, hardness, and ductility.

A common heat treatment process for Ti-6Al-4V is solution treatment followed by aging (STA). Solution treatment consists of heating the alloy to a temperature above the β-transus (usually between 950-1000°C). to dissolve α precipitates and achieve a homogeneous β phase. Following this, aging is performed at a lower temperature (usually between 500-600°C) to precipitate fine α phase particles within the β matrix, enhancing strength through precipitation hardening [Liu 2021; El-Hadad 2018] . Alternatively, the effectiveness of heat treatment depends on precise control of parameters including temperature, holding time, and cooling rate. These factors are essential for obtaining the desired microstructural properties and, consequently, the optimal mechanical properties of SLM-produced Ti-6Al-4V parts.

This paper aims to comprehensively review the current state of research on the impact of heat treatment on Ti-6Al-4V components produced by SLM. It will explore how different heat treatment parameters influence microstructural evolution and mechanical properties, providing valuable insights into optimizing heat treatment strategies for enhancing the performance and reliability of Ti-6Al-4V components fabricated via SLM. By elucidating these relationships, this study seeks to advance the application potential of AM in critical industries where material performance and reliability are paramount.

2 EXPERIMENTAL

2.1 Material and Equipment

The metal powder utilized in this study was Renishaw Ti-6Al-4V ELI-0406, with the composition details provided in Table 1. The powder specifications featured a particle size distribution between 15 and 45 µm, with an average particle size of 30 µm. Ti-6Al-4V parts were produced using the SLM technique on the TRUMPF TruPrint 1000 with a build chamber of 100 x 100 x 100 mm. An argon atmosphere was maintained in the build chamber to reduce oxygen exposure during the printing process. The specimens were fabricated with a layer thickness of 30 µm, a laser power of 200 W, hatch spacing of 100 µm, scan speed of 800 mm/s, and an additional layer thickness of 50 µm. The samples were then cut from the base plate by Fanuc Robocut Alpha-C600IC wire cutter.

Element	Composition
Ti (Titanium)	Balance
Al (Aluminum)	5.50 – 6.50
V (Vanadium)	3.50 – 4.50
Fe (Iron)	≤ 0.25
O (Oxygen)	≤ 0.13
C (Carbon)	≤ 0.08
N (Nitrogen)	≤ 0.05
H (Hydrogen)	≤ 0.012
Y (Yttrium)	≤ 0.005
Residuals	≤ 0.10 each, ≤ 0.40 total

Table 1. Chemical composition of the as-printed Ti6Al4V sample.

2.2 Heat Treatment

Heat treatments were applied to the Ti-6Al-4V specimens fabricated via SLM to investigate the effects on mechanical properties and microstructure using a Nabertherm NW300. The heat treatment process was carried out in a vacuum furnace. The specimens were carefully inserted into the furnace, where they were gradually heated at a rate of 5°C/min. The first step involved aging the samples at 650°C for 3 hours, allowing for the relief of internal stresses and microstructural refinement. After the aging process, the specimens were annealed at progressively higher temperatures: 850°C for 1 hour, 950°C for 1 hour, and 1050°C for 1 hour. The 850°C annealing temperature was selected to optimize the distribution of the α+β phases, enhancing strength and ductility while maintaining a refined microstructure. At 950°C, the alloy experiences further phase changes, dissolving finer α phase into the β phase, which may soften the material but still retain beneficial mechanical properties. The 1050°C treatment, above the β-transus temperature, was used to observe the effects of complete β phase formation, which results in coarsening of the β grains and a reduction in strength due to excessive ductility. Following the annealing treatments, the specimens were then furnace-cooled at a rate of approximately 10°C/min.

2.3 Mechanical Testing

Hardness testing was carried out on cuboid samples with the dimensions of 15x15x10mm to evaluate the hardness of the Ti6Al4V alloy. Hardness tests were conducted by taking five random measurements on a single sample at different locations. Hardness measurements were taken using a Qualitest Rockwell hardness tester, featuring a diamond cone indenter and a 150 kgf test load.

Compression tests were performed on cylindrical specimens with the dimensions of R6 x 18mm to evaluate the compressive strength. Tests were conducted at a strain rate of 0.01 mm/min until failure using an MTS Exceed® Series 40 Electromechanical Universal Test Systems.

2.4 Microstructure Analysis

The microstructure analysis was conducted using cuboid samples of Ti-6Al-4V with dimensions of 15 x 15 x 10 mm. Phase characterization was carried out using an X-ray diffraction (XRD) diffractometer (AERIS, Panalytical) with Co-Kα radiation. The resulting XRD data were processed with Jade 6.5 software to identify the phase composition of the Ti-6Al-4V samples produced by SLM. Surface morphology and detailed microstructure were further investigated using scanning electron microscopy (SEM) with a JSM-IT200 system (JEOL). For scanning electron microscopy (SEM), the samples were prepared by sequential grinding using SiC abrasive papers of different grit sizes (100, 200, 400, 800, 1200, and 2000 mesh). Following grinding, the samples were polished with chromium oxide powder to obtain a mirror-like surface finish. The polished samples were then etched using a Kroll's reagent (H_2O: HNO_3: HF = 93:5:2, volume%) to reveal the metallographic structures

3 RESULTS AND DISCUSSION

3.1 Microstructure properties

Scanning electron microscopy (SEM) was used to analyze the surface morphology and microstructure of both the Ti-6Al-4V powder and the specimens. Figure 1 presents the Scanning electron microscope (SEM) images of Ti-6Al-4V metal powder before and after printing. The printed powder exhibits high quality with a uniform structure and excellent surface finish. Additionally, the printing process does not significantly alter the powder, allowing it to maintain its quality and be reused for subsequent printing. Figure 2 shows SEM micrographs of the specimens. SEM analyses of Ti-6Al-4V specimens revealed that as-built samples exhibited a columnar grain structure with some porosity. Heat treatment significantly improved the microstructure by refining grain size and reducing porosity, which enhanced mechanical properties. At 850°C, the α+β (alpha-beta) microstructure was well-optimized, resulting in improved strength and toughness due to a fine phase distribution and reduced residual stresses. This temperature allowed for effective phase transformation and minimized defects. However, at 950°C, the increase in the beta phase led to coarser microstructure as finer alpha phases dissolved, reducing strength and hardness because beta grains are typically softer. At 1050°C, prolonged exposure resulted in a material predominantly in the beta phase with significant grain coarsening and complete dissolution of the alpha phase. This high-temperature treatment severely compromised mechanical properties, making the alloy softer and less strong, and decreasing ductility, which increased susceptibility to failure under stress [Jin 2021]. These results highlight the significance of accurate temperature control during heat treatment to achieve a balanced phase distribution and optimize mechanical performance.

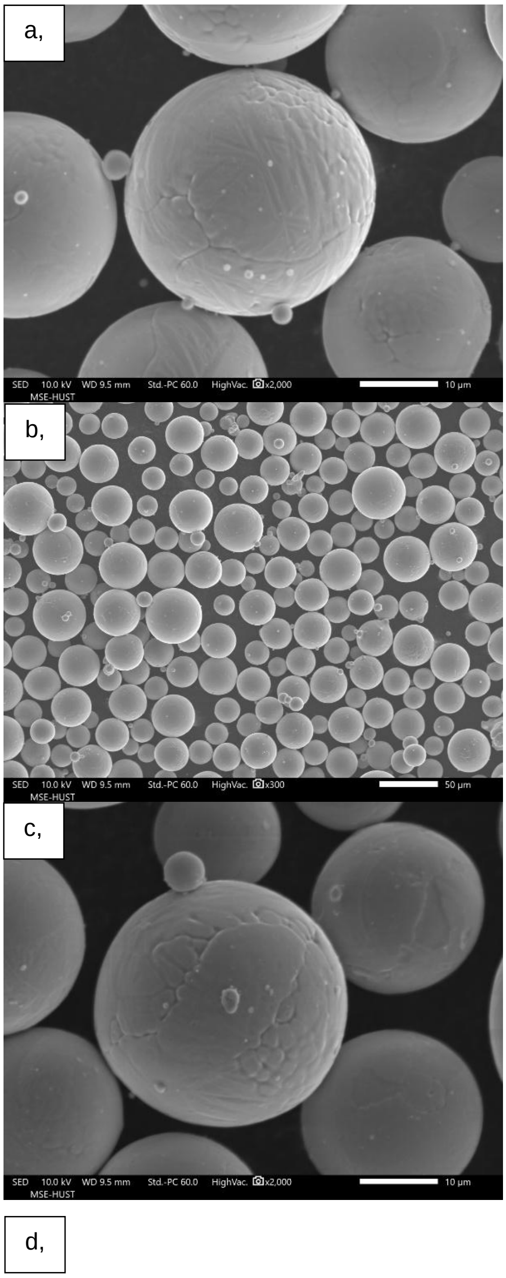

Figure 1. SEM micrographs of: a, b) virgin Ti-6Al-4V powder and c, d) printed Ti-6Al-4V powder

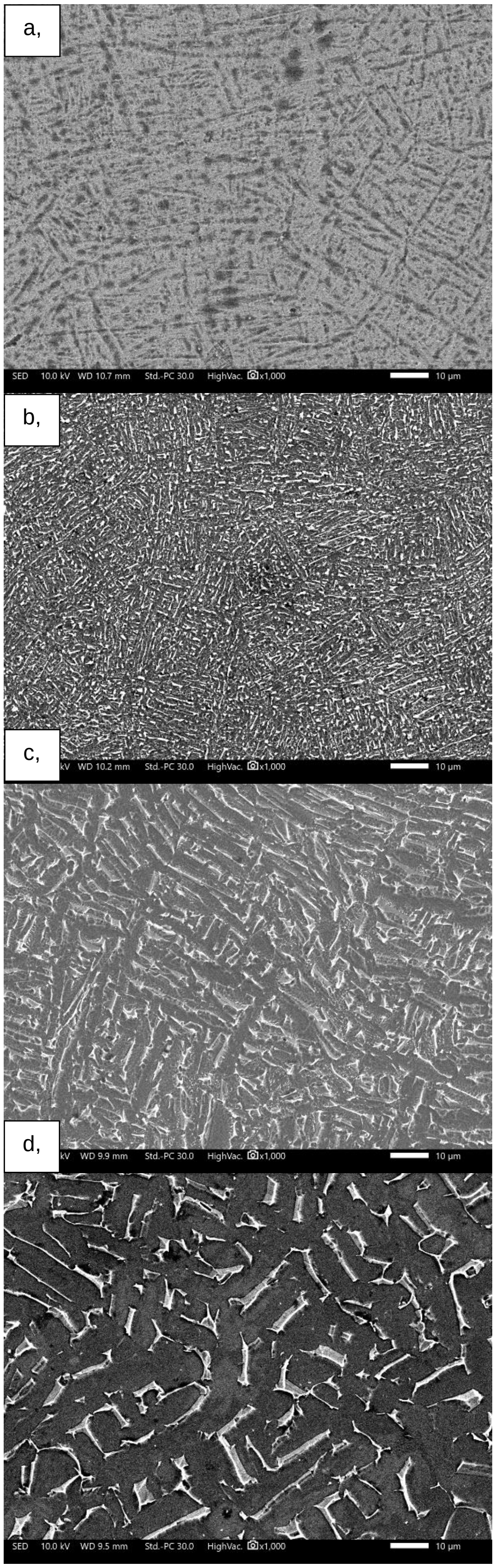

Figure 2. SEM micrographs of: a) as-built b) 850 ^{o}C annealed c) 950 ^{o}C annealed and d) 1050 ^{o}C annealed sample

3.2 Hardness result

The average hardness test results are presented in Table 2, while the hardness measurements at each point are shown in Figure 3. Heat treatment has a substantial impact on the hardness of Ti-6Al-4V alloy. The as-built specimens exhibited a hardness of 32.2 HRC due to its martensitic microstructure, formed during rapid solidification, which contained high residual stresses. To mitigate these stresses and stabilize the microstructure, a preliminary heat treatment at 650°C for 3 hours was conducted before annealing, promoting partial transformation of the α' martensite into a more stable α+β phase.

At 850°C, the hardness increased to 39.5 HRC, indicating improved strength due to an optimized α+β phase distribution. This temperature facilitated phase transformation while maintaining a refined microstructure with balanced mechanical properties. However, raising the annealing temperature to 950°C resulted in a decrease in hardness to 36.98 HRC, as finer alpha phases dissolved and beta phase grains coarsened. The loss of fine alpha grains resulted in a softer structure, reducing hardness and strength.

At 1050°C, the hardness further declined to 31.76 HRC, similar to the as-built condition. This significant reduction was due to complete dissolution of the alpha phase and excessive beta grain growth, which compromised mechanical performance by increasing ductility and reducing resistance to deformation. Prolonged exposure to temperatures exceeding the β-transus results in grain coarsening and the loss of strength-enhancing phases, ultimately reducing material hardness [Seo 2024].

Specimen Condition	Average hardness (HRC)
As-Built	32.22
Heat Treated (850°C/1h)	39.52
Heat Treated (950°C/1h)	36.98
Heat Treated (1050°C/1h)	31.76

Table 2. Summary of Hardness Test result.

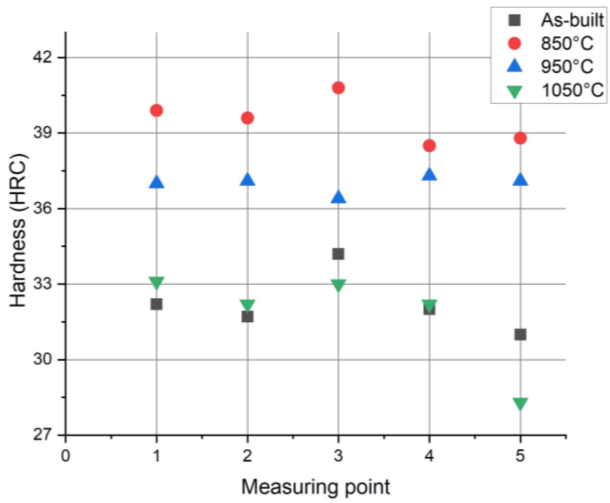

Figure 3. Hardness Test result

3.3 Compression result

Compression tests were conducted to evaluate the compressive strength of the Ti-6Al-4V specimens under different heat treatment conditions. Table 3 presents the compressive strength of the specimens. Figure 4 presents the corresponding compressive stress-strain curves. The compression test results for Ti-6Al-4V specimens indicate that heat treatment significantly enhances compressive strength compared to the as-built condition. The specimens heat-treated at 850°C for 1 hour achieve the highest compressive strength of 1933.07 MPa, suggesting that this treatment temperature optimally improves the material's mechanical properties. In contrast, higher temperatures of 950°C and 1050°C result in reduced compressive strengths of 1789.62 MPa and 1679.19 MPa, respectively. The decrease in compressive strength observed at higher temperatures (950°C and 1050°C) is likely due to the dominance of the beta phase, which, while more ductile, has lower strength compared to the alpha phase. In addition, grain coarsening at these elevated temperatures further reduces the material's overall strength. The alpha phase does not coarsen excessively at these higher temperatures; rather, it dissolves into the beta phase, leading to a microstructure that is less resistant to deformation and lowering the compressive strength [Seo 2024].

Specimen Condition	Compressive Strength (MPa)
As-Built	1933.07
Heat Treated (850°C/1h)	1789.62
Heat Treated (950°C/1h)	1679.19
Heat Treated (1050°C/1h)	1933.07

Table 3. Summary of Compression Test Result.

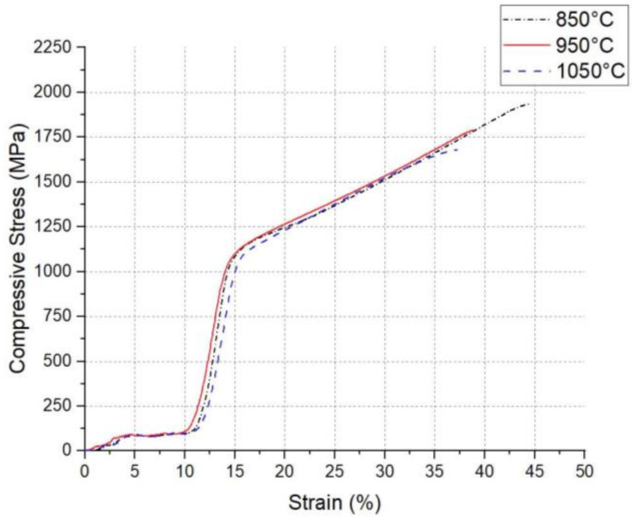

Figure 4. Stress-strain curve for Compression Test Result

3.4 XRD Analysis

X-ray diffraction (XRD) analysis was conducted to examine the crystallographic phases in the Ti-6Al-4V powder both before and after the printing process. Figure 5 shows the XRD patterns obtained. The XRD analysis of both virgin and used Ti-6Al-4V powder reveals no significant changes in the primary diffraction peaks corresponding to the α and β phases. This stability in peak positions indicates that the phase composition of the powder remains unchanged throughout the additive manufacturing process. These results indicate that the structural integrity and phase distribution of the Ti-6Al-4V powder are maintained, allowing the used powder to be effectively reused and thereby reducing waste.

Additionally, as observed in Figure 1, the SEM results further demonstrate that the quality of the powder before and after printing does not change significantly. However, further investigations using complementary techniques may be needed to fully assess any potential microstructural or surface modifications that are not detectable by XRD alone.

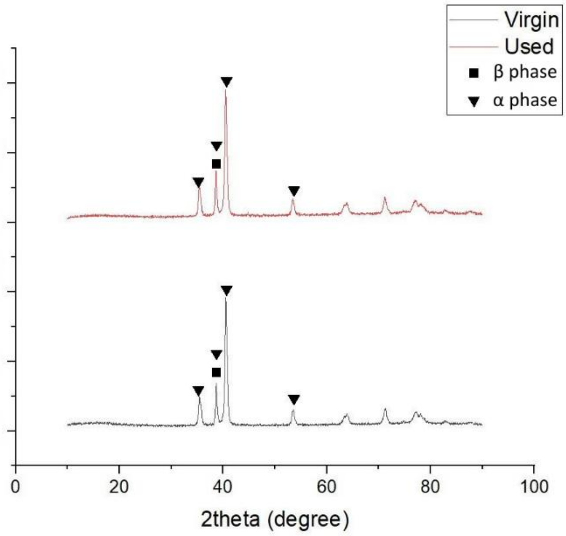

Figure 5. XRD patterns of used and virgin Ti6Al4V metal powder.

4 CONCLUSIONS

This study investigates the impact of heat treatment, particularly annealing at temperatures both below and above the β-transus, on the mechanical properties and microstructure of Ti-6Al-4V alloy produced via selective laser melting (SLM). The findings reveal notable alterations in material characteristics due to annealing.

Annealing resulted in a more uniform microstructure in the as-printed Ti-6Al-4V specimens. However, as the annealing temperature increased, the size of the phases within the alloy also grew, which was associated with a decrease in both hardness and compressive strength. In particular, temperatures exceeding the β-transus caused a notable decline in mechanical properties, which is attributed to the growth of beta phase grains and the resulting coarsening of the microstructure.

Additionally, X-ray diffraction (XRD) analysis showed that both virgin and used Ti-6Al-4V powders exhibited no significant changes in the primary diffraction peaks corresponding to the α and β phases. This stability in peak positions suggests that the phase composition of the powder remains consistent throughout the additive manufacturing process. Consequently, these results indicate that the structural integrity and phase distribution of Ti-6Al-4V powder are preserved, allowing for the effective reuse of powder and contributing to waste reduction.

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