OPTIMIZATION OF POST-PROCESSING HEAT TREATMENT FOR LPBF MODIFIED 2024 ALUMINIUM ALLOY

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Abstract

Additive manufacturing of high-performance aluminum alloys, such as the 2000 series, presents problems with the formation of hot cracks during the rapid solidification of LPBF methods. A promising solution involves inoculation, which induces a very fine and equiaxed microstructure with exceptional properties and crack-free formation. This study examines the effects of heat treatments on the microstructure and mechanical properties of an aluminum alloy commercially known as 2024 RAM2, an LPBF-ed aluminum matrix composite loaded with titanium and ceramic particles. The as-built microstructure presents fine and equiaxed grains around 1 µm, aided by the presence of inoculants. The T6 heat treatment led to partial homogenization, causing grain growth in some areas and resulting in heterogeneous grain sizes. In contrast, the T5 treatment preserved the fine-grained structure from LPBF, maintaining the microstructure and leading to a more uniform fine-grained material. After T6 heat treatment, the samples presented a microhardness of 150.3 ± 17.8 HV, while those of the T5 sample were 172.5 ± 4.2 HV, achieving higher values of microhardness with a reduction in variability.

Keywords: Additive manufacturing; laser powder bed fusion; microstructure; heat treatment; high-strength aluminum alloys

1. Introduction

Additive Manufacturing (AM), commonly known as 3D printing, has emerged as a revolutionary manufacturing process with extensive applications across various industries. Its capability to fabricate complex geometries directly from digital designs offers several advantages over traditional manufacturing techniques like casting. In the aerospace sector, where lightweight structures are crucial for enhancing fuel efficiency, payload and overall performance, AM has gained significant traction due to its ability to produce intricate components while maintaining structural integrity [1].

Unlike conventional methods like casting, which often involve extensive material removal and result in significant waste, AM enables precise material deposition only where needed. This minimizes material waste, and reducing costs, especially for high-value materials [2].

Among various AM techniques, laser powder bed fusion (LPBF) is one of the most widely adopted. In LPBF, a high-powered laser selectively melts a thin layer of powder, typically ranging from tens to one hundred of micrometers, according to a 3D CAD model. The molten pool solidifies extremely rapidly, on the order of 10^5 K/s [3], and a new layer is deposited on top of the previous one. This rapid cooling process ensures the formation of fine microstructures and desirable material properties.

Recent research has focused on the development of high-performance aluminum alloys in the 2xxx, 6xxx, and 7xxx series, which provide the superior performance required for aerospace applications. However, a major limitation of using these alloys in AM is their susceptibility to hot cracking, which can lead to defects such as hot tears [4]. This phenomenon is caused by the excessive solidification temperature range of the alloys, leading to grain shrinkage during the solidification process and generating significant residual stresses and cracks in the material.

To address the issue of hot cracking in AM, research has focused on optimizing process parameters and modifying alloy compositions. However, these approaches often significantly alter material properties. Recently, attention has turned to adding inoculants, such as Zr, Ti and Sc, as a solution to address this challenge. This method is preferable as the addition of small amounts of inoculants can greatly increase nucleation sites during the initial stages of solidification, transforming the structure from columnar to equiaxed. This new structure more evenly distributes segregation elements and reduce thermal stresses, thereby mitigating hot cracking during the AM process [5].

The formation of a fine microstructure is one of the primary strengthening mechanisms, known as Hall-Petch strengthening. The high number of grain boundaries impedes dislocation movement due to varying grain orientations, hindering dislocation movement on the same plane. Additionally, very fine inoculants, on the order of nanometers, can promote another reinforcement method known as the Orowan mechanism. This mechanism involves the interaction of nanoscale particles with dislocations, promoting dislocation bowing through Orowan loops [6].

In addition to these strengthening mechanisms, solid solution and precipitation strengthening also contribute to reinforcement [7]. Additive manufacturing often results in high percentage of trapped elements within the matrix, which act as barriers to dislocation movement. Furthermore, the formation of various precipitates can hinder dislocation motion, improving the material’s mechanical properties.

Through solution treatment, the amount of alloying elements within the matrix can be increased. If followed by rapid quenching, such as in water, the stable microstructure near the melting point can be feezed at lower temperature. This treatment essentially homogenizes the material erasing the microstructure produced during processing. Conversely, increasing the quantity of incoherent precipitates within the matrix reduces the amount of elements in solid solution [8].

This study focuses on modifying aluminum alloy 2024 with titanium and ceramic particles as inoculants. The objective is to investigate the effect of heat treatments on the microstructure formed through LPBF technology. Specifically, two heat treatment methods are compared: the conventional T6 treatment (solution treatment + aging), representing the industrial standard for this type of material, and a direct aging process (T5). The aim is to compare the resulting microstructures obtained and measure the mechanical properties through microhardness testing of specimens subjected to different treatments.

2. Materials and methods

The material used in this study is the aluminum alloy 2024 RAM2, an Al-Cu-Mg alloy enhanced with titanium and ceramic particles as inoculants. The chemical composition was found in literature and reported in Table 1. The printing parameters, including laser power and scan speed, were optimized to achieve a density greater than 99.5%. Density analyses were conducted visually by maximizing contrast and measuring the volumetric fraction of voids.

Table 1 – Chemical composition in % w of the 2024 RAM2 alloy [9].

	Al	Cu	Mg	Mn	Fe	Ti	Si
Nominal	Bal.	3.68	1.47	0.57	0.1	2.43	0.12

To analyze the behavior of inoculants and determine the optimal temperatures for heat treatments, Thermo-Calc software was utilized, specifically employing the TCAL8 v8.2 database for aluminum alloys.

Microhardness tests were employed to monitor the effect of heat treatments using the Vickers microhardness test method with a 50 g load and a 15 s dwell time of. For each sample, six measurements were taken: two in the upper region, two in the middle region, and two in the region closest to the build plate. This approach ensured a comprehensive characterization of the material's hardness across z direction (building direction), providing valuable insights into the variations induced by the process.

For the investigation of microstructure, a Nikon Eclipse LV150N optical microscope and a Hitachi TM3000 scanning electron microscope (SEM) were employed. These instruments enabled detailed examination of the microstructural features, such as grain morphology, phase distribution, and the presence of defects, at various length scales. By combining optical microscopy and SEM analysis, a thorough understanding of the microstructural evolution induced by the heat treatment processes was achieved.

The preparation of samples for metallographic analysis involved grinding and polishing to achieve a mirror-like finish using an automatic machine. Subsequently, a colloidal silica solution with nanometric particles was applied. Chemical etching was performed on some representative samples using Keller's acid.

As-Built condition was adopted as the baseline condition to evaluate the initial microstructure and mechanical properties obtained directly from the LPBF process. To enhance the mechanical properties, samples were subjected to three different heat treatment conditions:

1. Annealing and Aging (T6) Treatment:
    - The sample were heated to solubilization temperature to dissolve as many alloying elements as possible into the solid solution. Solubilization is the first step of T6 heat treatment.
    - After solution treatment, the samples were artificially aged. This T6 treatment protocol is commonly used in both scientific literature and industrial processes to improve the mechanical properties of aluminum alloys.
2. Solution treatment Only:
    - Another sample was subjected only to solubilization treatment to investigate the effect of solution treatment alone on the microstructure and mechanical performance.
3. Direct Aging (T5) Treatment:
    - The final set of samples was directly artificially aged. This treatment aimed to maintain the microstructure obtained through the LPBF process while increasing the quantity of precipitates to enhance mechanical properties.

3. Results and Discussion

3.1 Microstructure

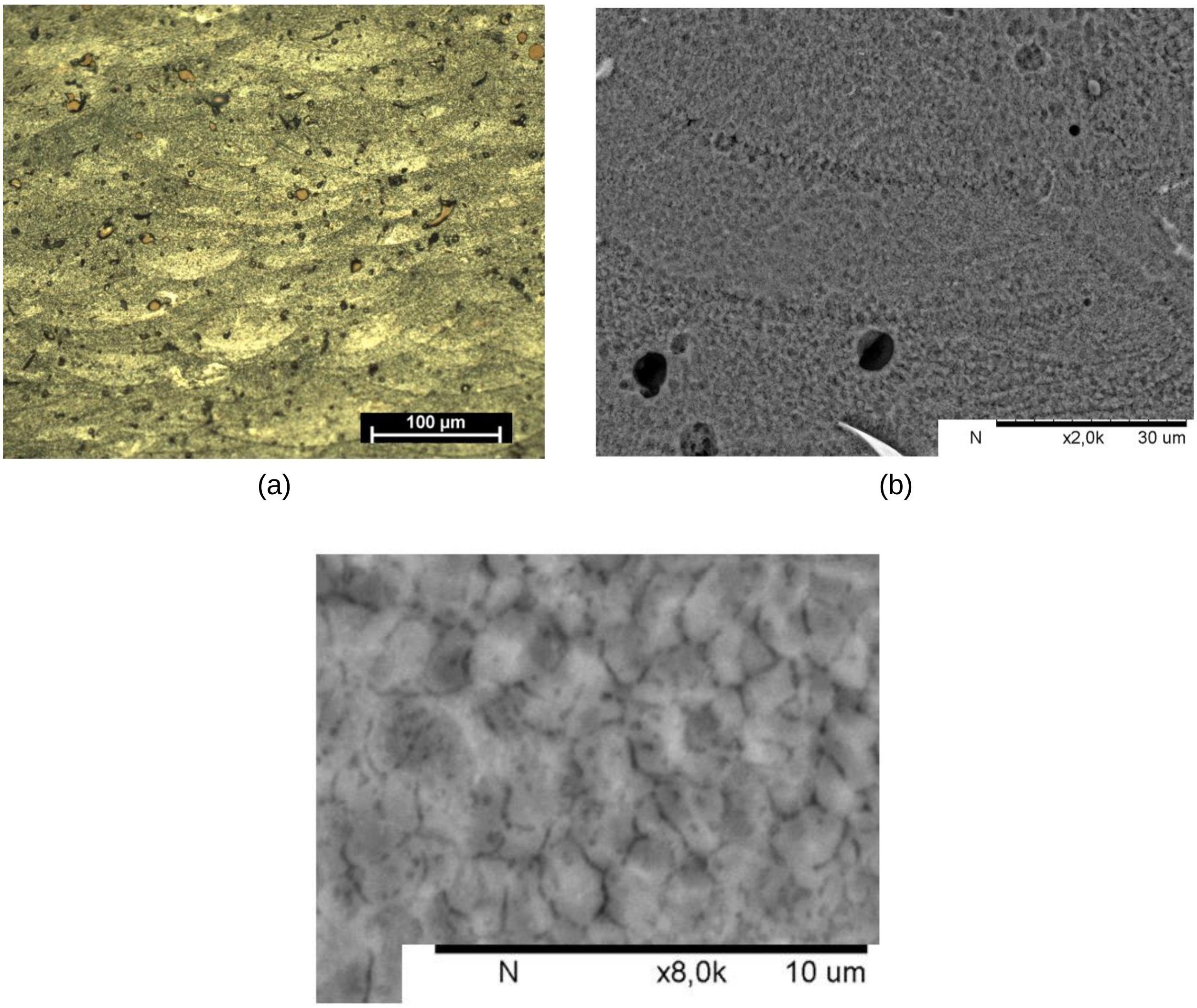
3.1.1 As-built

For the as-built sample, Figure 1a, captured under an optical microscope, reveals the typical melt-pool structure characteristic of materials produced through LPBF. The structure does not exhibit hot cracks and is nearly free of micropores. Grain sizes were measured using ImageJ software, with an average diameter of 1 µm. The microstructure is characterized by extremely fine grains, probably due to the “in-situ” reaction of titanium or ceramic particles during printing process [10]. This will be the subject of future studies to expand the comprehension on this specific material.

At higher magnifications, taken with SEM, brighter regions can be observed at the grain boundaries (Figure 4a). These regions are rich in copper and magnesium, which segregated during solidification, as reported by Konecna et al. [9]. This network can significantly improve tensile properties and Vickers micro-hardness [11].

Additionally, within many grains, black spots are frequently observed (see Figure 1b). Other studies have identified these regions as titanium-rich zones [9], which serve as nucleation sites and contribute to the formation of very fine and equiaxed grains. These titanium-rich zones play a crucial role in refining the microstructure and enhancing the mechanical properties of the alloy.

Moreover, the microstructure also reveals the presence of unfused titanium particles during the material printing, characterized by a regular spherical shape, as well as ceramic material particles exhibiting a more irregular shape and greater size variations. This heterogeneity in particle morphology may influence the local material properties and requires further investigation to understand its impact on the overall performance of the alloy.



(a) (b)

(c)

Figure 1 – Optical (a) and SEM (b, c) images at different magnifications of as-built samples.

3.1.2 Solution treatment and T6

The Al 2000 series alloys are typically subjected to a solution treatment to dissolve as many alloying elements as possible into solid solution. Subsequently, an artificial aging process is conducted to create nanometric precipitates that contribute to the enhancement of mechanical properties. From Thermo-Calc simulations, it was possible to derive the equilibrium melting temperature, which for the alloy composition given in Table 1 is 510°C. No chemical interaction is imposed by the simulation between metal matrix and ceramic material. To dissolve as many elements as possible into solution, an annealing temperature slightly belove the calculated equilibrium melting temperature was chosen to maintain a safety margin and avoid partial melting of the samples. However, from the graphs reported in Figure (2), it can be seen that most of the S (Al_2CuMg) and θ (Al_2Cu) phases can be dissolved even at lower temperatures.

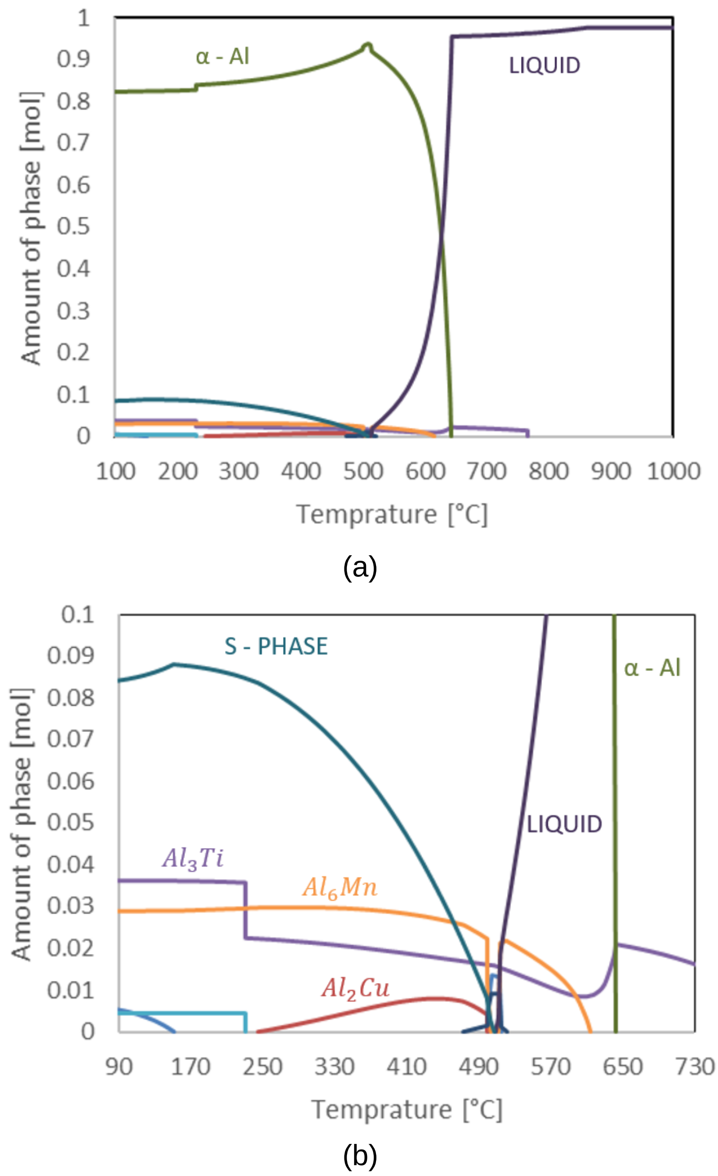


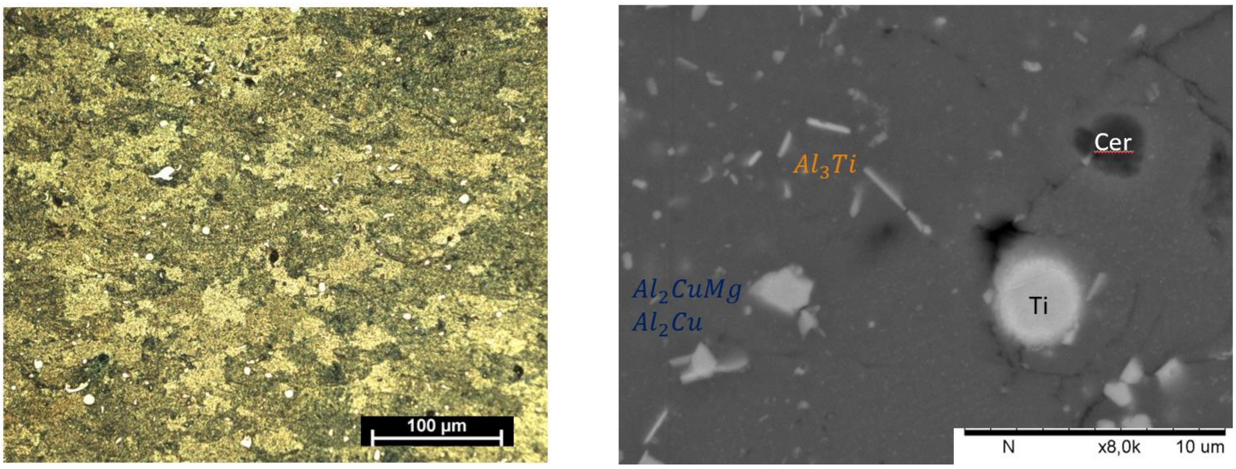
Figure 2 – Amount of phases of 2024Ti alloy against temperature, under equilibrium. (a) comprehensive plot and (b) magnification to highlight secondary phases behavior.

After solution treatment, a significant change in the structure can be observed in Figure (3). In some areas, the structure remains like that of the as-built state, while in others, significant growth occurs, with some grains reaching hundreds of micrometers. They form during the solution process and exhibit a randomly oriented crystallographic orientation [9]. The images also show how the globular titanium particles and the ceramic reinforcement remain present within the microstructure even after solution treatment, as can be seen in in Figure 3b.

At higher magnifications, it can be observed that the network structure present at the grain boundaries in the as-built sample has disappeared. After the solution treatment, some of the precipitates returned to the solid solution while the remaining precipitates agglomerated in certain areas of the matrix. After the T6 treatment, two types of precipitates can be noted: irregular ones, which are associated with the S and θ phases, and needle-like ones, which are typical of titanium-based compounds (e.g., Al_3Ti).

Comparing Figures 4c and 4d, which show the difference between a solution-treated sample and a fully T6-treated sample, aging is responsible for a significant increase in the quantity and size of the S and θ precipitates in the matrix. Figure 3b shows two other phases within the material: the rounded titanium particles remain nearly unchanged. This is because the solubilization temperature is too low to dissolve titanium particles that did not fuse during the printing process. The same applies to the ceramic particles, which have even higher melting points. These "foreign bodies" within the matrix hindered the homogenization process during the solution treatment, leading to a biphasic structure shown in Figure 3a.

These images demonstrate that the solution treatment is unable to homogenize the highly complex structure formed by this RAM2 2024 aluminum alloy produced with LPBF. In fact, this biphasic structure, with regions of very fine grains and coarse grains, could create issues for the long-term durability of the material, especially in fatigue testing and resistance to corrosive environments. Future developments are needed to explore these aspects.

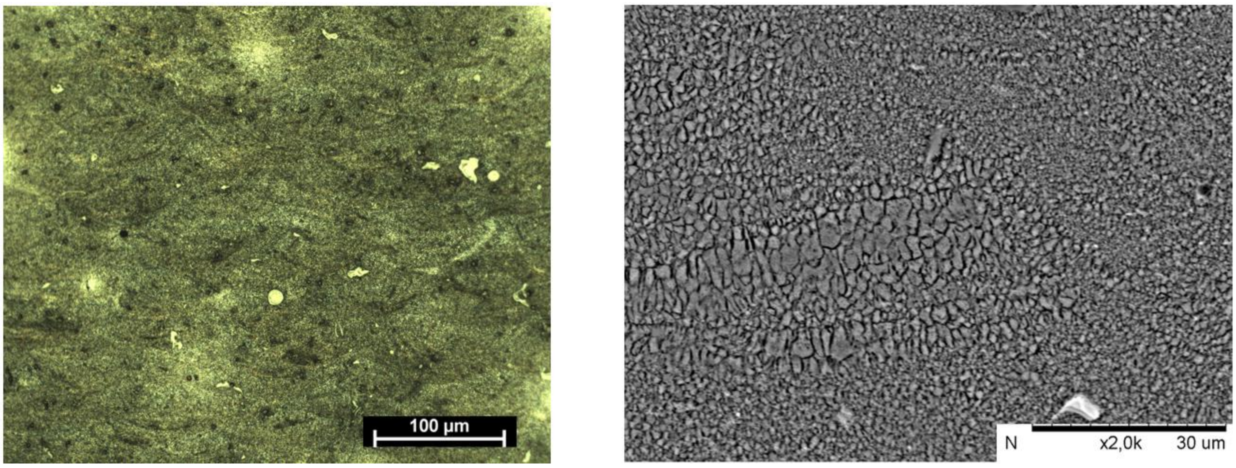


(a) (b)

Figure 3 – T6 sample: (a) optical image on chemical etched sample, (b) SEM high magnification image.

3.1.3 Direct aging

After the T5 treatment, a different microstructure from that obtained with the T6 treatment is observed in Figure 4. The chemically etched sample images show an almost complete disappearance of the melt pools; they are no longer easily distinguishable as in the as-built samples. However, at the grain level, there is substantial retention of the fine microstructure achieved through the LPBF process, with only a few rare areas exhibiting noticeable grain growth compared to that seen with the T6 treatment.



(a) (b)

Figure 4 – T5 sample: (a) optical and SEM (b) images on chemical etched sample.

The grain structure remains predominantly fine, likely due to the shorter aging duration and the lower temperature compared to the T6 treatment, which limits significant grain growth. The T5 treatment effectively maintains the desirable fine-grained structure produced by the LPBF process while increasing the quantity of precipitates within the microstructure. This increase in precipitates can contribute to improved mechanical properties by providing additional obstacles to dislocation movement, thereby enhancing the material's strength and hardness.

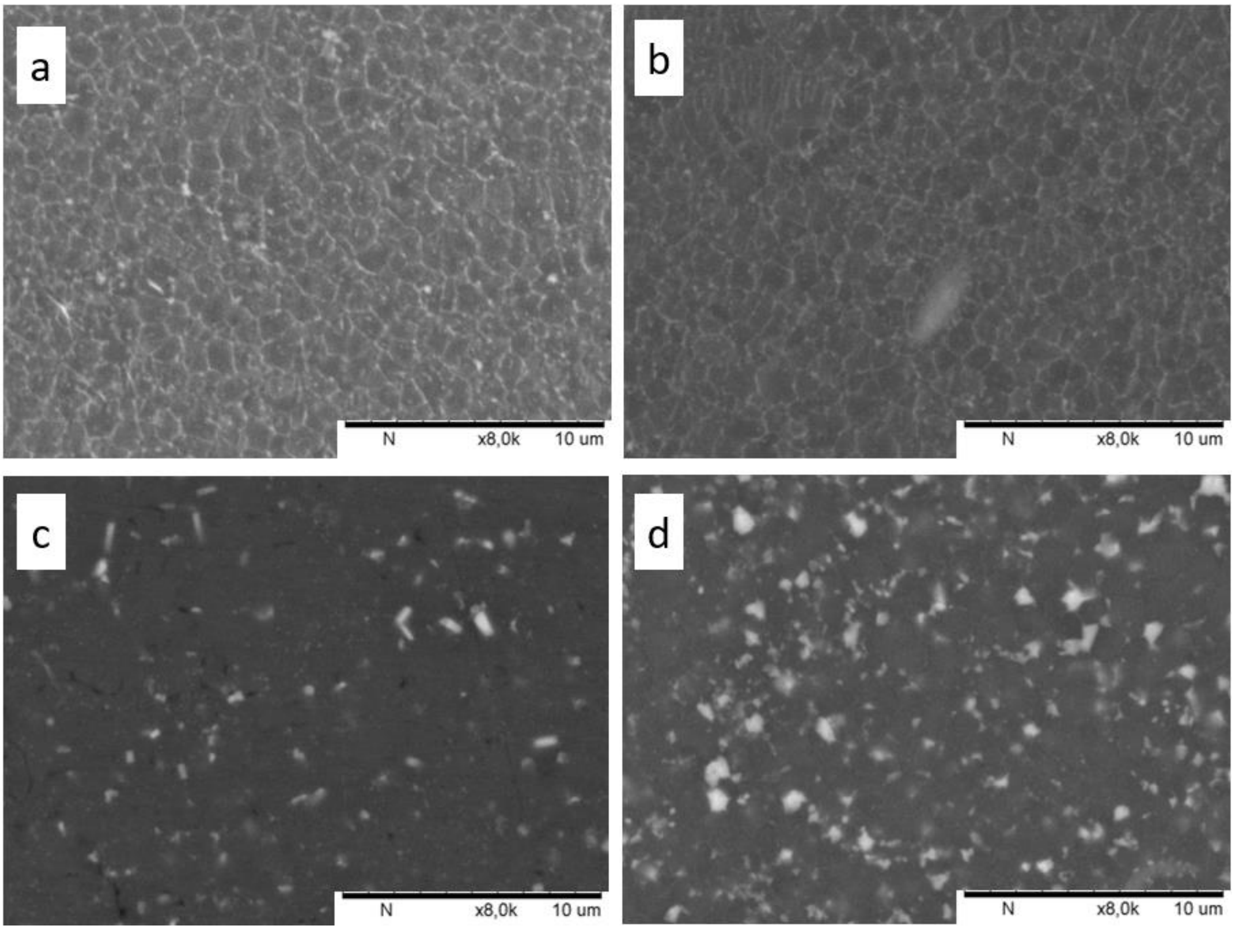


Figure 5 – SEM images captured at the same magnification. (a) As-built sample, (b) direct aging, (c) only solution treated and (d) solution treated + aging.

3.2 Microhardness

The microhardness results for the four samples in different conditions (as-built, T6, solution treated only, and T5) are presented below in Figure (6).

Comparing the microhardness values across the different treatments provides insights into the effectiveness of each process in changing the mechanical properties of the aluminum alloy 2024.

1. As-Built Condition:
    - The as-built sample exhibits a microhardness of 172.2 ± 12.9 HV. This is a notably high value for an as-built aluminum alloy and is likely due to the very fine microstructure and the presence of second phases of very hard materials such as titanium and ceramic particles. These factors contribute significantly to the overall hardness by providing barriers to dislocation movement.
2. T6 Treatment:
    - The T6-treated sample shows an average hardness of 155.4 ± 16.7 HV, with a greater dispersion in the values. This lower and more variable hardness can be explained by the

partial grain growth observed in this condition, which results in a reduction of mechanical properties. The existence of different zones, where grains range from the order of hundreds of micrometers to areas with grains still in the micrometer range or even smaller, contributes to the increased dispersion of hardness values. The varying microstructures within the sample lead to inconsistent resistance to indentation, thereby increasing the variability of the hardness measurements.

3. Solution Treated Only:
   - The solution-treated only sample demonstrates hardness values of 150.3 ± 17.8 HV, slightly lower than those of the T6 condition. This confirms the increase in precipitates observed in Figure (6), which occurred during the aging process. Solution treatment alone contributes to some increase in hardness by dissolving alloying elements into the solid solution. However, the absence of subsequent aging means fewer precipitates are formed, resulting in lower hardness compared to the T6-treated sample.

4. T5 Treatment:
   - The T5-treated sample shows a hardness of 172.5 ± 4.2 HV, a value very similar to that of the as-built condition. This similarity in hardness is confirmed by images showing grain sizes that are perfectly comparable to those in the as-built sample. The reduction in the dispersion of results can be attributed to the partial elimination of the melt pools, leading to a more uniform microstructure, and thus reducing the variability between the central and peripheral zones.

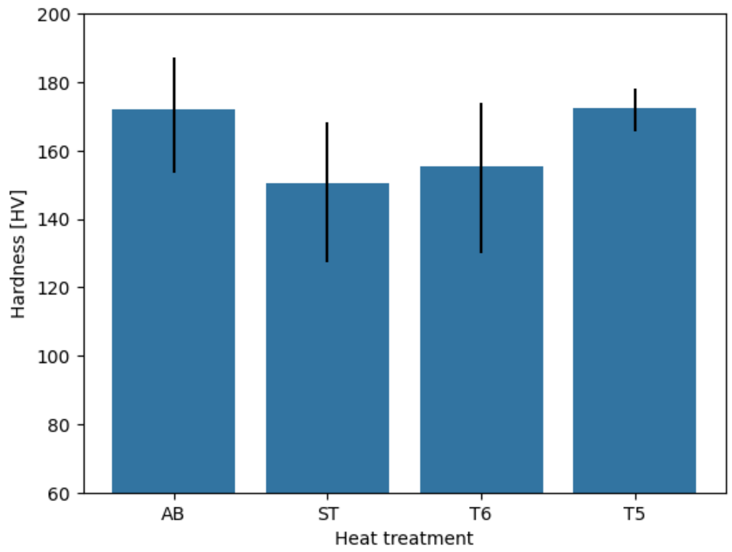


Figure 6 – Microhardness measures of samples treated in different condition.

4. Conclusions

This study investigated the effects of different heat treatments on the microstructure and mechanical properties of aluminum alloy 2024 RAM2 loaded with titanium and ceramic particles produced via Laser Powder Bed Fusion (LPBF). The key findings and conclusions are summarized as follows:

- A very fine as-built microstructure was achieved, with grain sizes around 1 μm. This equiaxed grain structure was facilitated by the presence of titanium and ceramic particles, which acted as inoculants. These inoculants improved printability by preventing the formation of hot cracks, resulting in a uniform and high-quality material.
- The T6 heat treatment resulted in partial homogenization of the microstructure. While some regions retained very fine grains, other areas experienced significant grain growth, with grains expanding by hundreds of times. This heterogeneous grain size distribution affected the overall uniformity of the material.

- The T5 treatment effectively preserved the fine-grained structure achieved through the LPBF process. This treatment maintained the beneficial microstructural characteristics obtained during production, leading to a uniformly fine-grained material.
- Heat treatments involving solution treatment led to a loss in mechanical properties due to grain coarsening. The larger grain sizes resulted in decreased hardness values. In contrast, the T5 treatment managed to maintain the mechanical properties of the as-built condition. The reduced variability in performance was likely due to the homogenization of the microstructure, which minimized differences between central and peripheral regions of the melt pools.

These findings highlight the significant influence of heat treatment processes on the microstructure and mechanical properties of LPBF-produced aluminum alloy 2024. The T5 treatment demonstrates a promising approach for maintaining fine microstructures and consistent mechanical performance.

In conclusion, the T5 treatment offers a promising alternative to the conventional T6 treatment for LPBF-produced aluminum alloy 2024 RAM2. It maintains the fine microstructure obtained from the LPBF process while reduces the variability due to its complex microstructure. Further optimization of heat treatment parameters could lead to even better performance, making this approach highly suitable for applications requiring high strength performance.

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6. Copyright Statement

7. Acknowledgements

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