Effect of heat treatment temperature on the microstructure and microhardness of TiC/Ti6Al4V composite manufactured with laser metal deposition

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Abstract. This study presented the investigation of the influence of post heat treatment temperature on the microstructure and hardness of TiC/Ti6Al4V composite manufactured with laser metal deposition. Heat treatment was performed to improve the microstructural homogeneity. It was found that the addition of TiC into Ti6Al4V results in the formation of a Widmanstätten microstructure with different grain sizes on the matrix. Heat treatment of the TiC/Ti6Al4V promoted phase transformations from acicular in the as built to lamella ($\alpha + \beta$) and equiaxial phases in the heat-treated samples. The 900°C heat treated sample showed a uniform distribution of $\alpha$ and $\beta$ phases, and 1100°C showed an increase in $\beta$ phases, which resulted in an equiaxial microstructure. Moreover, Heat treatment at 1100°C resulted in the highest microhardness of 665±13HV.

1 Introduction

Titanium and its alloys such as Ti6Al4V have shown their wide commercial importance for many industries including their applications in aerospace, biomedical and healthcare, and power and energy. In the aerospace industry application, Ti6Al4V components with dual phase $\alpha + \beta$ alloy, are used extensively due to their high specific strength, which leads to reduced weight and space savings [1]. The Ti6Al4V alloy used in aerospace have shown to contain low surface hardness, density, and wear resistance [2,3] and due to these poor mechanical properties, titanium matrix composites (TMCs) were established to strengthen the properties of titanium alloys and make them ideal for high temperature applications in the aerospace industry [4]. The TMCs are a class of metal matrix composites that are considered potential candidate materials for high temperature applications in aerospace due to their good combination of better wear resistance, high hardness of ceramic and high temperature durability [5]. Various research has proven the advantages of reinforced TMCs and their significant applications in aerospace, automobile, and biomedical fields amongst others [5,6].

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Additive manufacturing (AM) has been explored as potential manufacturing methods to produce the TMCs, due to their advantages over conventional methods such as materials and cost savings, reduced production time and ability to manufacture complex structures. Laser metal deposition (LMD) is a type of direct energy deposition (DED) AM technique, which involves manufacturing of 3D structures with high laser power that is used to melt the layers of metallic powder [7]. Because of its high flexibility process the LMD technique can manufacture new parts, and rebuild damaged components, in addition to preparing corrosion resistant coatings. However, LMD components have proven to show high residual stresses and defects such as cracks and pores, which eventually affect performance of the manufactured part [8]. The presence of pores and cracks in the LMD parts eventually degrade the mechanically properties, and thus leading to need of post processing techniques such as heat treatment and hot isostatic pressing (HIP) [9,10]. These residual stresses also cause a microstructure anisotropy of the LMD components, which gives a rise to inhomogeneous microstructure [11].

Heat treatment is regarded as a post processing technique that can be used to improve the microstructure of components, in so doing homogenizing the microstructure while also relieving the internal residual stress [11]. When adjusting the heat treatment temperature, the microstructure in the as built components is transformed and thus improved mechanical can be achieved. Various studies on heat treatment of Ti6Al4V have shown that this post processing technique is able to modify the microstructure of Ti6Al4V to the desired one for different industrial applications [13, 14]. Although this is the case, there are few studies on the impact of heat treatment on the microstructure properties of TiC/Ti6Al4V composite.

From a study conducted by Peterson, et al [12], post processing heat treatment was investigated on how it affects the microstructure of laser power bed fusion (LPBF) Ti6Al4V with dissolved TiC particles. The LPBF was used in their study to dissolve 0.75% TiC in grade 23 Ti6Al4V matrix, using 160W laser power and laser speed of 450mm/s. The hatch width was 110 µm and the layer thickness was 20 µm. Heat treatment was performed in a vacuum furnace at a heat rate of 20°C/s and various temperatures ranging from 500°C to 1100°C. It was reported that heat treatment at 500°C for 1 hour was suitable for high strength applications. However, at temperature of 700°C and residence time of 3 hours, there was a good balance of increased strength and ductility of the sample. The improved strength is attributable to slight grain refining. Additionally, the results indicated that grain growth was promoted during heat treatment because of the dissolved carbon that refines the martensitic grain size. Based on the above findings, this study aimed at investigating the impact of the post heat treatment on the microstructure of TiC/Ti6Al4V composite produced using LMD.

## 2 Research Methodology

### 2.1 Materials

Grade 5 Ti6Al4V powder with particle size distribution in the range of 45-100 µm, supplied by TLS Technik GmbH & Co company was used as the matrix for printing the samples. The TiC powder with particles size in the range of 45-100 µm, supplied by Sabinano company, was used as a reinforcement material during deposition. The samples were manufactured on a Ti6Al4V base plate and the morphologies of the Ti6Al4V and TiC powders are shown in Figure 1a and 1b, respectively. The Ti6Al4V alloy indicates spherical shaped surface, and the TiC shows irregular shaped surface (see Figure 1a and 1b). A similar observation was reported in other studies [15]. These irregular shaped particles of TiC were also reported in literature [15].
2.1 Literature

Reports of other studies have shown that the TiC phase shows irregularly shaped surface, as shown in Figure 1.

**Figure 1**

(a) Ti6Al4V base plate with TiC powder with particles size in the range of 45-200 μm. (b) SEM images showing grain size of TiC with a spherical-like appearance. The TiC powder was found to be 1.3 μm in diameter. The TiC particles were found to have a high density and a high probability of being used as a matrix material in different applications. Even though this may be the case, there are few studies on the impact of heat treatment on the microstructure of TiC/Ti6Al4V composite produced using Laser Metal Deposition (LMD).

2.2 Experimental procedure

The samples were produced using a LMD technique, where the deposition process was achieved by IPG Ytterbium Fiber laser fitted to the KUKA robot with 3-way nozzles. The processing parameters that were used during LMD are summarised in Table 1. The Ti6Al4V and TiC powders were delivered through two powder feed hoppers, using powder carrier gas that was blown at 1.5 l/min during the deposition process. Argon gas was used as a shielding gas to prevent oxidation on the samples, and was blown at 15 l/min. The mass percentages of Ti6Al4V and TiC powders was 95 % and 5 %, respectively. A laser energy density of 90 J/mm² was used to manufacture the samples.

<table>
<thead>
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<th>Parameters</th>
<th>Values</th>
<th>Units</th>
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<tr>
<td>Laser energy density</td>
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<td>J/mm²</td>
</tr>
<tr>
<td>Carrier gas</td>
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<td>l/min</td>
</tr>
<tr>
<td>Shielding gas</td>
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<td>m/min</td>
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<td>Mass % of Ti6Al4V</td>
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<td>%</td>
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<tr>
<td>Mass % of TiC</td>
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</table>

Post heat treatment was applied to the manufactured sample to homogenise the microstructure. Heat treatment was conducted at Council for Scientific and Industrial Research (CSIR) in a tube furnace, under argon protected atmosphere to protect the samples from reacting with oxygen. The samples were heat treated at temperatures 950°C and 1100°C for resident time of 2 hours. A constant heating rate of 10°C/min was used during all heat treatments and cooled down by furnace cooling (FC) method. The heat treatment process parameters are presented in Table 2.

<table>
<thead>
<tr>
<th>Sample no</th>
<th>Temperature (°C)</th>
<th>Residence time (hr)</th>
<th>Heating rate (°C/min)</th>
<th>Cooling method</th>
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</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>900</td>
<td>2</td>
<td>10</td>
<td>FC</td>
</tr>
<tr>
<td>Sample 2</td>
<td>1100</td>
<td>2</td>
<td>10</td>
<td>FC</td>
</tr>
</tbody>
</table>
The produced samples were later cut, mounted, ground, polished, and etched as part of sample preparation. The samples were cut into smaller samples (for easy handling purposes) for characterization, using a Struers Labotom-5 cutting machine that was available at the CSIR. The samples were mounted on AMP 50 automatic mounting press machine to obtain the required size and shape for analyses. An Aka Resin Phenolic SEM black conductive resin was used to mount all samples. The mounted samples were mechanically ground using the Struers Tegrapol-25 grinding and polishing machine that is available at CSIR. Silicon carbide (SiC) grinding papers from grit sizes 80, 320, 1200 to 4000 were used for grinding the samples, and polishing of the samples was conducted with Diapro MD-Mol 3 μm diamond suspension for 3 min and colloidal silica 0.04 μm OP-S suspension for 3 min as the final polishing stage. The samples were prepared for microscopy by etching with a solution consisting of 100 mL H2O, 1-3 mL HF, 2-6 mL HNO3 (Kroll's reagents), and etching time was between 3-8 seconds, cleaned with water and dried with ethanol.

The microstructure of the produced samples was studied using Olympus BX51M optical microscope (OM). For an overview microstructural representation, the images were taken at top, middle and bottom regions of the surfaces analysed respectively. A Matsuzawa Seiko Vickers MTH-1 microhardness model machine was used to measure the microhardness of the samples using a force of 300gf and dwell time of 10 seconds. Three hardness patterns with over 17 indentations were taken, and the average hardness of the material was calculated. The phases of the TiC/Ti6Al4V composite were analysed by X-Ray Diffraction (XRD) machine.

3 Results and discussion

3.1 Microstructure Analysis

3.1.1 Microstructure analysis of the as built sample

Optical microscope images of the top region of as built sample are illustrated in Figure 2. Various sizes of irregular grains were observed on the sample as shown in Figure 2a. The grains appeared small, medium, and large in sizes. Small irregular particles were also observed at the grain boundaries. The sample was also composed of unmelted particles that are unevenly distributed throughout the material. The unmelted particles vary in size and colour. At higher magnification, unmelted TiC particles with defects were found as described in Figure 2b. According to Wang, et al [16], defects in the unmelted particles indicate that the particles were not fully melted because of the high melting temperature of TiC (~3067°C), thus they become partially dissolved into the molten pool at high temperature. Long fine morphologies were observed on the matrix of the microstructure, which were identified as acicular morphology from literature study by Peterson, et al [12]. According to Yu, et al, [17], the acicular structure forms due to the rapid cooling that occurs in the melt pool during deposition. A coarse Widmanstätten /basket weave structure was also observed on the matrix of the sample. Similar results were obtained in literature studies by [8] and [16]. Similar microstructure properties were observed at the middle and bottom section of the as-built sample as shown in Figures 3 and 4, respectively. Figure 3 indicates the OM images of the as-built samples at the middle section, while Figure 4 shows the OM images of the as-built samples at the bottom section. However, fine Widmanstätten was only found at the bottom section of the as-built sample.
Similar results were obtained in deposition. A basket weave microstructure was found on the matrix of the microstructure of the as-built sample. Figure 2 indicates the optical images of the as built sample. Various sizes of irregular grains were observed on the sample as shown in Figure 2a. The acicular structure forms due to the rapid cooling that occurs in the melt pool during laser welding. According to literature studies, unmelted TiC particles with defects were also observed at the grain boundaries. However, fine defects in the unmelted particles indicate that they are not fully melted because of the high melting temperature of TiC (~3067°C). For the microstructure of large, medium and small irregular grains, the matrix structure is a coarse Widmanstätten/basket weave. The acicular morphology is found on the matrix of the as built sample. Figures 3 and 4 illustrate in an overview the microstructural patterns of the as-built sample. Results and discussion

Fig 2. Optical images of as built TiC/Ti6Al4V at top region of the polished surface at (a) low magnification and (b) high magnification

Fig 3. Optical images of as built TiC/Ti6Al4V in the middle region of the polished surface at (a) low magnification and (b) high magnification

Fig 4. Optical images of as built TiC/Ti6Al4V at the bottom region of the polished surface at (a) low magnification and (b) high magnification
3.1.2 Microstructure analysis of sample heat treated at 900°C for 2 h and FC.

Figure 5 shows the microstructure of the top region of polished surfaces of sample heat treated at 900°C for 2 h followed by FC to ambient temperature. The unmelted particles with defects were still evident on the microstructure of 900°C heat treated sample as shown in Figure 5a. This means that the particles were not melted during 900°C heat treatment temperature because of the high melting point of TiC as explained in section 3.1.1. Small irregular particles were found to be randomly distributed in the matrix, forming a linear pattern throughout the sample as presented in Figure 5b. At higher magnification, the matrix also showed the presence of small grain sizes.

![Figure 5](image)

Fig 5. Top region optical images of 900°C heat treated at (a) low magnification (b) higher magnification.

The middle section of the 900°C heat treated sample is described in Figure 6. A similar observation to the top middle section of the sample was shown in this section. The presence of unmelted TiC particles and small irregular particles that form lines is evident. However, the small irregular grains were hardly visible on the matrix of the sample.

![Figure 6](image)

Fig 6. Middle region optical images of 900°C heat treated TiC/Ti6Al4V at (a) low magnification and (b) higher magnification.
3.1.2 Microstructure analysis of sample heat treated at 900°C

Figure 5 shows the microstructure of the top region of polished surfaces of sample heat treated at 900°C for 2 h followed by FC to ambient temperature. The unmelted particles with defects were still evident on the microstructure of 900°C heat treated sample as shown in Figure 5a. This means that the particles were not melted during 900°C heat treatment temperature because of the high melting point of TiC as explained in section 3.1.1. Small irregular particles were found to be randomly distributed in the matrix, forming a linear pattern throughout the sample as presented in Figure 5b.

Figure 7 shows optical images of the 900°C-heat treated sample at bottom region. The images also displayed the presence of unmelted TiC and small irregular particles, like the top and middle areas discussed in the preceding paragraphs.

3.1.3 Microstructure analysis of sample heat treated at 1100°C for 2 h and FC.

Figure 8 showed the top section of the 1100°C heat treated sample. In the top section, the reduced amount of the unmelted TiC particles was observed compared to the as built sample. As stated by Wang, et al [16], when the temperature is increased, there is enough time and heat for the TiC particles to dissolve, thus few of the unmelted TiC particles were seen in the top section. Small irregular grains were unevenly distributed on the matrix. Small irregular particles formed lines that were in random directions on the sample. According to Peterson, et al [12], an increase in heat treatment time and temperature causes the grains to grow faster in all directions, resulting in increased average grain sizes. However, in this paper, when the temperature was increased to 900°C and 1100°C heat treatments, grain sizes reduced from large, medium, and small in as built to small grains in the 900°C and 1100°C heat treated.
In Figure 9, the optical microscopy images in the middle section of the 1100°C-heat treated sample are presented. It was found that the microstructure still contained unmelted TiC particles that were randomly distributed on the matrix. Medium and small irregular grains were obtained, along with the elongated equiaxial grains. According to Brandl, et al [18], furnace cooling after heat treatment at high temperatures may lead to a transformation of the acicular phase into an equiaxial phase. This transformation is observed in Figure 8, where the microstructure changed from acicular in the as built as presented in Figure 2 to long/equiaxial structures. Additionally, a study by Qian, et al [19] presented that the FC rate decreases as the sample heats up, which results in the formation of equiaxed grains.

![Optical images in the middle section of 1100°C heat treated sample at (a) low and (b) high magnifications.](image)

Optical image results of the bottom section of the 1100°C heat treated sample is illustrated in Figure 10. Similar observations to the middle section are evident in this region, i.e., small irregular particles forming a line and the equiaxial phase on the matrix.

![Optical images in the bottom region of 1100°C heat treated sample at (a) low and (b) high magnifications.](image)

The sample heat treated at 1100°C showed different microstructural characteristics compared to the as built to 900°C samples. The morphology of the as built changed moving from the top regions to the bottom of the sample. Comparing top region (Figure 2) and the bottom region (Figure 4) of the as built sample, the top section contained a combination of large, medium, and small grains, while the bottom had medium grains only. Again, in the top and
middle regions, a coarse Widmanstätten structure was observed on the matrix, while the bottom part contained both fine and coarse Widmanstätten microstructure on its matrix. From these observations, the as built sample was then considered to have an inhomogeneous microstructure. The sample was then heat treated at 900°C and 1100°C and cooled for 2 hours furnace cooling. The matrix of the 900°C heat treated sample indicated a distribution of small irregular particles forming lines throughout the sample (from top to bottom). The unmelted particles in this sample appeared to have more defects, indicating they were partially dissolved as the temperature was increased. The 900°C heat treated sample appeared to have a uniform microstructure. When analysing the 1100°C heat treated sample, the sample showed medium and small irregular grains in the middle section, as shown in Figure 9. Again, the top region presented small irregular grains on its matrix, whereas the bottom region had elongated grains that are identified as equiaxial microstructures. The microstructure of the 1100°C heat treated sample was therefore found to be inhomogeneous.

3.2 Phase analysis

The XRD analysis of the LMD as built and heat-treated TiC/Ti6Al4V samples is discussed in this section. In all three samples, the XRD patterns showed the presentation of three phases, namely, alpha titanium (α/α'-Ti), beta titanium (β-Ti), and titanium carbide (TiC). Similar XRD profiles were recorded in literature [21, 22, 23]. Figure 11 shows the XRD pattern of as built TiC/Ti6Al4V sample. The peak position of 2θ value of 42.28° is presenting α/α'-Ti, while the 2θ = 40.56° presents β-Ti. Few TiC particles are also shown in the pattern as indicated by figure 11. Also, unknown peaks are recorded at 2θ value of ~ 36.5°.

![XRD pattern of as built TiC/Ti6Al4V sample](image)

The phase composition after 900°C heat treatment and 2 hours, FC, is shown in figure 12. It was found that the peaks’ intensity was reduced after heat treatment at 900°C. This observation may be associated with the reduction in TiC phases because of the post heat treatment, as shown in figure 12. A shift in the peaks of the α/α'-Ti belonging to 2θ = 42.28° and β-Ti 2θ = 40.56° is observed after heat treatment at 900°C. The α/α'-Ti is present at 2θ value of 41.05°, and β-Ti at 2θ value of 40.70°. Also, it was found that new α/α'-Ti is formed
at $2\theta = 54.67^\circ$, suggesting that heat treatment promotes the formation of $\alpha/\alpha'$-Ti phases. According to a binary phase diagram plotted by Li et al [21], at $\beta$-transus temperature (~920°C), the $\beta$-Ti phase transforms into $\alpha$-Ti, promoting the growth of $\alpha$-Ti phases in TiC/Ti6Al4V composites. Another new unknown phase is recorded just near the $\alpha$-Ti, at $2\theta$ value of approximately 42.76°.

![Figure 12. XRD profile of the 900°C heat treated sample.](image)

Figure 13 shows the XRD profile of the 1100°C heat treated sample. Interestingly, the intensity peak of the $\alpha/\alpha'$-Ti to 40.56° shifted to $2\theta$ value of 40.46° after heat treatment at 1100°C. The peak shift was attributed to phase transformation. Additionally, it is important to note that it is typical to observe more peaks of the alpha titanium ($\alpha/\alpha'$-Ti), which is because of the slow cooling process associated with the study. A new peak of TiC is found at approximately $2\theta = \sim 38.41^\circ$. In addition, the TiC phase recorded at $2\theta = \sim 61.38^\circ$ in as built has interestingly disappeared after heat treatment of 1100°C.
The XRD profiles of the LMDed and heat treated TiC/Ti6Al4V samples are revealed in Figures 11-13. The peaks belonging to α-Ti, β-Ti and TiC are clearly shown and identified in the figures. For the as built, the microstructure illustrates the presence of high intensity peaks, which is supported by the high peaks of α/α’-Ti in Figure 11. The 900°C heat treated sample presents a phase transformation from α phase to α+β, attributing to the decreased TiC peaks in Figure 12. At 1100°C heat treatment for 2 hours FC, a new peak of TiC is present at $2\theta = ~38.41°$. Unknown phases are detected in all three XRD patterns and further analysis is being done to identify them.

### 3.3 Microhardness profile

The microhardness profiles of the as built and heat-treated samples are presented in Figure 14. The presence of unmelted TiC particles and the α acicular morphology in the as built resulted in a high microhardness of the material. A similar observation was reported in a study by Åkerfeldt et al [24], where a fine microstructure of the LMD Ti6Al4V was characterised by high strength and increased hardness.

A significant decrease in hardness profile was observed after heat treatment at 900°C, as shown in Figure 14 and Table 3. This was because of phase transformation from α to α + β as explained by the XRD results in Figure 12. And a reduction in residual stresses afforded by the heat treatment. Similar hardness profile results were reported by Bochetta, et al [25], where the lamellar (α + β) microstructure showed the lowest hardness profile of 394±19HV, compared to the α’ martensitic bimodal and equiaxed α microstructures, whereas the martensitic provided a hardness profile of 410±19HV. According to Cao et al [26], heat treatment at 730°C resulted in the transformation of the α phase into α + β lamellar microstructure, which promoted an increase in ductility, but the strength of the material was simultaneously decreased.

Furthermore, heat treatment at 1100°C and furnace cooling for 2 hours resulted in the highest hardness value of 665±13HV, compared to the as built and heat-treated sample at 900°C. This increase in microhardness was associated with the presence of unmelted TiC particles as depicted in Figure 10, as it is well known that TiC is a hard ceramic material.
Also, according to Wang et al. [27], TiC in the matrix have enough time to precipitate, promoting dislocation and thus high hardness is obtained.

The average microhardness of the samples is illustrated in Table 3. The as built sample provided an almost perfect linear hardness profile, with an average hardness of 584±19HV, as shown in Table 3. According to a study by Wang et al. [27], some TiC/Ti6Al4V composites fabricated by LMD achieved a microhardness of 500-550HV. This means that the TiC/Ti6Al4V composite used in this study achieved a higher hardness than the one in the study by Wang et al. The microhardness of the 900°C heat treated sample was recorded as 401±19HV. As explained in Figures 7 and 8, this was due to the reduced grain size of the partially melted TiC particles. The microhardness of the 1100°C heat treated sample was calculated as 665±13HV.

**Table 3: Average hardness of TiC/Ti6Al4V samples**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hardness (HV)</th>
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<tbody>
<tr>
<td>As built</td>
<td>584±19HV</td>
</tr>
<tr>
<td>900°C</td>
<td>401±19HV</td>
</tr>
<tr>
<td>1100°C</td>
<td>665±13HV</td>
</tr>
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</table>

### 4 Conclusion

In this study, the effect of heat treatment on the microstructure and microhardness of TiC/Ti6Al4V manufactured by LMD was investigated. The following conclusions arise from this work:

- Heat treatment of TiC/Ti6Al4V at both 900°C and 1100°C results in microstructural transformation from acicular martensitic into lamellae α+β.
- 900°C, 2 hours, FC results in a major decrease in hardness due to the produced α + β phases.
• Heat treatment at 900°C for 2 hours FC, improves the microstructural homogeneity of the TiC/Ti6Al4V composite.
• Heat treatment at 1100°C for 2 hours, FC, promotes an increase in hardness because of microstructural transformation in the presence of unmelted TiC particles.

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