Effect of heat treatment temperature on the microstructure and microhardness of TiC/Ti6AI4V 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 heattreated samples. The 900°C heat treated sample showed a uniform distribution of α and β phases, and 1100°C showed an increase in β 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 μ m and the layer thickness was 20 μ m. 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 μ m, 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 μ m, 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].



(a) (b) Fig 1. SEM images of (a) Ti6Al4V and (b) TiC powders.

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 1.5l/min during the deposition process. Argon gas was used as a shielding gas to prevent oxidation on the samples, and was blown at 15l/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.

Parameters	Values	Units
Laser energy density	90	J/mm ²
Carrier gas	1.5	l/min
Shielding gas	15	m/min
Mass % of Ti6Al4V	95	%
Mass % of TiC	5	%

Table 1: Experimental processing parameters of the LMD samples

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 residence 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.

Sample no	Temperature (°C)	Residence time (hr)	Heating rate (°C/min)	Cooling method
Sample 1	900	2	10	FC
Sample 2	1100	2	10	FC

 Table 2: Heat treatment process parameters

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 μ m diamond suspension for 3 min and colloidal silica 0.04 μ m 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 (\sim 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 course 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.



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.



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.



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

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.



Fig 7. Bottom region optical images of the 900°C heat treated TiC/Ti6Al4V at (a) low magnification and (b) high magnification.

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.



Fig 8. Optical images at (a) low and (b) high magnifications at the top region of 1100°C heat treated sample.

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.



Fig 9. Optical images in the middle section of 1100°C heat treated sample at (a) low and (b) high magnifications.

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.



Fig 10. Optical images in the bottom region of 1100°C heat treated sample at (a) low and (b) high magnifications.

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 course 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°.



Fig 11. XRD patterns of the as built TiC/Ti6Al4V sample

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 $2\theta = 42.28^{\circ}$ and β -Ti $2\theta = 40.56^{\circ}$ 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 α/α' -Ti phases. According to a binary phase diagram plotted by Li et al [21], at β - transus temperature (~920°C), the β -Ti phase transforms into α -Ti, promoting the growth of α -Ti phases in TiC/Ti6Al4V composites. Another new unknown phase is recorded just near the α -Ti, at 2θ value of approximately 42.76°.



Fig 12. XRD profile of the 900°C heat treated sample.

Figure 13 shows the XRD profile of the 1100°C heat treated sample. Interestingly, the intensity peak of the α/α' -Ti to 40.56° shifted to 2θ 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 (α/α' -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.



Fig 13. XRD patterns of the 1100°C heat treated sample

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 $\alpha+\beta$, 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 $2\theta = \sim 38.41^{\circ}$. Unknown phases are detected in all three XRD patterns and further analysis is being done to identify them.

3.3 4 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 $\alpha + \beta$ 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 ($\alpha + \beta$) 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 $\alpha + \beta$ 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.



Fig 3. Vickers hardness profiles of the as built and heat-treated TiC/Ti6Al4V samples

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.

Sample	Hardness (HV)
As built	584±19HV
900°C	401±19HV
1100°C	665±13HV

Table 3: Average hardness of TiC/Ti6Al4V samples

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 $\alpha+\beta$.
- 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.

The authors would like to thank the Department of Science and Innovation (DSI) for financial support through the Collaborative Program in Additive Manufacturing (CPAM), grant number HLM4BMX. We would also like to appreciate Mandy Madigoe 'assistance with conducting the heat treatment analyses.

References

- 1. R. Kolli and A. Devaraj, Metals (Basel), vol. 8, no. 7, p. 506, (2018)
- M. Waqas, D. He, Y. Liu, S. Riaz, and F. Afzal, *J Mater Eng Perform*, vol. 32, no. 2, pp. 680–694 (2023)
- 3. X. P. Li et al., Acta Mater, vol. 129, pp. 183–193 (2017)
- M. D. Hayat, H. Singh, Z. He, and P. Cao, *Comp Part A Appl Sci Manuf*, vol. 121, pp. 418–438 (2019)
- L. J. Huang, H. Y. Xu, B. Wang, Y. Z. Zhang, and L. Geng, *Mater in Engr*, vol. 36, pp. 694–698 (2012)
- 6. L.-C. Zhang, L.-Y. Chen, and L. Wang, Adv Engr Mater, vol. 22, no. 5 (2020)
- 7. M. Kalms and R. B. Bergmann, Addit Manuf, vol. 36 (2020)
- 8. A. Azarniya et al., J Alloys Compd, vol. 804, pp. 163–191 (2019)
- P. H. Li, W. G. Guo, W. D. Huang, Y. Su, X. Lin, and K. B. Yuan, Mater. Sci. Eng. C, A, vol. 647, pp. 34–42 (2015)
- A. Sterling, N. Shamsaei, B. Torries, and S. M. Thompson, *Procedia Engineering*, Elsevier Ltd, pp. 576–589 (2015)
- 11. M. Laleh et al., Prog in Mater Sci, vol. 133. Elsevier Ltd, (2023)
- J. Peterson, A. Issariyapat, J. Umeda, and K. Kondoh, *J Alloys Compd*, vol. 920, p. 165930, (2022)
- C. Zhang *et al.*, *Xiyou Jinshu Cailiao Yu Gongcheng/Rare Met Mater and Engr*, vol. 45, no. 12, pp. 3172–3176 (2016)
- 14. T. Vilaro, C. Colin, and J. D. Bartout, *Met Mater Trans A Phys Metall Mater Sci*, vol. **42**, no. 10, pp. 3190–3199 (2011)
- 15. Ramasobane, P., Mashinini, M.P. and Masina, B.N., (2022)
- 16. J. Wang et al., Ceram Int, vol. 48, no. 5, pp. 6985–69979 (2022)
- J. Yu, M. Rombouts, G. Maes, and F. Motmans, in *Physics Procedia*, Elsevier B.V., pp. 416–424 (2012)
- E. Brandl, F. Palm, V. Michailov, B. Viehweger, and C. Leyens, *Mater in engr*, vol. **32**, no. 10, pp. 4665–4675 (2011)
- M. Qian, W. Xu, M. Brandt, and H. P. Tang, *MRS Bull*, vol. 41, no. 10, pp. 775–784 (2016)
- 20. Muiruri, A., Maringa, M. and du Preez, W., Mater, 13(23), p.5355 (2020)

- Li, L., Wang, J., Lin, P. and Liu, H., *Ceramics Int.l*, 43(18), pp.16638-16651 (2017)
- Hao, Y., Liu, J., Li, J., Li, S., Zou, Q. and Chen, X., Mater & Dgn, 65, pp.94-97 (1980-2015)
- Xiang, W., Xuliang, M., Xinlin, L., Lihua, D. and Mingjia, *Mater & Dgn (1980-2015)*, 36, pp.41-46 (2012)
- P. Åkerfeldt, M. L. Antti, and R. Pederson, *Mater Sc and Engr A*, vol. 674, pp. 428–437 (2016)
- 25. P. Bocchetta, L. Y. Chen, J. D. C. Tardelli, A. C. Dos Reis, F. Almeraya-Calderón, and P. Leo, *Coatings*, vol. **11**, no. 5. (2021)
- 26. S. Cao et al., J Alloys Compd, vol. 744, pp. 357–363 (2018)
- Z. Wang, X. Lin, L. Wang, Y. Cao, Y. Zhou, and W. Huang, *Addit Manuf*, vol. 47 (2021)
 - **28.** Q.-D. Sun, J. Sun, K. Guo, S. Waqar, J.-W. Liu, and L.-S. Wang, *Adv Manuf*, vol. **10**, no. 4, pp. 520–540 (2022)

Rebuttal

Comment 1:

Avoid starting a sentence with symbols and abbreviations.

Response 1:

Thank you for the comment. The sentence was rectified, and it now reads as follows:

The Ti6Al4V alloy used in aerospace have shown to contain low surface hardness, density, and wear resistance.

Comment 2:

Correct this as guided in author's template. This should be written as [2, 3]" **Response 2:**

Thank you for the comment. This was corrected as per the template as given below: [2, 3]

Comment 3:

Are pores and residual stresses advantages of LMD? How do cracks and pores result in development of residual stresses in parts?

Response 3:

Thank you for the comment. This was a mistake, and the sentence was rectified as given below:

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.

Comment 4:

Just heat treatment of HIP?

Response 4:

Thank you for the comment. The sentence was rectified as given below: 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).

Comment 5:

Resistance to what? Inaccuracies in?

Response 5:

Thank you for the comment. The sentence was rectified as given below: These residual stresses also cause a microstructure anisotropy of the LMD components, which gives a rise to inhomogeneous microstructure.

Comment 6:

Change this to "transformed". **Response 6:** Thank you for the comment. The "altered" was changed to "transformed". **Comment 7:** Change figure captions to "Fig 1" (See the authors template) **Response 7:** Thank you for the comment. The figure caption was fixed as per template. **Comment 8:** Secondary electron images (SEIs) **Response 8:** Thank you for the comment. This comment is not accepted because the SEI is appearing or displayed on the images in figure 1.



Comment 9:

Write in full on the first instance and subsequently use abbreviations.

Response 9:

Thank you for the comment. The sentence was rectified as follows:

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.

Comment 10:

Avoid using below and above to refer to figures, tables or equations, rather refer the tables, figures and equations by the number such as Table 2, Figure 2, Equation 2 etc.

Response 10:

Thank you for the comment. This was fixed in the entire paper.

Comment 11:

Write the meaning of this symbol as footing in Table 2.

Response 11:

Thank you for the comment. This comment is rejected because the abbreviation of furnace cooling (FC) was given above table 2.

Comment 12:

After etching, were surfaces cleaned and dried?

Response 12:

Thank you for the comment. The sentence was corrected as shown below: 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.

Comment 13:

Rewrite this sentence.

E.g. The microstructure of the produced samples were studied using....." **Response 13:**

Thank you for the comment. The sentence was corrected as shown below: The microstructure of the produced samples was studied using Joel JSM-6010PLUS/LA scanning electron microscope (SEM).

Comment 14:

Where are the Secondary electron images in section on results and analysis? I can only see the optical images.

Response 14:

Thank you for the comment. The sentence was rectified as shown below: The microstructure of the produced samples was studied using Olympus BX51M optical microscope (OM).

Comment 15:

You need to indicate here that images were obtained at the top, middle and bottom of the surfaces analysed.

Response 15:

Thank you for the comment. The sentence was rectified as shown below: 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.

Comment 16:

You mean three indentations?

Were they enough to ascertain the microhardness of the material?

Response 16:

Thank you for the comment. I meant three hardness patterns, each with over 17 indentations.

Yes, the indentations were enough to ascertain the microhardness of the material.

Comment 17:

Was it only one sample?

Response 17:

Thank you for the comment. Yes, only one sample was presented for the as built sample.

Comment 18:

Change this to "Optical images".

Response 18:

Thank you for the comment. The caption was fixed, and it now reads as follows: **Fig 4**. Optical images of as built TiC/Ti6Al4V at top region of the polished surface at (a) low magnification and (b) high magnification

Comment 19:

"Acicular morphology"

Response 19:

Thank you for the comment. This was fixed in the microstructure displayed in figure 3 as shown below:



Comment 20:

One sample or were several analysed?

Response 20:

Thank you for the comment. Only one sample was analysed at 900°C for 2 hours furnace cooling.

Comment 21:

Rewrite this as guided in title of section 3.1.2

Response 21:

Thank you for the comment. This was fixed and title 3.1.3 now reads as follows: 3.1.3 *Microstructure analysis of sample heat treated at 1100°C for 2 h and FC*.

Comment 22:

Rewrite this sentence to make it clear.

Response 22:

Thank you for the comment. The sentence was rewritten as follows:

The sample heat treated at 1100°C showed different microstructural characteristics compared to the as built to 900°C samples.

Comment 23:

Unmelted particles having defects, or they are defects themselves?

Response 23:

Thank you for the comment. The defects were found inside the unmelted particles. **Comment 24:**

Rewrite this sentence to make it clear.

Response 24:

Thank you for the comment. The sentence was rewritten as follows: The microstructure of the 1100°C heat treated sample was therefore found to be inhomogeneous.

Comment 25:

How were these identified from peaks in Figure 11?

Response 25:

Thank you for the comment. The XRD peaks were identified through XRD analysis, and the literature was used to confirm. With the help of literature, a new graph was plotted and is represented in figure 11.





Comment 26:

Did you identify these phases as a result of intensity?

Response 26:

Thank you for the comment. No, the phases were identified with respect to the 2θ values not intensity.

Comment 27:

Consider removing the background from the XRD profile.

Response 27:

Thank you for the comment. This comment is not accepted because the XRD profile has been plotted to its best. Nothing else could have been done to reduce the noise on the profile.

Comment 28:

?? Which titanium? Alpha or beta? How is the cooling rate associated with the formation of either of these phases?

Response 28:

Thank you for the comment. The sentence was corrected to α -Ti, as shown below: Additionally, it is important to note that it is typical to observe high peaks of the alpha titanium (α -Ti), which is because of the slow cooling process associated with the study.

High cooling rates always lead to formation β -Ti phase and slow cooling rates result in formation of α/α' -Ti phases.

Comment 29:

Write this sentence. E.g. a similar observation was reported in Åkerfeldt et al [24], where....

Response 29:

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.

Comment 30:

Transformation of microstructure and reduction in residual stresses(defects) upon heat treatment.

Response 30:

Thank you for the comment. The sentence was restructured as follows: This was because of phase transformation from α to $\alpha + \beta$ as explained by the XRD results in Figure 12. And reduction in residual stresses afforded by the heat treatment.

Comment 31:

This statement is not clear and should be rewritten.

Response 31:

Thank you for the comment. The sentence was restructured as follows:

According to Cao et al [26], heat treatment at 730°C resulted in the transformation of the α phase into $\alpha + \beta$ lamellar microstructure, that promoted an increase in ductility, but the strength of the material was simultaneously decreased.

Comment 32:

This heat treatment will result to increase in beta content as the temperature is above the alpha-beta transformation temperature.

It is anticipated that this microstructure to have the lowest microhardness due grain growth seen in Figure 10.

Response 32:

Thank you for the comment, the increase in hardness was mainly associated with the presence of TiC unmelted particles that were observed in the microstructure. The sentence was rewritten a shown below:

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.

Comment 33:

Which advantage better than the literature? Please rewrite the statement.

Response 33:

Thank you for the comment. the sentence was restructured as follows:

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.

Comment 34:

Rewrite this conclusion.

Response 34:

Thank you for the comment. The conclusion was rewritten as follows: 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 $\alpha + \beta$.
- 900°C, 2 hours, FC results in a major decrease in hardness due to the produced $\alpha + \beta$ 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 increase in hardness because of microstructural transformation in the presence of unmelted TiC particles.

Comment 35:

The references are not in the format prescribed in the authors template provided in the conference website. This should be revised.

Response 35:

Thank you for the comment. The references were corrected according to the template.

Reviews:

Review 1				
2023 RAPDASA-RobMech-PRASA-CoSAAMI Conference Review Form	 (weak accept) Paper follows template, but is not suitable for consideration for SAJIE as it is not within the journal's scope. The paper's idea is okay, but the are a number of errors and omissions in the paper which need to be addressed before it can be considered suitable for publication. 			
Does the title reflect the contents of the paper? (Yes/no)	Yes			
Does the paper relate to what has already been written in the field? (Yes/no)	Yes it does. There is a lot of published research on the addition of TiC to Ti6Al4V using laser metal deposition, as well as other techniques. It is therefore uncertain how the current results are showing something new. This aspect is critical and needs to be addressed.			
Do you deem the paper to be proof of thorough research and knowledge of the most recent literature in the field of study? (Yes/no)	No. The authors need to review papers based on LMD of TiC added to Ti6Al4V - numerous papers available which can be cited to provided a proper background for the research undertaken. Mainly used SLM research which is not really applicable. Have not shown evidence of the scientific gap being addressed.			
Is the paper clearly structured, easy to read and with a logical flow of thought? (Yes/no)	Yes, some minor grammatical errors.			
Are the arguments employed valid and supported by the evidence presented? (Yes/no)	Yes to some extent, but as mentioned in earlier comments, the authors have not clearly demonstrated that their results are new or adding value to what is already known about the addition of TiC to Ti6Al4V using LMD and heat treatment processes to improve the alloy hardness.			
	Some of the microstructure images have 3 micron markers! Please insert a single correct micron marker in all images. Some images are too dark and some text is too small. Please revise accordingly. Some figures and tables are referred to incorrectly. The hardness unit is not correct in most instances.			
	Some of the explanations of the results do not match what is shown in the graphs for hardness. Please revise for accuracy. Be careful of using words such as 'significant' differences when this is not the case based on the results. Explain the 4 data points which do not fit into the trends.			
	The legends for some XRD do not match the peaks in the graphs? Some explanations of the XRD don't seem to match what is shown in the XRD and the microstructure?			
Are the conclusions clear and valid? (Yes/no)	Yes to some extent the authors have concluded what they results have shown. The scientific gap addressed has not been concluded and this should be addressed to state what is new.			
Are the conclusions clear and valid? (Yes/no)	Yes to some extent the authors have concluded what they results have shown. The scientific gap addressed has not been concluded and this should be addressed to state what is new.			
Does the paper conform to accepted standards of language and style? (Yes/no)	Yes to some extent. Please check and revise grammatical errors. Some misalignment between figures mentioned in the text and the actual figures being referred to, e.g. text states Fig. 15 refers to XRD results, but there is no Fig. 15. Please check for these errors in entire paper.			
Is the paper suitable for publication in SAJIE? (Yes/no)	No it does not fit the scope of the journal			
Does the paper make a contribution to the field of industrial engineering in terms of theory, methodology or practice? (Yes/no)	No			
Does the paper conform to accepted standards of language and style to be published as a Journal article? (Yes/no)	N/A			
Review 2				

Review 2			
2023 RAPDASA-RobMech-PRASA-CoSAAMI Conference Review Form	 (weak accept) The paper requires serious revision before being accepted for presentation in the conference. 		
Does the title reflect the contents of the paper? (Yes/no)	Yes. However, the term "hardness" on the title should be changed to microhardness.		
Does the paper relate to what has already been written in the field? (Yes/no)	Yes.		
Do you deem the paper to be proof of thorough research and knowledge of the most recent literature in the field of study? (Yes/no)	Yes.		
Is the paper clearly structured, easy to read and with a logical flow of thought? (Yes/no)	No. Several statements in this work are poorly constructed and lack logic. See more of the comments on the paper.		
Are the arguments employed valid and supported by the evidence presented? (Yes/no)	No. Some arguments employed in sections 3.2 and 3.3 should be revised.		
Are the conclusions clear and valid? (Yes/no)	No. Some conclusions should be rewritten to make them clearer and concise.		
Does the paper conform to accepted standards of language and style? (Yes/no)	No. The references should conform to the style prescribed in the authors' template.		
Is the paper suitable for publication in SAJIE? (Yes/no)	No.		