



Heat treatment response and characterization of Ti6Al4V + xMo produced by laser metal deposition

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ABSTRACT

During the laser metal deposition additive manufacturing processing of Ti6Al4V ELI alloy, the parts produced were exposed to high levels of thermal gradients, which resulted from rapid heating and cooling rates in the material. This had an adverse effect on the material properties, as tensile residual stresses were created in the parts and increased the strength while significantly reducing ductility. Additionally, the presence of columnar grains compromised the material properties because it resulted in inhomogeneous microstructures that exhibit anisotropy in parts. This study investigated the influence of β annealing temperatures on the microstructure of Ti6Al4V ELI alloy produced during laser metal deposition, and the Ti6Al4V ELI in-situ alloyed with varying molybdenum content. The observations made included a temperature driven phase transformation, which resulted in a change from columnar to equiaxed grains due to heat treatment of the Ti6Al4V ELI alloy, while the solidification structure of the alloy changed from planar to cellular due to the addition of Mo. The Ti6Al4V ELI alloy heat treated at 1000 °C reported a hardness profile of $204 \pm 5 \text{ HV}_{0.3}$, which was comparable to the reported hardness ($206 \pm 34 \text{ HV}_{0.3}$) of the Ti6Al4V ELI in-situ alloyed with 10 mass percent Mo (10% Mo). This implies that the effects of the in-situ alloying of Ti6Al4V ELI with 10% Mo are comparable to the heat treatment of Ti6Al4V ELI alloy at a β annealing temperature of 1000 °C, in terms of stabilization of the β -phase.

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1. Introduction

The Laser-based additive manufacturing (AM) of Ti6Al4V ELI alloy results in thermal gradients, which results from the rapid heating and cooling rates in the material. This negatively affects the properties of the material because of the tensile residual stresses that are created. These tensile residual stresses ultimately cause an increase in strength and reduces ductility [1–3]. Additionally, the presence of columnar grains that run along the build direction compromises the material properties because it results in inhomogeneous microstructures that exhibit anisotropic behavior in parts [4]. Several studies report an improvement in microstructural defects through post-processing heat treatment to reduce residual stress build-up and alleviate the negative effect

associated with it, by modifying the microstructure to the more favorable equiaxed grain morphology that promotes high strength and improved ductility [1,5].

The properties of titanium (Ti) alloys are typically improved in two ways, i.e., through alloying and processing [5]. In alloying, specific elements are added to the material in varying amounts in order to either (a) improve its properties or (b) make it easier to process. Examples in property improvements include an increase in build density and reduced crack susceptibility of crack sensitive materials through alloy modifications [6–7], whereas the addition of specific elements to alloys such as aluminium, making the material susceptible to heat treatment and natural ageing, is an example of improving the processability of an alloy [8]. Moreover, processing, on the other hand, establishes a balance of mechanical properties through microstructural control, which is achieved by means of thermal and thermomechanical treatments [9–10].

Materials produced by laser metal deposition (LMD) technique exhibit different starting microstructures in comparison to other

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manufacturing techniques. Therefore, it is possible that the mechanical properties of LMD processed parts would differ from the properties of parts produced through conventional manufacturing practices. According to Yan et al. [5], optimizing the AM process parameters and post heat-treatment conditions was essential in controlling the grain structure. There were parallels drawn between the processing strategies, grain structure and material properties to shed light on common methods for powder bed AM of metallic materials, and the specific grain morphologies recognized to promote a good balance between mechanical properties and ductility. Typically, the columnar prior β grains are observed during layer-wise fabrication of metals due to epitaxial growth [5,11], however, the grain size can be controlled by limiting the columnar grain growth by increasing the nucleation sites (among other methods) during processing, which would result in a grain refining effect to promote the formation of equiaxed grains.

In Ti alloys, the mechanical properties directly correlate to specific features such as presence of α colonies and β grain sizes. Thus, the general goal is in achieving the best combination of phases which would lead to the formation of the most favorable microstructures for improved mechanical properties in the alloy. Equiaxed microstructures are regarded as having exceptional ductility and fatigue crack nucleation resistance. Lamellar microstructures report elevated fracture toughness, creep strength and fatigue crack growth resistance, while the bi-modal or duplex microstructures combine the benefits of both the lamellar and equiaxed microstructures [9].

This investigation dovetails from previous work that considered the LMD processing of Ti6Al4V ELI alloy treated with beta-stabilizing elements (such as molybdenum (Mo)). Fig. 1 shows the phase diagram for Ti alloys and illustrates how alloying affects the stability of phases in titanium. The elements are classified according to which phase they stabilize. Alpha (α) stabilizers increase the transus temperature to allow for this phase to be stable at higher temperatures, while beta (β) stabilizers lower the transus temperature, thus allowing this phase to be stable at lower ranges than typically found. The beta stabilizers can further be subdivided into β -isomorphous (Mo, V, Nb, Ta) and β -Eutectoid (Fe, Cr, Ni, Cu, W, Co) based on the extent of its solubility in β -Ti [12].

The present study seeks to investigate the influence of different β annealing temperatures on the evolved microstructure of Ti6Al4V ELI alloy before and after addition of different quantities of Mo, performed through in-situ alloying for property improvements. LMD exhibits different starting microstructures compared to other manufacturing techniques. In addition to the build-up of thermal stresses inherent to the manufacturing process, this contributes to reduced performance in AM produced components. The microscopy, hardness and tensile measurements were utilized to characterize the resulting alloys. Observations made are highlighted and discussed further.

2. Materials and methods

This study is a progression from previous work that considered the LMD processing of Ti6Al4V ELI alloy treated with beta-stabilizing elements, and thus the materials characterized in this study were fabricated and prepared according to the experimental setup described by Arthur et al. [3]. For the purpose of the study Grade 23 Ti6Al4V ELI alloy will be referred to as Ti-64. Ti-64 sample coupons of dimensions $12 \times 12 \times 5$ mm were laser printed on the Laser Engineered Net-Shaping (LENS) 3D printing system at an energy density (ED) of 287 J/mm^3 , with different quantities of Mo additions to produce three types of samples for analysis, that is, one sample that consisted of only Ti-64 as a control sample, a second sample that consisted of Ti-64 with a 10 mass% (10% Mo) Mo content, and a third sample that consisted of Ti-64 with a 20 mass% (20% Mo) Mo content as indicated in Table 1. In-situ alloying was performed by depositing the powders simultaneously into the melt pool.

The heat treatment was carried out in a Carbolite STF 16/180 high temperature tube furnace at β annealing temperatures of 900°C and 1000°C and in an argon (Ar) rich environment to ensure that heat treatment occurs above the β -transus temperature and in the β -phase field to promote an increase in the relative phase amount of the β -phase. The sample coupons were furnace cooled, following a two-hour dwell time.

The samples were metallographically prepared for microstructural analyses. The specimens were grinded with SiC grinding papers and water as a lubricant. Using a Md-Chem cloth soaked in a $0.04 \mu\text{m}$ OP-S suspension fluid after grinding, the desired mirror finish surface was produced. Etching of the polished surface with Kroll's reagent for 30 s adequately revealed the microstructure. An Olympus BX51M microscope together with Stream Essentials software and a JEOL JSM-6510 scanning electron microscope (SEM) with an energy dispersive spectroscope (EDS) was used in microstructural observations and elemental analyses.

The mechanical properties were determined by hardness measurements using a Matsuzawa Vickers micro-hardness tester. A load of 300 g was applied for Vickers at a dwelling time of 10 s, and with a spacing of $250 \mu\text{m}$ between the indentations. An Instron 5988 tensile tester was used to test the Ti6Al4V ELI alloy both prior to and after heat treatment. A total of three tensile tests were conducted per sample to ensure reliability and repeatability of the results.

3. Results and discussions

3.1. Influence of temperature on microstructure

Fig. 2 presents SEM and optical micrographs of the typical microstructure observed in Ti-64 alloys produced by LMD [3,14].

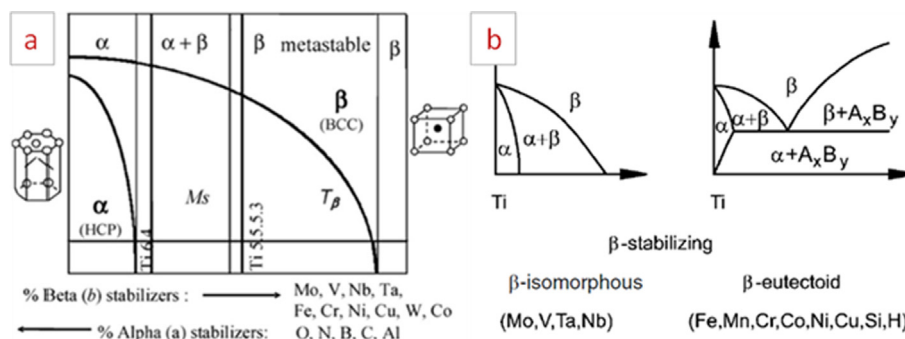


Fig. 1. Titanium alloy phase diagrams showing (a) phases of Ti64 and Ti555.3 with alloying elements [12]; (b) phases present due to β stabilizers [13].

Table 1
LENS fabricated titanium alloy sample coupons.

Alloy	Alloying Element Composition
Ti-64	0% Mo
Ti-64	10% Mo
Ti-64	20% Mo

Fig. 2(a, d) illustrates the initial microstructure before post-processing heat treatment, while Fig. 2(b, e) and Fig. 2(c, f) represent microstructures after the application of heat treatment in the β -phase field at 900 °C and 1000 °C respectively. The columnar prior β grains observed in the initial microstructure (Fig. 2a) change as the temperature increases into the β -phase field, resulting in a temperature driven phase transformation [9], with the prior β grains presenting an equiaxed morphology [5,10,15], as seen in Fig. 2(b, c). This is consistent with observations recognized by Yan et al. [5]. Thus, implying that heat treatment temperatures of 900 °C and above are sufficient to stabilize the β phase. Similarly, the prior β grain boundaries observed in the as built alloy (represented by Fig. 2d) are more pronounced after the specimen was heat treated at a temperature of 900 °C (Fig. 2e) and 1000 °C (Fig. 2f) when observed at the same magnification.

Fig. 3 shows the results from grain size analysis done on the Ti-64 alloy expressed as length and breadth. Fig. 3a represents the alloy in the as built state with most of the grains below 60 μm long and 35 μm wide. Fig. 3b shows that most of the grains were shorter than 40 μm in length and thinner than 20 μm in width after heat treatment of the alloy at 1000 °C. This suggests a grain refining effect upon heat treatment of the alloy within the β -phase field.

Furthermore, the presence of tiny white phase observed along the β grain boundary suggests that the Ti-64 alloy underwent a β -to- α transformation during the cooling process from the β -phase field [16]. The α phase is reported to nucleate and grow along the β grain boundaries and is referred to as grain boundary α (αGB) layers [9,16]. This is represented by Fig. 4, with image (b) indicating a close-up view of the region of interest highlighted by a red block in image (a).

The formation of αGB layers is undesirable as it has a detrimental effect on mechanical properties such as tensile and fatigue properties [9,17]. The result is a reduction in ductility, which is counter intuitive to the purpose of β annealing of the Ti-64 alloy. The presence of αGB leads to a build-up of strain that gets concentrated in particular locations. This leaves the material susceptible to the formation of cracks in the location of the strain build-up, in addition to inter-granular fracture through the β grain boundaries due to the presence of αGB layers [17]. All of which would lead the material to premature failure during testing, or to report a considerably low level of ductility.

3.2. Influence of alloy Composition on microstructure

Arthur et al. [3], reported a stabilizing effect of the β phase that was associated with the addition of 10% and 20% Mo to Ti-64 when using in-situ alloying strategies. Additionally, a change in solidification structure from planar to cellular was observed, while a softening of the alloy was detected through hardness measurements. That is to say, the reported hardness of Ti-64 ($380 \pm 13 \text{ HV}_{0.3}$) decreased to $206 \pm 34 \text{ HV}_{0.3}$ upon in-situ alloying with 10% Mo, and a decrease to $324 \pm 13 \text{ HV}_{0.3}$ upon in-situ alloying with 20% Mo. The softening effect observed is attributed to an increase in the β -phase, or rather, an increase in the amount of β -Ti present within the alloy, which is reported to promote a softening effect within the material and is associated with an improved ductility [18]. This is because the crystal structure of β -Ti is softer than that of α -Ti due to an increased number of slip planes at different orientations [18]. Therefore, an increase in the amount of β -Ti as compared to α -Ti will present a change in strength of the material since there is a lower amount of the phase responsible for producing high strength. Moreover, XRD analysis revealed an additional hexagonal phase with lattice parameters that correspond to that of omega (ω) phase (4.60 Å (a) and 2.82 Å (c)).

Fig. 5 shows the results of this study from examining the influence of the annealing temperature and Mo content on the evolution of the microstructure. This is illustrated by the microstructures presented in Figs. 2 and 5. The initial microstruc-

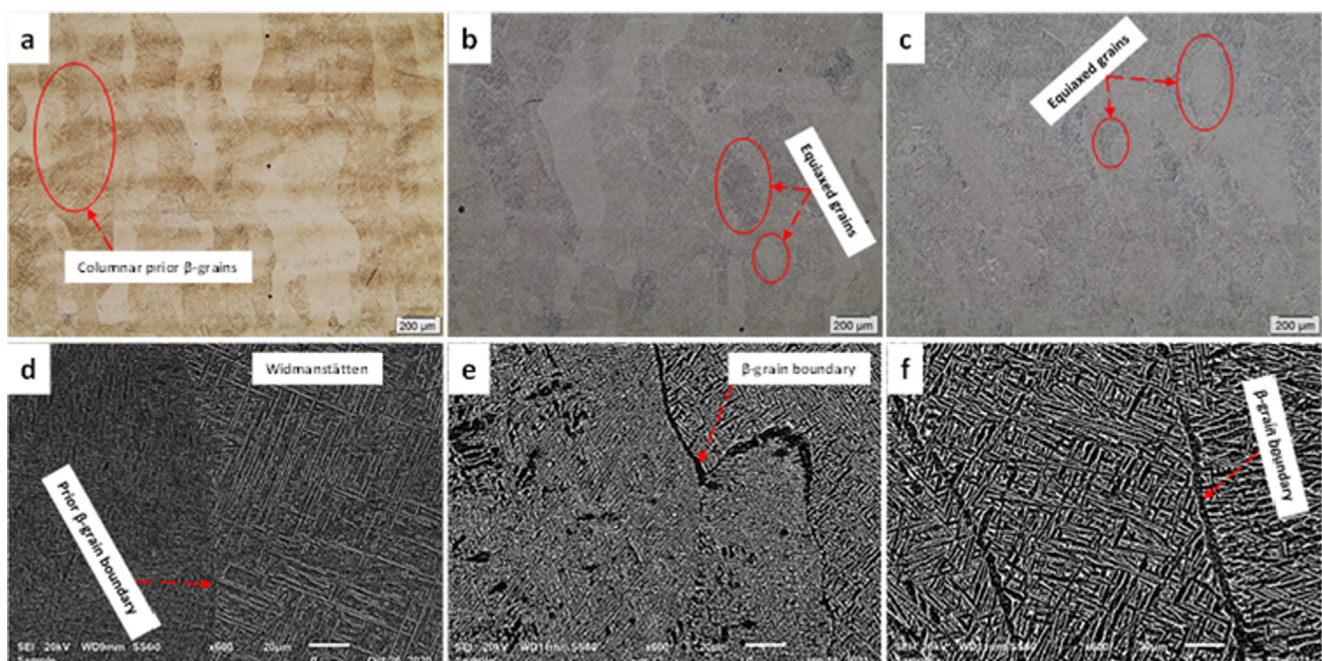


Fig. 2. Optical and SEM Micrographs (top–bottom) of LENS fabricated Ti-64 samples (a, d) as built; (b, e) heat treated at 900 °C; and (c, f) heat treated at 1000°C.

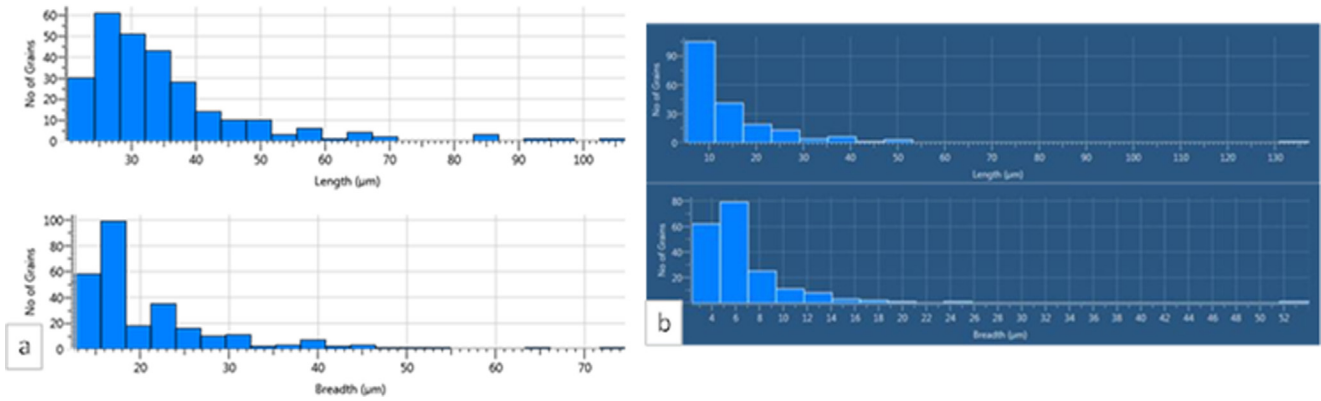


Fig. 3. EBSD grain size histograms of LENS fabricated Ti-64 samples (a) as built; (b) heat treated at 1000 °C.

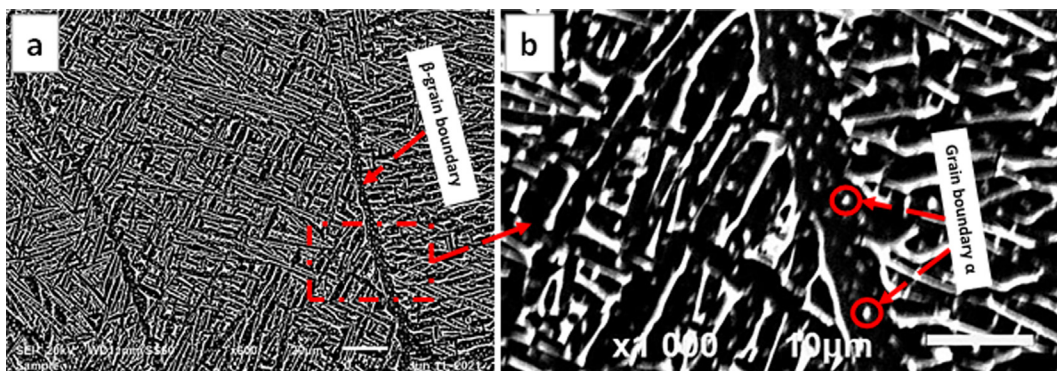


Fig. 4. β -Grain boundary showing nucleation of α layers of Ti-64 heat treated at 1000 °C.

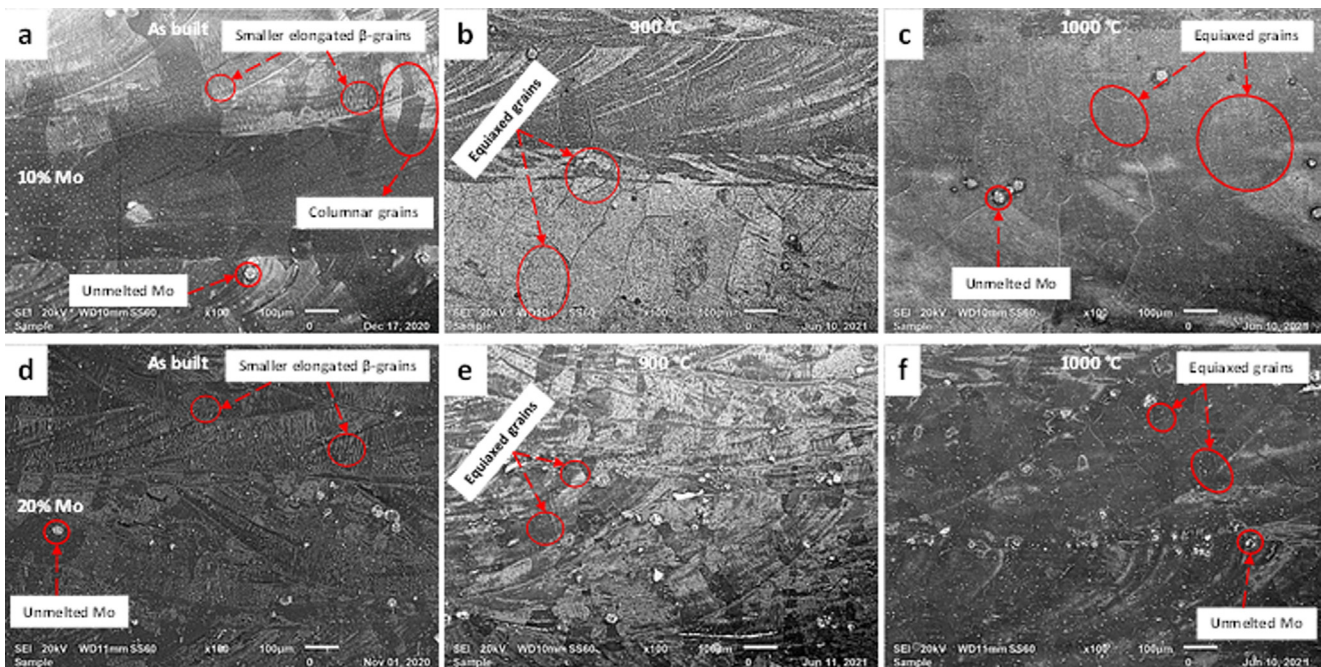


Fig. 5. SEM images of Ti-64 with increasing amounts of Mo additions (top to bottom) at increasing heat treatment temperatures (left to right).

ture before post-processing heat treatment (Fig. 5(a, d)) presents a reduction in grain size experienced due to the addition of refractory metals such as Mo [3]. The difference in microstructures due to the presence of Mo is evident through comparison of Fig. 2a with

Fig. 5(a, d). This observation of a reduction in grain size is consistent with literature that states that a change in morphology accompanies a change in solidification structure [5]. Thinner and smaller β grains begin to form within the melt pool, with the

20% Mo alloy seemingly promoting finer β grains than that of the 10% Mo alloy. Furthermore, the presence of columnar prior β grains is still observed within the microstructure of the 10% Mo alloy, whereas the 20% Mo alloy shows a greater shift from columnar to cellular solidification.

The introduction of heat treatment in the β phase field for the Ti-64 alloy brought about a phase transformation that led to a change in morphology. This presented as equiaxed grains that showed slight grain refinement. A similar effect was observed in the case of the alloys containing Mo additions (illustrated by Fig. 5), whereby post-processing treatments at 900 °C and 1000 °C resulted in a change of microstructure to reveal non-uniform equiaxed grains. However, the 20% Mo alloys reported an average grain size of approximately 100 μm . In addition to this, the microstructure of the alloys shows the presence of finely dispersed white particles, which are equally distributed within the alloys. Yan et al. [5] observed that the occurrence of partially melted Mo particles can serve as heterogeneous nucleation sites, suggesting that the finely dispersed white particles may be due to the nucleation of α at these nucleation sites.

3.3. Mechanical properties

Fig. 6 shows the effect of heat treatment temperature on the micro-hardness of the as built Ti-64 alloy and alloys with Mo additions. The Ti-64 alloy reports a sharp decrease in average hardness from $380 \pm 13 \text{ HV}_{0.3}$ to $213 \pm 4 \text{ HV}_{0.3}$ upon treatment at 900 °C. A further increase in temperature (to 1000 °C) showed a slight decrease in hardness to $204 \pm 5 \text{ HV}_{0.3}$, which is comparable to the micro-hardness of the as built 10% Mo alloy. The expectation was for a softening of the material based on the observed microstructures reported in Fig. 2. Lewandowski et al. [1] reported a similar observation in which the Ti-64 hardness decreases with an increase in temperature. Typically, the resulting equiaxed microstructure leads to optimized strength and ductility [5,9]. However, the 10% Mo alloy reported a sharp increase in micro-hardness from $206 \pm 34 \text{ HV}_{0.3}$ to $389 \pm 34 \text{ HV}_{0.3}$ upon heat treatment of the as built alloy at 900 °C and with a further increase in temperature to 1000 °C, which showed a continued increase in

hardness to $420 \pm 38 \text{ HV}_{0.3}$. Similarly, the 20% Mo alloy reported a sharp increase in hardness when the as built alloy was heat treated at 900 °C ($324 \pm 13 \text{ HV}_{0.3}$ to $341 \pm 8 \text{ HV}_{0.3}$). However, no substantial change in micro-hardness was observed ($343 \pm 10 \text{ HV}_{0.3}$) once the temperature was increased to 1000 °C.

The observed increase in micro-hardness profile of the alloys with Mo additions can be attributed to the presence of ω phase that develops when Ti alloys are alloyed with β stabilizing transition metals like Mo [19,20]. This ω evolves as a homogeneous distribution of fine particles spread out along the β grains in metastable β alloys, whereby the onset of ageing conditions in these alloys is likely to promote the formation of very fine and uniformly distributed secondary α (α'') [9]. The newly formed α'' phase had a strengthening effect on the alloy, as a refined microstructure was evolved through its occurrence. It is therefore worthwhile to adopt a heat treatment process that avoids the formation of intermetallic phases and/or very fine particles that lead to increased hardness. In addition, a heat treatment process able to promote the evolution of a homogeneous microstructure that exhibits either an equiaxed, or a bi-modal (combination of equiaxed and lamella) structure is likely to be beneficial. As reported by Barriobelo [9], this microstructure results in optimized strength and ductility.

Fig. 7 shows the tensile property response of the Ti-64 alloy in the as built state and that of the two β annealed alloys. A substantial decrease (greater than 10%) in yield strength (YS) and ultimate tensile strength (UTS) was observed, which agrees with findings by Lewandowski et al. [1] that indicates that a decrease in YS and UTS is observed based on an increase in temperature. According to ASTM standards for the additive manufacturing of Ti-64 with powder bed fusion (PBF) [21], an acceptable minimum limit for YS and UTS is 825 MPa and 895 MPa respectively, which needed to be satisfied by the alloy in the different treatment conditions. Unfortunately, the acceptable minimum tensile elongation of 10% by ASTM standards was not satisfied. However, although the elongation criterion was not met, the reduction in YS and UTS shows the effectiveness of the post-processing heat treatment in mitigating the high internal stresses inherent to the LENS LMD process.

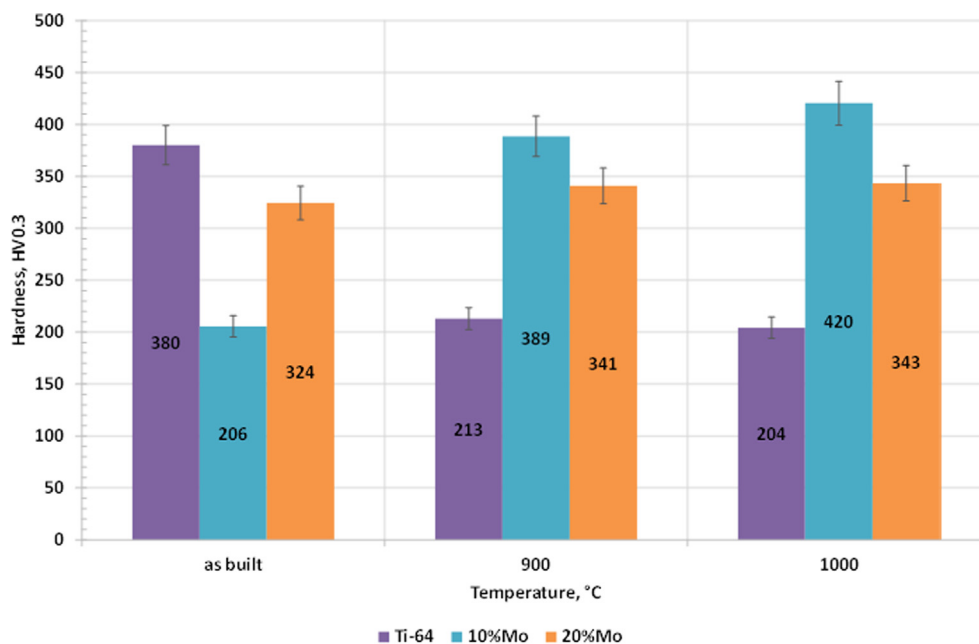


Fig. 6. Influence of heat treatment temperature and Mo additions on micro-hardness of the Ti-64 alloy.

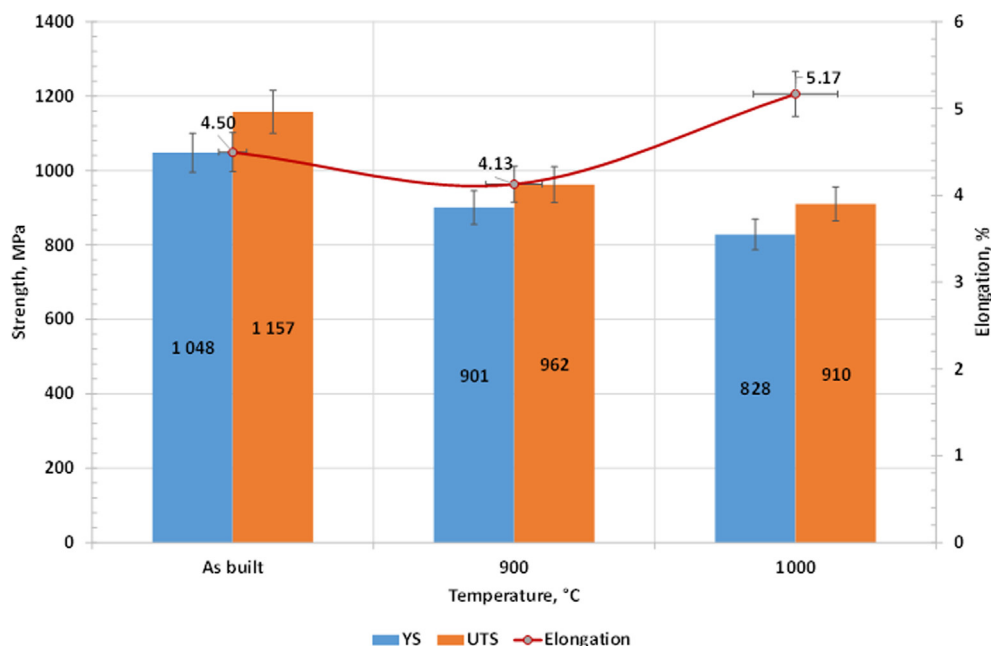


Fig. 7. Influence of heat treatment temperature on tensile properties of LENS produced Ti-64 alloy.

The results presented suggest that the combination of heat treatment processes after alloying techniques results in the formation of phases with detrimental effects to mechanical properties such as tensile strength and ductility. The finished product would most likely require a two-stage heat treatment procedure in order to avoid the formation of intermetallic phases and achieve an equiaxed or bi-modal microstructure. The two-stage process will improve microstructural homogeneity and its associated mechanical properties, which will prevent premature failure of AM produced parts (with particular reference to LMD techniques) during service.

Conclusions.

Specifically, this study investigated post-processing heat treatment and alloying as means for improving properties of the Ti-64 alloy produced by LMD technique. The reported findings led to the following conclusions:

- Post-processing heat treatment was effective in mitigating the high internal stresses inherent to the LENS LMD process.
- β annealing heat treatment temperatures of 900 to 1000 °C were sufficient to stabilize the β -phase of LMD produced Ti-64 alloy and achieved a softening effect in the alloy.
- Alloying Ti-64 with β stabilizing elements like Mo offers a viable solution to property improvements, as Mo additions of 10 mass% yields similar effects as β annealing at 1000 °C.

CRedit authorship contribution statement

Nana K.K. Arthur: Conceptualization, Investigation, Writing – original draft, Visualization. **Charles W. Siyasiya:** Conceptualization, Writing – review & editing, Supervision. **Sisa L. Pityana:** Conceptualization, Writing – review & editing, Supervision. **Monnamme Tlotleng:** Conceptualization, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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