Eliminating continuous grain boundary α phase in laser melting deposited near β titanium alloys by heat treatment

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Abstract: Continuous grain boundary α phase (αGB) is widely present in laser melting deposited near β and β titanium alloys, and it can lead to intergranular fracture and low ductility. Because of the preferential nucleation and growth of α phase at β grain boundaries, the continuous αGB cannot be effectively eliminated by traditional heat treatment. Thus, in this study, we develop a new heat treatment including beta solution + ultra-slow furnace heating up + traditional heat treatment, to eliminate continuous αGB in laser melting deposited Ti-5Al-5Mo-5V-1Cr-1Fe near β titanium alloy, and the microstructures and tensile properties are investigated. The results indicated that, after beta solution + normal heating up + traditional heat treatment, the continuous αGB still exists, and the elongation is still low about 7.5%. However, after beta solution + ultra-slow heating up + traditional heat treatment, there are almost no continuous αGB, and the elongation increases to 15.2%. The mechanism about the αGB formation during ultra-slow heating up process is simply revealed. Furthermore, this new heat treatment is also suitable to eliminate continuous αGB for laser melting deposited Ti17 alloy.

1. Introduction
Laser melting deposition (LMD) is a powder feeding additive manufacturing technology, which can be used to fabricate large-scale titanium aerospace components with low buy-to-fly ratio and short time. For the purpose of application, many studies have been carried out to investigate the microstructures and mechanical properties of laser melting deposited titanium alloys [1-5]. For α and α+β titanium alloy (such as Ti-6Al-4V etc.), the mechanical properties can be similar with or even higher than those of wrought materials [6, 7], thus laser melting deposited Ti-6Al-4V alloy large-scale components has been applied in aerospace industry [5]. However, for near β and β titanium alloys, such as Ti-5Al-5Mo-5V-1Cr-1Fe [8] and Ti-6Al-2Sn-4Zr-6Mo [3], their low ductility largely restricts their applications. It has been found that the continuous grain boundary α (αGB) in laser melting deposited near β and β titanium alloys can lead to the intergranular fracture, which is an important reason for the low ductility [1, 3, 8].

For wrought titanium alloys, the continuous αGB can be broken by the deformation in α+β phase field. However, for laser melting deposited titanium alloys, which are near-net-shape components, the
wrought based processing obviously cannot be used due to its accompanied obvious shape changes. Thus, post heat treatments turn to be the most promising way to reduce continuous α GB. However, the formation of continuous α GB is due to the preferential nucleation and growth of α phase at β grain boundaries, it is very stable. Although various efforts have been made and the continuous α GB can be reduced or changed [1, 9, 10], uniform microstructure without continuous α GB still cannot be obtained.

Hence, in the present study, we develop a new pre-heat treatment including beta solution which will entirely annihilate the as-deposited microstructure and results in single β phase, and ultra-slow furnace heating up processes which will lead to the re-formation of α phase, to eliminate continuous α GB in laser melting deposited Ti-5Al-5Mo-5V-1Cr-1Fe (VT22 in Russia) high strength near β titanium alloy, the microstructures and tensile properties of the heat treated samples are investigated. Furthermore, to demonstrate its generality for near β titanium alloys, the pre-heat treatment is applied for laser melting deposited Ti-5Al-2Sn-2Zr-4Mo-4Cr alloy (Ti17 in US) to verify the effectiveness.

2. Experimental Procedures
Plate-like samples of VT22 and Ti17 with dimensions approximately 400 mm × 250 mm × 40 mm was fabricated by laser melting deposition in Beihang University. The detailed laser melting deposition fabrication process had been presented in authors’ previous papers [1, 11]. The β-transus temperatures (Tβ) of as-deposited TC18 and Ti17 alloy were 880 ± 5 °C and 895 ± 5 °C, determined by metallographic method.

For the sake of discussion, three types of heat treatments are applied. The first one is traditional heat treatment (THT) for VT22 alloy, which contains three steps as shown in Fig. 1a [1, 12]: (i) heating to 830°C, holding for 2h, and furnace cooling (FC) to 750°C (T2); (ii) holding at 750°C for 2h and air cooling (AC); (iii) aging at 600°C for 4h and air cooling. The furnace cooling rate was 3 °C/min. The second one is pre-heat treatment + THT. The pre-heat treatment is a simple solution treatment in β phase field at 900°C for 0.5 h followed by water quench (WQ) as shown in Fig. 1b. The third one is also β solution pre-heat treatment + THT, the only difference is the heating rate during initial furnace heating (FH) up in THT as shown in Fig. 1c. For the second heat treatment, it is the normal heating rate about 10 °C/min, and for the third one, it is the ultra-slow heating rate about 0.56 °C/min.

Metallographic specimens of the heat treated samples were prepared by conventional mechanical polishing method. A mixture of 1 ml HF, 6 ml HNO3 and 100 ml H2O was used as the etching agent. The microstructures of samples were characterized by optical microscopy (OM). The tensile tests were performed according to the test standard of ISO 6892-1: 2009. Round specimens with 5 mm diameter, 35 mm gauge length and 71 mm total length were prepared for tensile tests at room temperature. Here, the axial direction of tensile specimens was parallel to the deposition direction. The mechanical properties were characterized by averaging the measured values over three tensile specimens. Besides, the fracture surfaces of the tensile test specimens were examined by SEM.
3. Results and Discussion

After THT, laser melting deposited VT22 alloy consists of continuous $\alpha_{GB}$ and lamellar $\alpha$ phase inside $\beta$ grains as shown in Fig 2a and b, which has been reported in our previous studies [1]. Because the continuous $\alpha_{GB}$, the cracks preferentially initiate and propagate along the continuous $\alpha_{GB}$ (see Fig. 2b) during the tensile stress loading, resulting in intergranular fracture (see Fig. 2b and c), and hence leading to low ductility as listed in Table 1.

The continuous $\alpha_{GB}$ in laser melting deposited VT22 alloy is very stable, and is difficult to be eliminated during the heat treatments in $\alpha+\beta$ phase field, including THT. Then, the beta solution 900°C/0.5 h, WQ is applied, which will entirely annihilate the as-deposited microstructure. It should be noted, for near $\beta$ titanium alloys, the water quench will not result in martensite [13], and hence the beta solution leads to the single $\beta$ supersaturated solid solution. Meanwhile, due to the relative low annealing temperature (900°C) and short annealing time (0.5h), the $\beta$ grain size will not change [10]. Then, during the subsequent THT process, the $\alpha$ phase includes $\alpha_{GB}$ reforms. The final microstructures of beta solution + THT with different heating rate during the initial heating up process are shown in Fig. 2.

After beta solution + THT with normal initial heating up, the microstructure of VT22 alloy is similar with the microstructure of the sample after THT, and still consists of continuous $\alpha_{GB}$ as shown in Fig 2d. The cross section image of tensile specimen shows that the cracks also preferentially propagate along the continuous $\alpha_{GB}$ (see Fig. 2e), which indicates the intergranular fracture. Hence, the heat treatment still leads to low ductility of VT22 alloy as listed in Table 1. The small and low dimples are in agreement with the low ductility. The above results suggest that the beta solution + THT with normal initial heating up has not effect on improving the ductility of laser melting deposited VT22 alloy.
Interesting results are found in the samples after beta solution + traditional heat treatment with ultra-slow initial heating up process. It can be seen that there are almost no continuous α GB as shown in Fig. 2g, and due to the microstructure change, the tensile fracture mode changes to be transgranular as expected as shown in Fig. 2h. The fracture surface also exhibits large and deep dimples as shown in Fig. 2i. All the above results suggest the samples have good ductility. Then, the strength and ductility are given in Table 1. We can find that the strength including high ultimate tensile strength (UTS) and yield strength (YS) have a slightly decrease, which may be caused by the coarsen of laminate α phase, as can be seen from Fig. 2b and h. The long time heating up process promote the coarsen of α phase. Meanwhile, the elongation and reduction of area can be up to 15.2% and 37.3%, respectively, are about 2 times that of the samples after THT and beta solution + THT with normal initial heating up. Obviously, the great improvement of ductility is mainly due to the elimination of continuous α GB.
The above results indicate that heating rate during initial heating up process after beta solution has significant effect on the re-formation of $\alpha_{GB}$. Then, the mechanism is preliminary proposed in Fig. 3. After beta solution, the sample consists of single $\beta$ phase as schematically shown in Fig. 3a. Then, during the heating up process, $\alpha$ phase will nucleate and grow up.

As we know, $\alpha$ phase nucleates and grows preferentially at $\beta$ grain boundaries during $\beta \rightarrow \alpha$ phase transformation, because of the low activation energy required for $\alpha$ phase nucleation and the high elements diffusion rate at the $\beta$ grain boundary. Hence, to eliminate $\alpha_{GB}$, we should obtain the uniform nucleation and growth of $\alpha$ phase as much as possible.

During the normal heating up process, the temperature rapidly rise to relative high temperature, so the nucleation of $\alpha$ phase mainly occurs at high temperature (see Fig. 3b), and the driving force for the nucleation of $\alpha$ phase is relative small at high temperature, hence the preferential nucleation of $\alpha$ phase at $\beta$ grain boundaries is more likely to occur as schematically shown in Fig. 3b. Then, $\alpha_{GB}$ preferentially grow to be continuous layer morphology as shown in Fig. 3c.

During the ultra-slow heating up process, the sample is heated at low temperature for a long time, so $\alpha$ phase can nucleate completely at low temperature (see Fig. 3e). Meanwhile, the driving force for the nucleation of $\alpha$ phase is very high at low temperature, which makes the influence of $\beta$ grain boundaries on the preferential nucleation of $\alpha$ phase being largely reduced, hence the nucleation of $\alpha$ phase will be much more uniform as shown in Fig. 3e. Then, the temperature is rising ultra-slowly, $\alpha$ phase will grow up slowly and more uniformly (see Fig. 3f and g), which can inhibit the preferential growth of $\alpha_{GB}$. At last, the $\alpha$ phase formed during ultra-slow heating up process will distribute much more uniform than that formed during normal heating up process as shown in Fig. 3h.

![Figure 3](image-url) Schematic illustration showing the nucleation and growth of the $\alpha$ phase during the normal heating up process (a, b, c, d) and ultra-slow heating up process (a, c, e, g, h) after beta solution.

The above discovery is significance for improving the ductility of additive manufacturing high strength near $\beta$ and $\beta$ titanium alloys, which are are deeply troubled by continuous grain boundary $\alpha$. Then, to demonstrate its generality for near $\beta$ titanium alloys, the pre-heat treatment is applied for laser melting deposited Ti17 alloy to verify the effectiveness, as shown in Fig. 4. After the heat treatment with normal heating up process, the microstructure still consists of continuous $\alpha_{GB}$ as shown in Fig 4a and b. After the heat treatment with ultra-slow heating up process, there are almost no continuous $\alpha_{GB}$ as shown in Fig 4c and d. Hence, we can find that the pre-heat treatment including beta solution and ultra-slow furnace heating up processes can effectively eliminate continuous $\alpha_{GB}$ for additive manufacturing near $\beta$ and $\beta$ titanium alloys.
4. Conclusions
In this study, to eliminate continuous $\alpha_{GB}$ in laser melting deposited near $\beta$ titanium alloy, we develop a new pre-heat treatment including beta solution and ultra-slow furnace heating up processes. The microstructures and tensile properties of the heat treated samples are investigated. The main finding can be succinctly summarized as follows:

1. After traditional heat treatment, there are continuous $\alpha_{GB}$ in laser melting deposited Ti-5Al-5Mo-5V-1Cr-1Fe alloy, which will lead to intergranular fracture and low ductility.
2. After beta solution + normal heating up + traditional heat treatment, the continuous $\alpha_{GB}$ still exists, and the ductility is still low. After beta solution + ultra-slow heating up + traditional heat treatment, there are almost no continuous $\alpha_{GB}$, and the ductility is largely improved.
3. The pre-heat treatment including beta solution and ultra-slow furnace heating up process is also suitable to eliminate continuous $\alpha_{GB}$ for laser melting deposited Ti17 near $\beta$ titanium alloy.

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