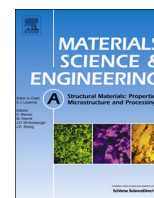




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journal homepage: [www.elsevier.com/locate/msea](http://www.elsevier.com/locate/msea)Subtransus triplex heat treatment of laser melting deposited Ti–5Al–5Mo–5V–1Cr–1Fe near  $\beta$  titanium alloyC.M. Liu<sup>a</sup>, H.M. Wang<sup>a,b</sup>, X.J. Tian<sup>a,b,\*</sup>, H.B. Tang<sup>a,b</sup><sup>a</sup> Laboratory of Laser Materials Processing and Manufacturing, Beihang University, 37 Xueyuan Road, Beijing 100191, China<sup>b</sup> Key Laboratory of Aerospace Materials of the Ministry of Education, Beihang University, 37 Xueyuan Road, Beijing 100191, China

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## ABSTRACT

Laser melting deposition (LMD), which fabricates near-net-shape components directly by melting powder layer by layer, is used to manufacture Ti–5Al–5Mo–5V–1Cr–1Fe near  $\beta$  titanium alloy samples. In as-deposited alloy, ultrafine basket-weave microstructure and continuous grain boundary  $\alpha$  ( $\alpha_{GB}$ ) are observed. And the as-deposited alloy exhibits high strength but low ductility. Then the annealing treatment is applied to improve the ductility, but the strength of the annealed alloy is too low. To obtain good comprehensive tensile properties, the traditional standard triplex heat treatment usually applied for wrought Ti–5Al–5Mo–5V–1Cr–1Fe alloy is employed. The results show that the alloy has high strength. The ductility is also improved, but still not satisfactory, because the continuous  $\alpha_{GB}$  remains and is accompanied with soft  $\alpha$  phase precipitate-free zones (PFZ) which results in the intergranular fracture. Accordingly, a subtransus triplex heat treatment is designed, which can significantly reduce the continuous  $\alpha_{GB}$ . And as expected, the ductility is significantly improved, and the predominant fracture model changes to be transgranular.

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## 1. Introduction

Laser melting deposition (LMD) is a powder-based additive manufacturing process that fabricates full density near-net-shape metal components layer-by-layer [1,2]. It can be single-step and waste-free to fabricate the components using metal powders. Compared with traditional wrought-based method, it can greatly reduce the buy-to-fly ratio and lead time for aerospace titanium alloy components. Accordingly, many studies have been carried out to investigate laser melting deposited titanium alloy.

However, the as-deposited titanium alloys by LMD hardly fulfill the comprehensive mechanical property requirements for aerospace titanium alloy components [3–8]. To increase application performance of laser melting deposited titanium alloys, many studies on heat treatments have been carried out to improve their mechanical properties [4,9–14]. Brandl et al. [9] found that the 600 °C/4 hours (h) heat treatment could lead to a significantly higher strength of laser melting deposited Ti–6Al–4V alloy, but also decreased ductility. Vrancken et al. [4] demonstrated that when laser melting deposited Ti–6Al–4V alloy was annealed at 850 °C /2 h followed by furnace cooling, the elongation would be  $12.84 \pm 1.36\%$ , which was significantly higher than that of the as-

deposited sample ( $7.36 \pm 1.32\%$ ). Tian et al. [13] annealed the laser melting deposited Ti–4Al–1.5Mn alloy at 955 °C /0.5 h followed by air cooling, obtained unique bi-modal microstructure with approximately 35% crab-claw like primary  $\alpha$ , and largely improved the impact toughness. However, the bulk of the existing work has been concerned with laser melting deposited near  $\alpha$  or  $\alpha + \beta$  titanium alloy, including Ti–6Al–4V, Ti–6Al–2Zr–1Mo–1V, Ti–4Al–1.5Mn etc [4,9–14]. No such studies have been carried out in addressing the suitable heat treatment for laser melting deposited near  $\beta$  titanium alloys.

Recently, near  $\beta$  and  $\beta$  titanium alloys have continued on an increasing basis to be a very important class of materials, because they offer the highest strength to weight ratios and very attractive combinations of strength, toughness, and fatigue resistance at large cross sections [15,16]. Ti–5Al–5Mo–5V–1Cr–1Fe alloy, as a typical high strength near  $\beta$  titanium alloy, is widely used for aerospace structural components [16–18]. Thus, it is chosen to be investigated in this paper. For wrought Ti–5Al–5Mo–5V–1Cr–1Fe alloy samples, the triplex heat treatment is found to be an effective way to obtain good comprehensive mechanical property [19–23]. The alloy after triplex heat treatment generally exhibits duplex microstructure with coarse primary  $\alpha$  ( $\alpha_p$ ) and fine secondary  $\alpha$  ( $\alpha_s$ ) in the  $\beta$  matrix [22,23]. The triplex heat treatment process mainly contains three steps (see Fig. 1): (i) heating to 820–850 °C ( $T_1$ ), holding for 1–3 h, and furnace cooling (FC) to 740–760 °C ( $T_2$ ); (ii) holding at 740–760 °C ( $T_2$ ) for 1–3 h and air cooling (AC); (iii) aging at 500–650 °C ( $T_3$ ) for 2–6 h and air cooling [21]. As pointed

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out in the previous studies [22–24], the recommended triplex heat treatment process is 830 °C/2 h, FC+750 °C/2 h, AC+600 °C/4 h, AC as shown in Fig. 1, which is named as standard triplex heat treatment.

The natural question then is whether the standard triplex heat treatment is also suitable for laser melting deposited Ti–5Al–5Mo–5V–1Cr–1Fe alloy. The present work will firstly answer this question and investigate the microstructure and tensile properties of the standard triplex heat treated samples. It is found that the ductility is improved, but still unsatisfactory. Thus, for laser melting deposited samples, there must be other factors which influence the ductility. Our work will then focus on finding these factors and designing a more suitable heat treatment method to further improve the mechanical properties of laser melting deposited Ti–5Al–5Mo–5V–1Cr–1Fe alloy.

## 2. Experimental procedures

The laser melting deposition system consisted of a YSL-10000 fiber laser, a BSF-2 powder feeder together with a co-axial powder delivery nozzle, and a Fagor-8055 computer numerical control (CNC) four-axis working table. This was used to fabricate plate-like samples. In order to prevent the melt pool from oxidizing, the experiments were conducted inside an argon-purged processing chamber with oxygen content less than 100 ppm. The LMD processing parameters were as follows: laser power was 5000 W, beam diameter was 6 mm, laser scanning speed was 800–1500 mm/min, and powder feed rate was 15–25 g/min. The dimensions of the plate-like sample were approximately 400 mm × 300 mm × 40 mm.

Table 1 showed the heat treatment processes. Firstly, a simple annealing treatment 750 °C/2 h, AC was applied. Then the standard triplex heat treatment was investigated, which was 830 °C/2 h, FC+750 °C/2 h, AC+600 °C/4 h, AC. Here, 830 °C/2 h, WQ (water quench) and 830 °C/2 h, FC+750 °C/2 h, AC were selected to

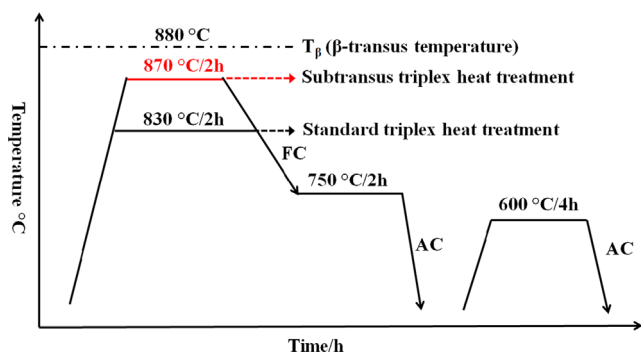


Fig. 1. Schematic illustration of triplex heat treatment process for Ti–5Al–5Mo–5V–1Cr–1Fe alloy.

Table 1

Heat treatments for investigating the microstructure evolution and tensile properties.

Number	Heat treatment process	Application
1	750 °C/2 h, AC	Annealing treatment
2	830 °C/2 h, FC+750 °C/2 h, AC+600 °C/4 h, AC	Standard triplex heat treatment
3	830 °C/2 h, WQ	
4	830 °C/2 h, FC+750 °C/2 h, AC	
5	870 °C/2 h, FC+750 °C/2 h, AC+600 °C/4 h, AC	Subtransus triplex heat treatment
6	870 °C/2 h, WQ	
7	870 °C/2 h, FC+750 °C/2 h, AC	

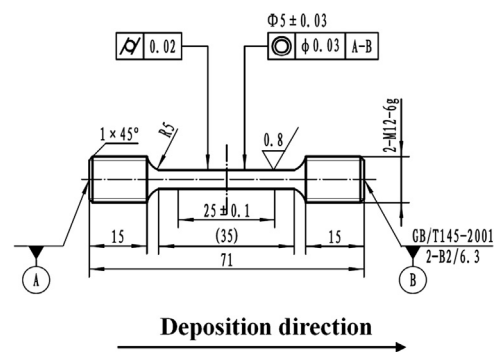


Fig. 2. Geometrical shape and size of tensile test specimen.

investigate the microstructure evolution in this process. In the following, a subtransus triplex heat treatment was designed (see Fig. 1), which was 870 °C/2 h, FC+750 °C/2 h, AC+600 °C/4 h, AC. Moreover, 870 °C/2 h, WQ and 870 °C/2 h, FC+750 °C/2 h, AC were selected to investigate the microstructure evolution in this process. The  $\beta$ -transus temperature ( $T_\beta$ ) of the as-deposited Ti–5Al–5Mo–5V–1Cr–1Fe alloy was  $880 \pm 5$  °C, determined by metallographic method. All heat treatments were performed in air in an electric-resistance furnace.

Metallographic specimens were prepared by conventional mechanical polishing method. A mixture of 1 ml HF, 6 ml HNO<sub>3</sub> and 100 ml H<sub>2</sub>O was used as the etching agent. The microstructures of samples were characterized by optical microscopy (OM) and scanning electron microscopy (SEM). Quantitative measurement of the width, length and aspect ratio of primary  $\alpha$  were conducted on at least five SEM micrographs with a magnification of 1000 for each specimen.

The dimensions of the heat treated samples for tensile test were 70 mm × 45 mm × 15 mm. After heat treatments, three columnar specimens with length 70 mm and diameter  $\phi$  12 mm were machined by electric discharge wire cutting from the center of heat treated samples. Then, the columnar specimens were processed into the tensile specimen with  $\phi$  5 mm as shown in Fig. 2. During the tensile sample machining, the thickness of the removed parts was at least 3.5 mm. Thus, although the heat treatment was in air, the oxidized layer about 0.2 mm was removed completely and would not affect the experiments. Room-temperature tensile properties were tested according to the test standard of ISO 6892-1: 2009. Here, the axial direction of specimens was parallel to the deposition direction. The deformation strain was measured by an extensometer. Three tensile test samples were tested for each treatment to determine the mechanical properties. The fracture surfaces and cross sections of the tensile test specimens were examined by SEM and OM, respectively.

## 3. Results and discussion

### 3.1. As-deposited alloy and annealing treatment

The as-deposited Ti–5Al–5Mo–5V–1Cr–1Fe alloy exhibits two microstructural characteristics as shown in Fig. 3a. One is the ultrafine basket-weave microstructure, with the width of  $\alpha$  laths about 0.15  $\mu$ m. This results from the high cooling rate during LMD. The other is the continuous  $\alpha$  phase at the  $\beta$  grain boundary ( $\alpha_{GB}$ ). The width of  $\alpha_{GB}$  is about 0.45  $\mu$ m. The formation of  $\alpha_{GB}$  is associated with the  $\alpha$  phase heterogeneous nucleation during the  $\beta$ → $\alpha$  phase transformation [25]. For titanium alloys,  $\alpha$  phase always preferentially precipitates at the  $\beta$  grain boundaries [26] and tends to form continuous  $\alpha_{GB}$ . It should be mentioned that the martensite phase is not observed in the as-deposited sample,

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