



# Development of a pre-heat treatment for obtaining discontinuous grain boundary $\alpha$ in laser melting deposited Ti–5Al–5Mo–5V–1Cr–1Fe alloy

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## ABSTRACT

Continuous grain boundary  $\alpha$  ( $\alpha_{GB}$ ) is widely present in near  $\beta$  and  $\beta$  titanium alloys, and is deleterious to the ductility. Although traditional wrought-based processing can reduce the continuous  $\alpha_{GB}$ , they are not suitable for titanium alloy components fabricated by near-net-shape processing methods, because the wrought-based processing is accompanied by shape change. Hence, the aim of this work is to develop a new heat treatment method, which can obtain discontinuous  $\alpha_{GB}$  and apply to near-net-shape titanium alloy components. First, the detailed formation process of the continuous  $\alpha_{GB}$  was investigated to determine the factors which control the continuous extent of  $\alpha_{GB}$ . Then, based on that investigation, a novel pre-heat treatment was designed through directly controlling the nucleation rate and growth morphology of  $\alpha_{GB}$ . The results indicated that the pre-heat treatment could lead to coarse and discontinuous  $\alpha_{GB}$  particles, and hence the ductility of Ti–5Al–5Mo–5V–1Cr–1Fe alloy was significantly improved. Furthermore, a parameter named discontinuous  $\alpha_{GB}$  density was first proposed to quantitatively characterize the discontinuous extent of  $\alpha_{GB}$ . A simple linear relationship between this parameter and the ductility was discovered, which made it possible to predict the ductility of Ti–5Al–5Mo–5V–1Cr–1Fe alloy by the observation of  $\alpha_{GB}$ .

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## 1. Introduction

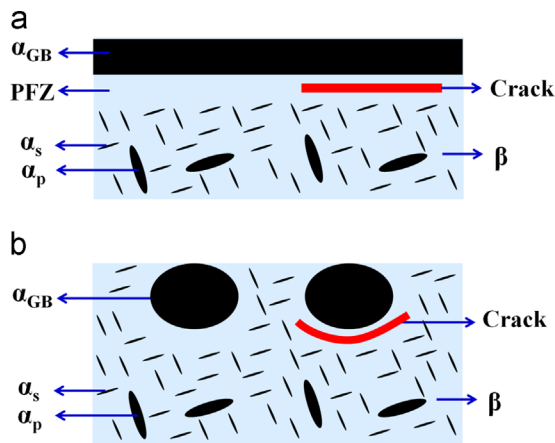
Near  $\beta$  and  $\beta$  titanium alloys, such as VT22 (Ti–5Al–5Mo–5V–1Cr–1Fe), Ti–6246, Ti–17,  $\beta$ -CEZ and Ti–10–2–3, are widely used in the area of aerospace, because they can offer high strength to weight ratios and very attractive combinations of strength, toughness and fatigue resistance [1–4]. However, the procedure to obtain such good mechanical properties is not simple. The mechanical properties of these alloys are determined by many factors, including grain boundary  $\alpha$  ( $\alpha_{GB}$ ),  $\beta$  grain size, as well as the shape, size and volume fraction of primary  $\alpha$  ( $\alpha_p$ ) and fine secondary  $\alpha$  ( $\alpha_s$ ) [1]. The focus of this paper is laid on the effect of  $\alpha_{GB}$ , since it is of critical importance to the ductility of titanium alloys [5–7]. Its influence mechanism on ductility can be simply understood as follows [5–8] (see Fig. 1). The  $\alpha_{GB}$  generally exhibits continuous layer morphology, because the  $\alpha$  phase nucleates and grows preferentially at  $\beta$  grain boundaries during  $\beta \rightarrow \alpha$  phase transformation. Accompanied by the formation of  $\alpha_{GB}$ ,  $\alpha$  stabilizing elements (Al and O) diffuse into the  $\alpha_{GB}$  from the adjacent

zone where the  $\beta$  phase is subsequently stabilized. This prevents the precipitation of the fine hardening  $\alpha_s$ , and results in the formation of soft precipitate-free zones (PFZ) along the  $\alpha_{GB}$  [5,8] as schematically shown in Fig. 1a. Then the high yield stress difference between the soft PFZ and the strengthened  $\beta$  matrix can easily lead to the early crack nucleation [2]. Meanwhile, since the PFZ is continuous and widespread, the crack naturally propagates along the PFZ (see Fig. 1a), causing intercrystalline fracture and low ductility [5,8]. In contrast, if the  $\alpha_{GB}$  is discontinuous, the fine hardening  $\alpha_s$  between the discontinuous  $\alpha_{GB}$  will inhibit the crack propagation along the grain boundary as schematically shown in Fig. 1b, and hence can lead to transcrystalline fracture and high ductility [5,8]. Thus, obtaining discontinuous  $\alpha_{GB}$  is very important for improving the ductility of titanium alloys.

Several attempts have been made to obtain discontinuous  $\alpha_{GB}$ . The widely used methods are the wrought-based processing [2], such as  $\beta$  processing, through-transus processing, etc. The development of these methods is one of the keys for the successful application of high-strength wrought titanium alloys. However, with rising titanium alloy usage, the near-net-shape processing methods have received a great deal of attention [2], because they can realize high material utilization and fabricate complex-shaped components. One important question then is how to obtain discontinuous

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**Fig. 1.** Schematic illustration of the crack initiation and propagation in titanium alloys with (a) continuous  $\alpha_{GB}$  and (b) discontinuous  $\alpha_{GB}$ .

$\alpha_{GB}$  in near-net-shape titanium alloy components. Obviously, the wrought-based processing cannot be used because it is accompanied by obvious shape change. Currently, there are still very limited efforts attempting to address this question in the literatures.

Heat treatment may offer an alternative pathway toward obtaining discontinuous  $\alpha_{GB}$ , because it can significantly affect the  $\beta \rightarrow \alpha$  and  $\alpha \rightarrow \beta$  phase transformations. Two types of heat treatment methods can be considered. The first one is dissolving the continuous  $\alpha_{GB}$  during the  $\alpha \rightarrow \beta$  phase transformation by heat treatment at the  $\alpha + \beta$  phase field. In our previous studies [8], we designed the subtransus triplex heat treatment (STHT), which could make the continuous  $\alpha_{GB}$  effectively dissolve and globularize, and hence led to discontinuous  $\alpha_{GB}$ . The method sheds some light on obtaining discontinuous  $\alpha_{GB}$  by heat treatment, but possibly cannot be widely used in industrial applications. Because the subtransus temperature (about  $T_{\beta} - 10^\circ\text{C}$ ) is very close to the  $\beta$ -transus temperature ( $T_{\beta}$ ), the titanium alloy components may be heated to the  $\beta$  phase field. Then, the  $\beta$  annealed microstructures may form and the continuous  $\alpha_{GB}$  will re-form. The second method is directly obtaining discontinuous  $\alpha_{GB}$  through controlling the nucleation rate and growth morphology of  $\alpha_{GB}$  during the  $\beta \rightarrow \alpha$  phase transformation by heat treatment. To the best of our knowledge, this kind of heat treatment has not yet been proposed. Thus, the present work aims at designing such heat treatment.

Furthermore, previous studies only stated that the discontinuous  $\alpha_{GB}$  was beneficial to the high ductility of titanium alloys [2,5,6]. Little attention has been devoted to quantitatively characterizing the discontinuous extent of  $\alpha_{GB}$ , and establishing the link between the discontinuous extent of  $\alpha_{GB}$  and ductility. Apparently, this is valuable not only to improve our understanding of the influence of  $\alpha_{GB}$  on ductility, but also to predict the ductility of titanium alloys simply by the observation of  $\alpha_{GB}$ . In this paper, an easily measurable parameter is defined to characterize the discontinuous extent of  $\alpha_{GB}$ , and the connection between this parameter and ductility is preliminarily addressed.

## 2. Experimental procedures

The material used in this work is Ti–5Al–5Mo–5V–1Cr–1Fe. It is a typical high strength near  $\beta$  titanium alloy. The  $T_{\beta}$  of the alloy was determined as  $880 \pm 5^\circ\text{C}$  by the metallographic method. The applied near-net-shape processing method was chosen as laser melting deposition (LMD), which could fabricate full density metal components layer-by-layer using metal powders [8–15].

The detailed LMD fabrication process has been reported in our previous papers [9,10]. A plate-like sample with dimensions approximately  $400\text{ mm} \times 300\text{ mm} \times 40\text{ mm}$  was fabricated by LMD. The 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.

To investigate the formation process of the continuous  $\alpha_{GB}$ , three types of heat treatment were applied, including (a)  $900^\circ\text{C}/0.5\text{ h}$  (h), furnace cooling (FC)+ $830^\circ\text{C}$ , water quench (WQ); (b)  $900^\circ\text{C}/0.5\text{ h}$ , FC+ $800^\circ\text{C}$ , WQ; and (c)  $900^\circ\text{C}/0.5\text{ h}$ , FC+ $770^\circ\text{C}$ , WQ. The furnace cooling rate was  $3^\circ\text{C}/\text{min}$ . To obtain discontinuous  $\alpha_{GB}$ , three types of pre-heat treatment were designed, including (a)  $900^\circ\text{C}/0.5\text{ h}$ , FC+ $830^\circ\text{C}/2\text{ h}$ , WQ; (b)  $900^\circ\text{C}/0.5\text{ h}$ , FC+ $840^\circ\text{C}/2\text{ h}$ , WQ; and (c)  $900^\circ\text{C}/0.5\text{ h}$ , FC+ $850^\circ\text{C}/2\text{ h}$ , WQ. For simplicity, the above three types of pre-heat treatment were called P<sub>830</sub>, P<sub>840</sub>, and P<sub>850</sub>, respectively. To investigate the effect of pre-heat treatment on the tensile properties of Ti–5Al–5Mo–5V–1Cr–1Fe alloy, the traditional triplex heat treatment  $830^\circ\text{C}/2\text{ h}$ , FC+ $750^\circ\text{C}/2\text{ h}$ , air cooling (AC)+ $600^\circ\text{C}/4\text{ h}$ , AC [8,16] (THT) and the pre-heat treatment+THT (P<sub>830</sub>THT, P<sub>840</sub>THT and P<sub>850</sub>THT) were applied for comparison.

Metallographic specimens were prepared by the 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 the samples were characterized by optical microscopy (OM) and scanning electron microscopy (SEM). To quantitatively characterize the discontinuous extent of  $\alpha_{GB}$ , the number of  $\alpha_{GB}$  ( $n$ ) and the length of  $\beta$  grain boundary ( $L$ ) were measured using the metallographic image analysis software SISC IAS v8.0. These quantitative measurements were conducted on at least 10 OM micrographs with a magnification of 500 for each specimen. The tensile tests were based on 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 for three tensile specimens. Moreover, the fracture surfaces of the tensile test specimens were examined by SEM.

## 3. Results and discussion

### 3.1. Formation process of continuous $\alpha_{GB}$

The detailed formation process of the continuous  $\alpha_{GB}$  during cooling from the  $\beta$  phase field to the  $\alpha + \beta$  phase field is investigated as shown in Fig. 2. When the sample is cooled to  $830^\circ\text{C}$ , the degree of undercooling is low. Thus, the amount of  $\alpha_{GB}$  nuclei is small [17] and only a few  $\alpha_{GB}$  particles form as shown in Fig. 2a. Then, when the sample is cooled to  $800^\circ\text{C}$ , the  $\alpha_{GB}$  exhibits nearly continuous morphology with large numbers of small  $\alpha_{GB}$  particles as shown in Fig. 2b. This suggests that large amounts of  $\alpha_{GB}$  nucleate and grow to connect with each other. When the sample is cooled to  $770^\circ\text{C}$ , the  $\alpha_{GB}$  completely exhibits continuous morphology (see Fig. 2c). Moreover,  $\alpha$  Widmanstätten grain boundary ( $\alpha_{WGB}$ ) nucleates through the surface instability and protuberance of  $\alpha_{GB}$  [18], and then grows into  $\beta$  grains with colony morphology.

The formation of continuous  $\alpha_{GB}$  consists of nucleation and growth [19]. Based on the above results, it can be found that the nucleation is the dominant factor. For such large amounts of  $\alpha_{GB}$  nuclei, they can easily grow to connect with each other, and hence result in continuous  $\alpha_{GB}$  as schematically shown in Fig. 3a. Thus, to obtain discontinuous  $\alpha_{GB}$ , the amount of  $\alpha_{GB}$  nuclei should be small. It is natural to think that the few  $\alpha_{GB}$  nuclei can be obtained

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