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The vertical and triangular morphology in the as-deposited Ti-6Al-4V

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ABSTRACT

The martensite plates in the metal alloys form the special morphologies, such as the triangular, spike, diamond and butterfly morphologies. The Ti-6Al-4V processed by laser melted deposition shows the fine martensite in the prior columnar beta grains. No relevant report has shed light on the specific morphology of the martensite plate group. The current work reveals that the martensite plates cluster as the vertical and the triangular morphologies. The vertical morphology is readily observed by optical microscope and transmission electron microscope. Whereas the triangular morphology is solely characterized in the transformation electron microscope. It reveals that the martensite plates in the triangular morphology are finer than that in the vertical morphology. Besides, the triangular and vertical morphology are related to the misorientations 60° or 90° or so. The misorientation distribution indicates the proportion of these morphologies to some degree. Thus the microstructure model, including the information of the size and the proportion of these morphologies, is appropriately proposed. Noting that the thermal cycles happen in laser melted deposition, it is concluded that the vertical morphology forms as the primary martensite and the triangular morphology forms as the secondary martensite.

1. Introduction

Titanium alloys possess a high strength to weight ratio, better biocompatibility over other alloys. And they are widely used in the military field, medical applications and sports components. The two phases alloy Ti-6Al-4V can balance various properties and has become one of the most popular titanium alloys.

The additive manufacturing (AM) is characterized for its material saving, precise formation and decreased defect properties. Shaped metal deposition [1], selective laser melting [2], the selective electron beam melting [3] and laser melted deposition belong to the process of AM. Researchers have paid much attention on the process parameters, microstructure, microtexture and mechanical properties of the Ti alloys processed by AM. Generally speaking, the mechanical properties depend on the microstructure and microtexture. For example, Ti-6Al-4V alloy processed by laser melted deposition (LMD) has good strength and poor ductile property. It results from the fine martensite in the corresponding microstructure. And the relevant research on the process parameters [6,13] aims to control the microstructure. Therefore, the preference is given to study the microstructure and microtexture in the Ti alloys processed by AM. And there are significant reports on the microstructure of the Ti alloy processed by AM. Xinhua Wu et al. [5] reported that the laser-deposited Ti alloy consisted of the columnar β grains with martensite in Ti-6Al-4V alloy. S. M. Kelly and S. L. Kampe [4] revealed the microstructure evolution with the increasing layers in the laser-deposited Ti alloy. W. Xu et al. [6] obtained the ultrafine lamellar α + β microstructure in the Ti alloy made by selective laser melting. The ultrafine microstructure improved the yield strength. S. L. Lu et al. [7] observed the massive microstructure in Ti-6Al-4V processed by selective electron beam melting. However, the reports on the microstructure of Ti alloy processed by AM are far from satisfying the industrial application and solving the production problem. There are many points worth investigating. At least up to now there are few reports involving in these martensite groups in the laser melted Ti alloys. In other words, the martensite clusters of the Ti alloy processed by LMD have not been observed experimentally. In contrast, many papers have illustrated the special martensite groups in forged or ingot alloys [8,16–21]. These special morphologies, including the triangular, butterfly or spike martensite groups in forged alloys, are characterized and analyzed extensively and deeply. These martensite clusters play an important role in the phase transformation. Especially, the triangular morphology is a kind of typical morphologies. And it receives much attention [15]. The triangular morphology in laser melted Ti alloy is first characterized in the present paper.

The object of the study is the Ti-6Al-4V processed by LMD. The present paper aims to point out the special morphology from the viewpoint of the microstructure and microtexture. The vertical or triangular morphology is observed by optical microscope (OM), scanning electron microscope (SEM), and transmission electron microscope (TEM) observation. Furthermore, the proportion of these morphologies

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is obtained from the misorientation distribution in electron back-scattered diffraction (EBSD).

2. Experimental Procedure

A cubic sample with approximate dimension 100 mm × 100 mm × 12 mm was fabricated by LMD. A fiber laser with a 1070 nm wavelength was used as the heat source. The LMD process is protected by an argon shielding gas (purity of 99.99%) fed through a coaxial nozzle with a flow rate of 6 l/min. The processing parameters are as follows: laser power is 500 W, beam diameter is 0.6 mm, powder feeding rate is 5 g/min. The sample was machined in super computer numerical control (CNC) wire cutting machine.

The Kroll's reagent (HNO_3:HF:H_2O = 2:4:100) was used to etch the samples to reveal the microstructure. A TESCAN LYRA3 SEM was used to examine the microstructure with an EBSD component. The EBSD measurement was performed with an accelerating voltage of 20 kV and a tilt angle of 70°. And the corresponding data was treated with the software Channel 5. Samples for TEM investigations were prepared by a standard twin-jet electropolishing technique using a STruers TehnPol-5 device. Discs of 3 mm in diameter were then punched from the thinned slice and then electro-polished at − 30 °C at a voltage of 30 V, using a solution of 60% methyl alcohol, 35% n-butanol and 5% perchloric acid. TEM investigations were conducted using a JEOF 2010 TEM operated at 200 kV. Noting that the samples involved in characterization are cut from the as-deposited cubic sample in random position, i.e., it is random sampling. It is related to the discussion later.

3. Results

3.1. OM and SEM

The triangular morphology is identified readily in the ingot Ti alloys or forged Ti alloys [16], as shown in Fig. 1.The as-deposited components consist entirely of the martensite α′ phase in the prior columnar β grains that are growing epitaxially across many layers and opposite to the heat flow (seen in Fig. 2(a), (b)). The columnar grains are 300–400 μm in width and 1–2 mm in height. The martensite plate is ultra-fine (Fig. 2(a), (b) and (c)). There is obvious vertical morphology as marked with white line in Fig. 2(b). The vertical morphologies are used to describe the martensite plate clusters in which the adjacent plates are nearly vertical with each other (not totally vertical, allowing 5–10° deviation). The distinct triangular morphology has not been observed in the laser-deposited Ti alloy by OM (Fig. 2(b)). Even though the micrograph in Fig. 2(b) possesses the same scale of 1000 magnitude with Fig. 1.

3.2. TEM

The vertical morphology is also verified in the careful TEM observation (Fig. 3).

Its width is 200–300 nm. It is worth mentioning that the triangular morphology is observed clearly in TEM (Fig. 4). It is difficult to detect the triangular morphology in the optical micrograph. This is because the width of the martensite plates in the triangular morphology (50–200 nm) is significantly smaller than that of the vertical morphology (200–300 nm). The observation result is different from the model that Jingjing Yang et al. have proposed in which the martensite plate is vertical with each other [13]. In their work, the Ti alloy is made by selective laser melting, and all the martensite plates are nearly vertical. In the present case, the Ti alloy is processed by laser melted deposition, and the martensite clustered as the triangular morphology with the near 60° angles. The alternative reason for this is the difference on the process. The selective laser melting possess higher cooling rate compared with the laser melted deposition. The cooling rate plays an important role in the martensite clusters. And it will be discussed later.

As shown in Fig. 4(e), the mother phase β and product phase α′ obey the Burgers orientation relationship (BOR):

$$(110)_{\beta}//(0001)_{\alpha'}, \quad [1-11]_{\beta}//[11-20]_{\alpha'}$$

It is validated in the following pole figure (Fig. 5(c) and (d)). And the dark-field micrographs, shown in Fig. 4(f), reveal that the three laths possess the same orientation relationship.

3.3. EBSD

In the pole figure, the oval line in $(0001)_{\alpha'}$ and $(110)_{\beta}$ are in the same position, so do the rectangular line in $(11-20)_{\alpha'}$ and $(111)_{\beta}$. Therefore the as-deposited sample follows the BOR. It is consistent with the references [10,11] and the SAED result in Fig. 4(e) and (f).

Emphasis is put on the discussion of the misorientation distribution. On one hand, the misorientation refers to the phase boundaries of the α. The misorientation frequency is obtained from the statistics of the EBSD data experimentally. On the other hand, the misorientation is used to describe the orientation relationship of two neighbouring variants of the martensite α′. These variants obey the BOR first, then is described by the misorientation further. These types of the misorientations are list in Table. 1.

According to Fig. 5(b), specific types of misorientations have ambitious frequency, especially the near angle 60 or angle 90°. That is, there are two dominant peaks at misorientations of around 60° and 90°. As shown in Table. 1, the 60°, 60.83° and 63.26° belong to type 2, type 3 and type 4, respectively. 90° belong to the type 5. Misorientations type 2 and type 4 occupy more proportion than type 3 since they are advantageous to reduce more shape strain when forming the martensite plates [14]. And the misorientation 90° accounts for less proportion compared with the 60° and 63.26°. Therefore, to be precise, the misorientation type 2 and type 4 occupy first and the misorientation type 5 comes second. It is safe to conclude that the vertical morphology cause the 90° misorientations, while the triangular morphology causes 60° and 63.26° misorientations. The detailed reason will be discussed later. The misorientation distribution reveals that the vertical morphology is less than the triangular morphology in number. And it is consistent with the fact that the triangular morphology is frequently observed in TEM. In order to understand the number of these two morphologies in depth, the complex TEM analysis is needed and the total shaped strain energy of the triangular morphology should be calculated. Some researchers have done the similar work on Ti—Nb alloy [16].

4. Discussion

4.1. The BOR and Misorientation

The as-deposited Ti alloy obeys the BOR, which is verified in TEM observation (Fig. 4(e)) and the pole figures (Fig. 5(c) and (d)). Also, many papers involving in the other additive manufacturing (like the selective laser melting) confirmed the BOR [11]. There are twelve equivalent variants of the BOR relating the β matrix to the α product phase. There are 144 combinations from a combination of any two of the 12 variants. However, many of the combinations are equivalent and only six distinct types of α/α exist (Table 1). The misorientations, discussed in the following part, is based on the BOR. That is, the five types of misorientations are conditional on the BOR.

EBSD samples are typically tilted towards the detector by 70°. The vertical and triangular morphology with different special orientations lie in polishing surface of the sample. The misorientation frequency statistics deriving from the EBSD is the observation result viewing from the random directions for the cuboid or the tetrahedron structure in the as-deposited Ti alloy microstructure. In contrast, the vertical or the triangular morphology characterized by TEM is viewed from the axis paralleling or near paralleling to the habit plane. Strictly speaking, the angles measured in the vertical or triangular morphology are consistent with the angles among these habit planes. But it is not consistent with