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Microstructure evolution of isothermal holding treatment during melt solidification of Ti−6Al−4V alloy

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Abstract: Effect of isothermal holding treatment in the solidification process on the microstructure of Ti−6Al−4V alloy was studied by temperature controlled induced melting apparatus. The result shows that with isothermal holding treatment above the β transus temperature during solidification, the colony structure consisting of parallel lamellae was obtained. While the isothermal holding treatment was set at 960 °C, a unique bi-modal microstructure consisting of coarse primary α and fine secondary lamellar α was obtained. The primary lamellar α tended to break into several pieces, globularize and present equiaxed morphology. The formation mechanism of the equiaxed α can be explained with the atom immigration, high density dislocations, combined action with the interface tension of formed α phase during the isothermal holding treatment. After the isothermal holding, the retained β matrix transformed into fine lamellar α, thus, bi-modal microstructure was acquired. Compared with the lamellar structure, the grain boundary α presented discontinuously and cannot be distinguished from the primary α lamellae easily. The size of colonies α was greatly decreased. The microstructure tended to be much more homogeneous in the whole section of the samples.

Key words: titanium alloy; isothermal holding treatment; bi-modal structure; grain boundaries; equiaxed α phase

1 Introduction

Titanium alloys are important aerospace materials for aircraft and aeroengine applications because of their high specific strength and ability to withstand elevated temperatures up to 600 °C [1]. Like other cast metals, the in-service and processing properties of titanium castings are very dependent on the microstructure including microstructure type, phase fraction and grain size [2]. However, the microstructure of titanium alloy cannot easily be refined by normal melt treatment due to its high reactivity. Meanwhile, with the low cooling rate induced by its low conductivity, the grain size of titanium alloys tends to be coarse, and the microstructure of titanium alloy cast part is inclined to be inhomogeneous with different thicknesses [3]. Consequently, how to acquire refined and homogeneous microstructure is a big challenge to the titanium alloys.

The multiple phase transformations that occur in titanium alloys provide the opportunities for microstructure designing according to specific applications [4]. The two-phase α–β titanium alloy Ti–6Al–4V is the backbone of the titanium industry especially for aerospace applications [5]. Many works have been delivered on the heat treatment of Ti−6Al–4V castings [6–10]. Beta solution treatment and overaging (BSTOA) patented by Boeing Company, which is widely used for titanium alloys, has been successfully used to overcome the property losses, such as reductions in tensile strength as the grain size becomes coarser, in part through the microstructural refinement within the grains [6,7]. MUTOMBO et al [8] draw a conclusion that mill-annealed and furnace cooled Ti−6Al−4V alloy has the most acceptable combination of strength and ductility. The influence of the solution treatment at 1050, 950 and 800 °C with water or air cooling followed by aging treatment at 550 °C was investigated on the specimens from Ti−6Al−4V model titanium alloy by PINKE et al [10], and the corresponding microstructure as well as the properties of the Ti−6Al−4V alloy was acquired. It was found that the highest increase of hardness (above

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HV 100) relative to the initial hardness was detected after the heat treatment at (1050 °C, 1 h, water) + 550 °C aging. OI et al [11] recommended a heat cycle to guarantee uniform microstructure of Ti−6Al−4V, consisting of heat treatment to introduce nuclei for α grains and further heat treatment to let fine α grains grow uniformly. In conclusion, the main aim of these researches is to eliminate grain boundary α phase, large α plate colonies and individual α plates, which can refine the microstructure and elevate the strength at evaluated temperature.

However, previous studies mainly focused on the conventional heat treatment, which requires a heating process from room temperature. It is not only time-consuming and power-costly, but also may result in casting distortion even crack when the residual stress of solidified alloy is high. In this regard, an interesting way of microstructure control for Ti alloy is put forward in the current study, that is, an additional isothermal holding treatment in β phase is performed following the melt solidification. The purpose is to obtain a refined and homogeneous microstructure via once solidification without a following heat treatment. Based on the experimental results with different holding temperatures, the microstructure evolution and solidification behavior under isothermal holding treatment are discussed.

2 Experimental

The starting material was hot rolled Ti−6Al−4V plate. Samples were induction-heated and melted in zirconium oxide crucibles with the inner diameter of 20 mm and the depth of 25 mm, as seen in Fig. 1(a). To minimize the oxygen contamination, the zirconium oxide crucibles were coated with yttrium oxide and roasted in a vacuum furnace before installation. The working chamber was made of quartz and was initially evacuated to about 7.0×10^{-2} Pa, then backfilled with high purity argon gas (the purity is higher than 99.999 %). The temperature was measured by ZMPAC ZSQ5 two-colour infrared pyrometer with an accuracy of ±5 °C. The process of temperature control during solidification is presented in Fig. 1(b). The melt temperature is 1750 °C, which is about 100 °C higher than the liquidus of Ti−6Al−4V alloy. When the cooling begins, the bulk temperature decreases to β phase transus temperature with the cooling rate of 10 K/s. Then, the isothermal treatments were performed at two temperatures for 30 min followed by a furnace cooling. Further, the samples to be analyzed were prepared following standard grinding, electrolytic polishing and etched in etchant containing 5% HF, 10% HNO_3 and 85% H_2O for 10 s. The OLYMPUS/PMG3 optical microscope (OM) and JSM−6700F FEG scanning electron microscope (SEM)

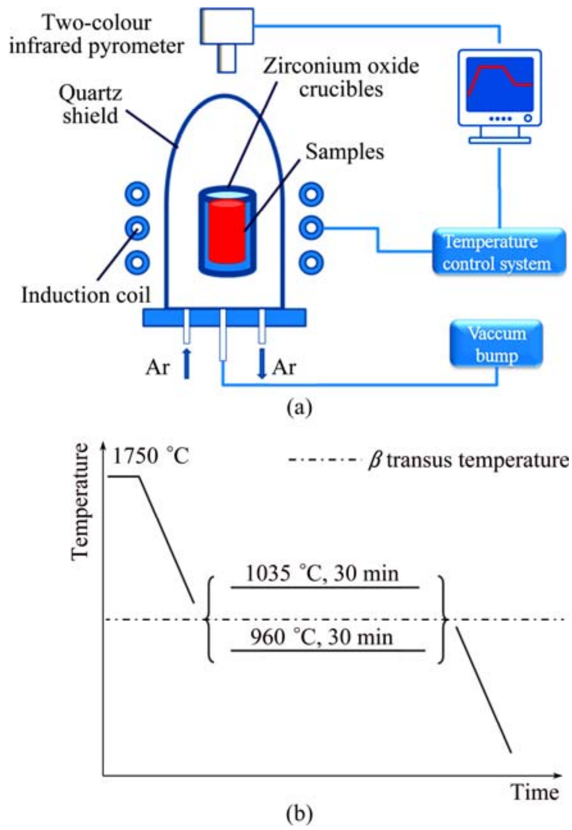

Fig. 1 Schematic illustration of temperature controlled induced melting apparatus (a) and isothermal holding treatment during solidification process (b)

were used to observe the microstructure. The FEI Tecnai G2 F30 transmission electron microscope (TEM) was applied to analyzing the dislocations of equaixed α and index the orientation between α precipitates and β matrix.

3 Results and discussion

The β phase decomposes by a diffusional process (nucleation and growth) or by a diffusionless process (martensitic transformation) [12]. At lower cooling rates, α phase precipitates as a result of diffusional transformation [13]. It is universally known that β transus temperature of Ti−6Al−4V is about 995 °C [14]. When the samples are cooled directly after melting and the temperature decreases to $\alpha+\beta$ phase field, as shown in Fig. 2, the β grains present columnar appearance with the direction of heat flux, which can be distinguished from the grain boundary α as marked in the OM image. As the temperature decreases into the phase transformation field, the α plates form with their basal (close-packed) plane parallel to a special plane in the β phase. Upon slow cooling, a nucleus of α phase forms, and because of the close attomic matching along this common plane, the α phase thickens relatively slowly perpendicular to this

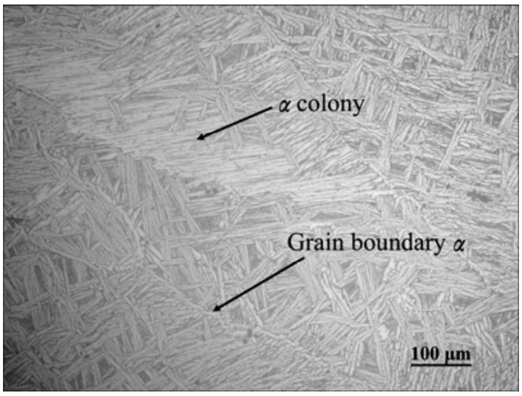

Fig. 2 OM image of microstructure of Ti−6Al–4V alloy cooled directly after melting

plane but grows faster along the plane [15], thus lamellar structure is developed as shown in Fig. 2.

Figure 3 shows the microstructure of Ti−6Al−4V samples isothermally held at 1035 °C and 960 °C for 30 min during solidification process. It can be seen that when preserving heat slightly above the β transus temperature, i.e., 1035 °C, the colony microstructure consisting of parallel lamellae is obtained, with much finer lamellae than the microstructure cooled directly as shown in Fig. 2. The continuous and straight grain boundary α can be distinguished clearly. When the preservation heat temperature is decreased to α+β phase area, the obtained microstructure consists of coarse lamellar α phase and fine secondary α phase. The microstructure is much more homogeneous in the whole section (Figs. 3(c) and (d)). The grain boundary α phase cannot be distinguished easily.

In α+β titanium alloys, the continuous α layers lead to significant softer zones along the boundaries as compared with the surrounding β matrix [16]. A lowering in tensile properties and fatigue strength under existence of grain boundary α has been recognized by FROES [17]. It is reported that microstructure without grain boundary α can be obtained when the cooling rate is kept higher than 20 K/s [11]. However, for normal casting, the cooling rate is far lower than this threshold, so grain boundary α cannot be inhibited. From the microstructure obtained at different isothermal holding treatment temperatures during solidification, it can be observed that when the heat is preserved above β transus temperature, grain boundary α phase can be clearly observed and appears straight and continuous. When the heat is preserved in the α+β phase area for 30 min, grain boundary α phase has a zig-zagged appearance and is hard to be distinguished, as shown in Fig. 3(d). This transformation might alleviate the deteriorate effect caused by continuous and soft grain boundary α phase.

The amount of equiaxed α phase and the coarseness or fineness of the transformed β products affect the properties of titanium alloy [9]. The equiaxed α phase can decrease the anisotropy of α colonies, and minimize the discrepancy of different sizes of α colonies, which is universal in the microstructure of casting. The SEM images of the microstructure isothermally held at 960 °C are shown in Fig. 4. It can be observed that the lamellar α phases present curved morphology, rather than straight

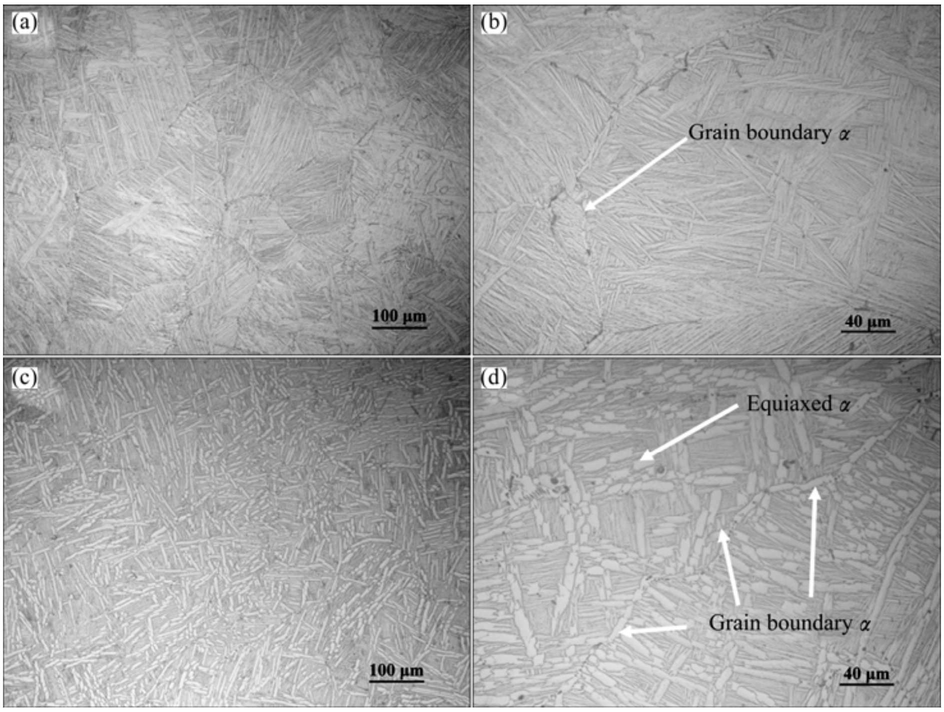

Fig. 3 OM images of Ti–6Al–4V alloy isothermally held at 1035 °C (a, b) and 960 °C (c, d) for 30 min

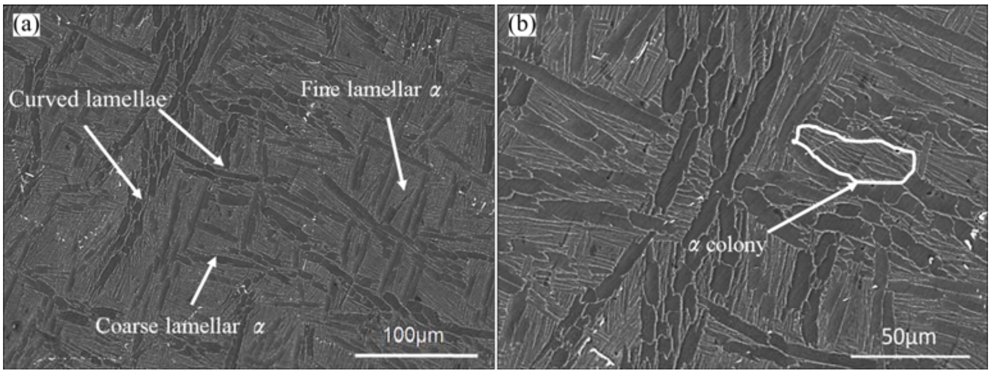

Fig. 4 SEM images of microstructure of Ti−6Al−4V alloy isothermally held at 960 °C for 30 min

morphology as the lamellar structure shown in Fig. 2. A great majority of lamellar α phase coarsen, break into pieces and globularize, present equiaxed morphology eventually by isothermal holding treatment in α+β phase area. A unique bi-modal microstructure consisting of coarse and equiaxed primary α phases and fine lamellar α phases was obtained. The size of lamellar α colony is about 50 μm, as shown in Fig. 4(b). It is universally known that the most influential microstructural parameter on the mechanical properties of fully lamellar structures is α colony size [18]. The small size of α colony can decrease the slip length, improve the ductility and increase the yield stress [19].

As mentioned above, due to the slow cooling rate of isothermal holding in the solidification process, β phase decomposes to α phase as a result of diffusional transformation. Evaluation of Al and V contents was performed on the obtained equiaxed α phase by means of energy disperse spectroscopy (EDS). The yellow line that crosses the equiaxed α phase in Fig. 5(a) is the position of line scanning. The results show that aluminium concentration decreases gradually from the centre of the plate to the retained β phase. While β-stabilizing element vanadium was rejected into retained β phase from primary α phase, with concentration increasing from the center of lamellar α to the border (Fig. 5(b)). This tendency is also verified in Ref. [12]. During the isothermal holding treatment, atoms migrate from the terminations of primary α lamellae to the flat interface because of the different curvature rates, resulting in the dissolution of termination tip and the thickening of the adjacent plate. With straight phase dissolved and β phase stabilized elements released, the immigration of α/β phase boundaries leads to the formation of curved lamellae, as shown in Fig. 4(a).

In order to elucidate the formation mechanism of the equiaxed α phase, further research was studied with TEM. As can be seen from Fig. 6(a), a large amount of dislocations are observed in the coarse α plate. As is known, the phase transformation of titanium is characterized by a small volume effect. The volume

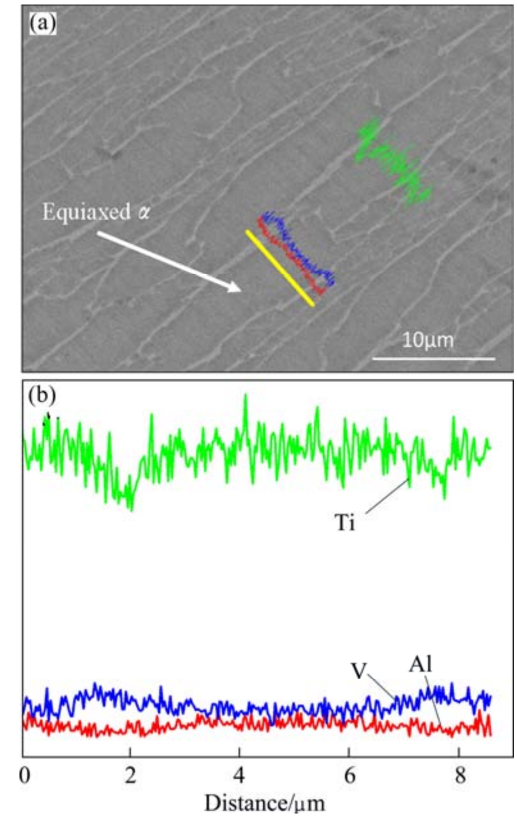

Fig. 5 SEM image (a) and energy disperse spectroscopy (EDS) elemental scanning (b) of equiaxed α phase

change upon β−α is equal to 0.17% [20,21]. The internal stresses that appear in titanium during the phase transformation are estimated to be 30 MPa, which equals approximately 1/10 of the stresses calculated for γ–α transformation in iron [21,22]. Accordingly, it is impossible to refine the grains of titanium alloys by recrystallization. Nevertheless, the phase transformation stress can accumulate during the continuous isothermal holding treatment in the phase transformation area, consequently, the accumulation of phase transformation stress might aggravate the density of dislocation. Meanwhile, as discussed above, the immigration of α/β phase boundaries during the isothermal holding treatment leads to the formation of curved lamellae. It

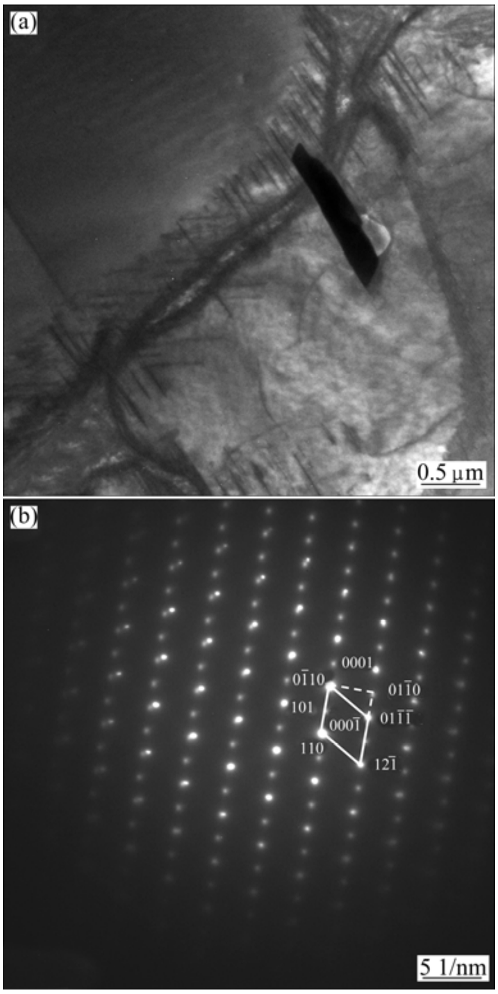

Fig. 6 Bright-field image of equiaxed *α* phase (a) and corresponding electron diffraction pattern of [1 1 1] *β* zone axis (b)

can be speculated that such curved lamellae and dislocations will promote the formation of crack in the coarse primary lamellar *α*. Furthermore, the broken *α* can be globularized by the interface tension of *α* phase to minimize the interface energy. Hence, by coexistence of these effects, the coarsening lamellae *α* phases fracture and turn out to be equiaxed eventually. The formation mechanism can also be used to interpret the discontinuous morphology of grain boundary *α*. At the process of cooling after the isothermal holding proceeding, the retained *β* matrix transforms into fine lamellar *α* because of the growth inhibition by the high concentration of element vanadium, which is repelled from the primary *α* phase in the isothermal holding treatment as discussed above. Eventually, the unique bi-modal microstructure consisting of equiaxed primary *α* and fine lamellar *α* was obtained.

Despite the morphology of *α* phase exhibits equiaxed morphology, the diffraction pattern from the [1 1 1] *β* zone axis also shows reflections consistent with a variants obeying the Burgers relationship as illustrated in Fig. 6(b). The formation mechanism of equiaxed *α* phase can be illustrated in Fig. 7.

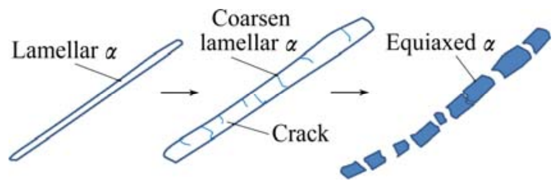

Fig. 7 Schematic diagram illustrating formation mechanism of equiaxed *α* phase

4 Conclusions

1) A unique bi-modal microstructure consisting of coarse, equiaxed primary *α* phase and fine lamellar *α* phase is obtained by isothermal holding treatment at 960 °C for 30 min during solidification.

2) The coarsening lamellar *α* phases break, globularize and present equiaxed morphology by isothermal holding treatment in *α*+*β* phase area. The globularization mechanism of primary *α* is due to atom immigration, high density dislocations in primary *α* lamellae and interface tension of *α* phase.

3) The grain boundaries of *α* phases become discontinuous and have a zig-zagged appearance, which can decrease the negative effect of grain boundary *α*.

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等温处理对 Ti−6Al−4V 合金凝固过程中组织演化的影响

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摘 要：通过控温感应熔炼装置研究 Ti−6Al−4V 在凝固过程中等温处理对组织的影响。结果表明，在 β 相区等温处理，得到由平行片层组成的集束组织；在 960 °C 等温处理，得到一种特殊的钛合金双态组织，由粗化、等轴的初生 α 相和细化的片层组织组成。初生 α 片层倾向于断裂、球化，呈现等轴化形态。凝固过程中在(α+β)两相区进行保温，原子迁移、高位错密度以及 α 相界面张力的共同作用，促使初生 α 片层等轴化。随等温处理结束，未转变的 β 基体进一步转化为细的片层，形成双态组织。与直接冷却得到的片层组织相比，晶界 α 呈不连续形状，片层团尺寸大大降低，整个截面组织均匀。

关键词：钛合金；等温处理；双态组织；晶界；等轴 α 相

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