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Microstructural control of duplex Ti–46.5Al–2Cr–1.5Nb–1V alloys

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Abstract

The microstructural control of duplex Ti–46.5Al–2Cr–1.5Nb–1V (at%) is carried out in two steps: pretreatment and subsequent treatment. The pretreatment of the alloy includes fast cooling from α single-phase field and subsequent annealing at α + γ two phases field. The results show that the sample, which is oil quenched from 1320 °C and then annealed at 1250 °C has the most uniform and finest microstructure, and is suitable for subsequent microstructural control. In subsequent treatment, special heat-treatment processes are employed to obtain the duplex microstructure TiAl alloys with either different grain size at a constant lamellar grain fraction or different lamellar grain fraction at a constant grain size. The investigation of the microstructure transformation in the heat-treatment processes indicates that the grain size is strongly affected by the aging time in pretreatment process. The aging temperature plays a key role in controlling the lamellar grain fraction in subsequent treatment.

Keywords: γ-TiAl based alloy; Duplex; Microstructure; Heat treatment

1. Introduction

Titanium–aluminide based intermetallic compounds have low density, high strength and stiffness up to high temperature, and excellent creep and oxidation resistance. Therefore, they have come to be one of the candidate high-temperature structural materials. But the applications of γ-TiAl alloys are impeded by their poor formability and ambient-temperature ductility [1–4]. It is now well established that mechanical behavior in these alloys is strongly dependent on their microstructures [5–7]. Depending on the composition and heat-treatment procedure, four microstructures of γ-TiAl, i.e., near γ (NG), duplex (DP), near lamellar (NL) and fully lamellar (FL), can be obtained. FL microstructure TiAl alloys display high fracture toughness and high fatigue/creep resistance, but poor ductility, whereas DP microstructure TiAl alloys have relatively higher ductility, but poor fracture toughness. Considerable efforts have been devoted to microstructural design of two-phase TiAl alloys with balanced properties for potential structural applications. In particular, a great deal of research has been focused on various thermomechanical treatments of FL TiAl alloys in order to improve their ambient-temperature properties [8–11]. Some studies have been carried out on the relationship between the mechanical behavior and grain size of duplex TiAl alloys [12–16]. But the results are unclear because grain size and volume fraction of lamellar grain (VFLG) were changed simultaneously in these studies. VFLG is suggested to have a significant influence on the mechanical behavior of DP TiAl alloys [8,17]. It is necessary to control the microstructure and then to systematically study the effects of each microstructure parameter on the mechanical behavior of DP TiAl alloys.

In the course of α → γ transformation, the microstructure changes form a fully α/γ lamellar structure, through a feathery like γ structure to a massively transformed γ structure with an increase in cooling rate from high-temperature α phase field [18,19]. A lot of efforts have been put into the investigations of the formation mechanism and the subsequent aging decomposition of these microstructures [20–22]. It is found that the massive transformation appears to be a suitable path for microstructure refinement.

In this paper, the microstructural evolution of Ti–46.5Al–2.0Cr–1.5Nb–1.0V (at%) that is cooled from α phase field and subsequent aged at α + γ field has been studied in detail. Based on this work, different heat-treatment procedures are carried out to yield two series of duplex microstructure for this alloy. One series has the same grain size but different VFLG. Another

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series has the same VFLG and different grain sizes. Effects of heat-treatment procedure on the microstructure parameters are discussed.

2. Experimental

The alloy with a nominal composition of Ti–46.5Al–2Cr–1.5Nb–1V (at%) was prepared by vacuum induction melting within a water-cooled copper crucible. The ingots were remelted twice to minimize compositional segregation and inhomogeneity. The obtained ingots were hot isostatically pressed (HIP) at 1260 °C under 172 MPa in an argon atmosphere for 4 h to seal casting porosity. Samples with a dimension of 1 cm × 1 cm × 1 cm were cut from the ingots by an electric discharge machine (EDM) and prepared for heat treatment by wrapping in Ta foil and sealing in quartz tubes back-filled with argon to 3000 Pa. All the heat treatments were conducted in a SiC high-temperature furnace. The duplex microstructural control was carried out in two steps: pretreatment and subsequent treatment. In pretreatment, the specimens were firstly solution-treated in the single α region and cooled to room temperature by the following means: air cool (AC), oil quench (OQ) and water quench (WQ). Then they were aged at 1250 °C for different periods of times and air cooled to room temperature. In subsequent treatment, the samples were aged at a temperature between 1180 and 1300 °C for different periods of times and then furnace cooled to room temperature. All samples for microstructural investigation were ground, and electro-polished at 45 V with a solution of 8% perchloric acid + 92% ethanol, and then etched using a solution: HF:HNO_3:H_2O = 1:6:7. The microstructures of the materials were studied by NEOPHOT-2 optical microscopy (OM). A linear intercept method was used to obtain the statistics on the grain size and lamellar-grain fraction.

3. Results and discussion

The microstructure in the as-HIPed ingot consisted of columnar grains, as shown in Fig. 1. These columnar grains contain some lamellar structure grains and a few recrystallized equiaxial gamma grains.

The microstructure of the HIPed TiAl alloy ingots is unchanged even after a long time of aging in the α + γ field. The microstructure (Fig. 2) of the samples that were aged at 1200 °C for 72 h is very similar to Fig. 1. No noticeable difference between them can be observed except the fact that the equiaxed gamma grains grow a little in Fig. 2. It is clear that the initial microstructure plays a critical role in the subsequent microstructure control. It is hard to control the microstructure of HIPed Ti–46.5Al–2.0Cr–1.5Nb–1.0V if only the thermodynamics method is employed. So that stable near fully lamellar (NFL) microstructure of the HIPed ingots must be destroyed firstly by a pretreatment, which contains thermodynamics and kinetics procedure.

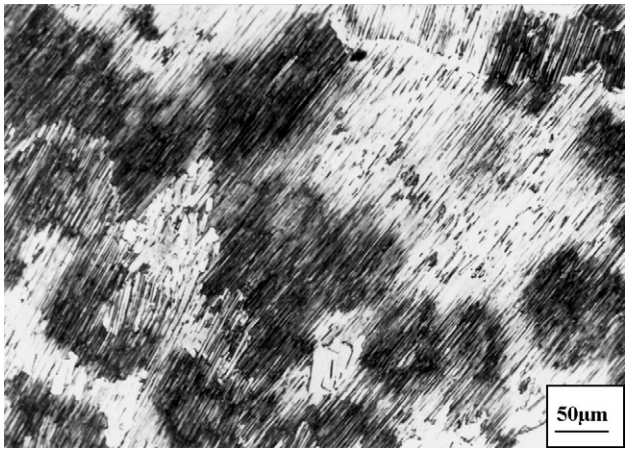

Fig. 1. Metallograph of hot isostatically pressed Ti–46.5Al–2Cr–1.5Nb–1V ingots.

Fig. 2. Metallograph of HIPed Ti–46.5Al–2Cr–1.5Nb–1V which was aged at 1200 °C for 72 h and then air cooled.

3.1. Pretreatment

Solution treating in the single α region and quick cooling will give rise to the sufficient driving force for microstructural transformation of TiAl alloys. The α transus temperature of Ti–46.5Al–2.0Cr–1.5Nb–1.0V that has been determined by phase graph, is 1310 ± 5 °C. So the samples were solution treated at 1320 °C for 10 min and cooled by different cooling rates, i.e., air cool (AC), oil quench (OQ) and water quench (WQ). The microstructures are shown in Fig. 3. It is found that the microstructure of the alloy is transformed to a feather like microstructure (AC), massive microstructure (OQ) and nearly α_2 microstructure (WQ) in turn with increasing cooling rate.

According to the α–γ transformation kinetics [18], the cooling rates show very significant influence on the phase transformation while the samples are cooled from single-phase α region. But for the alloys with different composition, the microstructure is different at the same heat-treatment procedure.

Fig. 3(a) displays a feather like γ (γ_f) microstructure of Ti–46.5Al–2.0Cr–1.5Nb–1.0V after air-cooling from 1320 °C. The feather like appearance, which is made up of large number of fine black needles and fine black massive γ, is the characteristic of this kind of microstructure. The grain boundaries of γ_f are fuzzy and few lamellar microstructures can be found. Actually there are two kinds of feather γ needles. A typical primary feathery (γ_{pf}) grows divergently. It has been shown that most of these needles have a close orientation with a misorientation within 5–8°. The other feathery morphology is termed as secondary feathery (γ_{sf}), which is randomly distributed between the γ_{pf}. No orientation relationship between the γ_{sf} needles has been found.

Fig. 3(b) shows an microstructure of the sample, which was oil quenched from the single α phase field. The picture exhibits

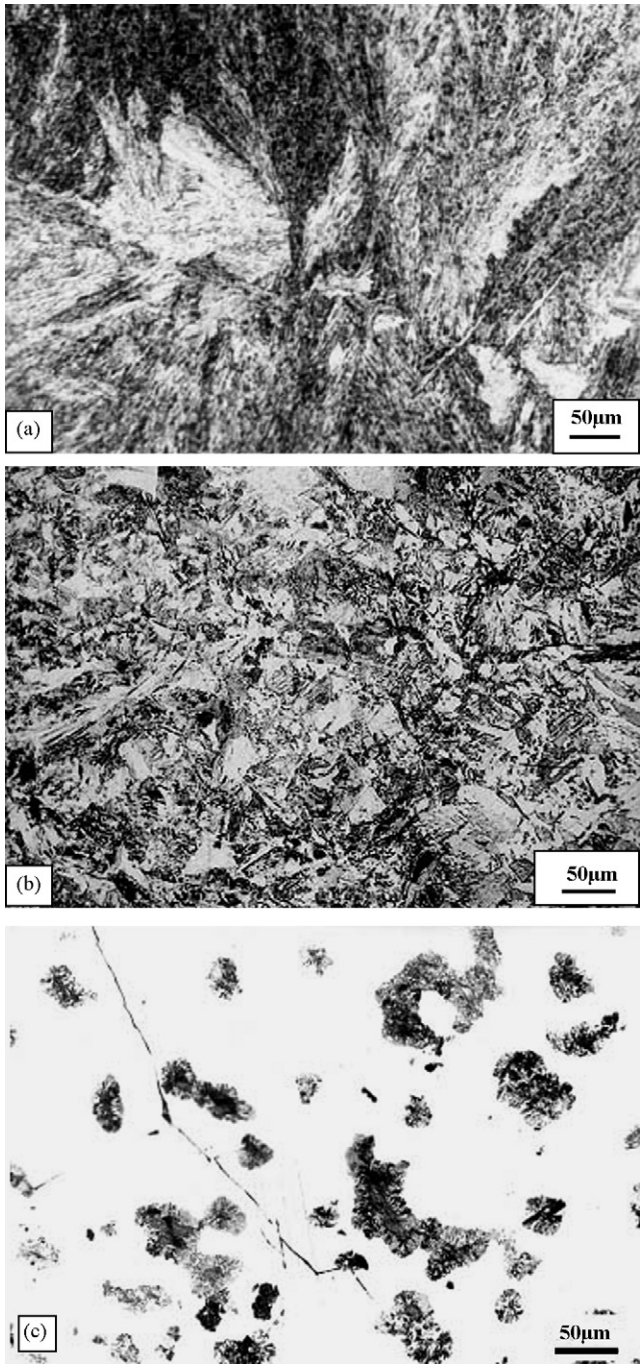

Fig. 3. The microstructures of Ti–46.5Al–2.0Cr–1.5Nb–1.0V cooled from 1320 °C by (a) AC, (b) OQ and (c) WQ respectively.

a fine grain structure with patchy morphology, i.e., so called massively transformed γ structure. It can be seen that the primary NFL microstructure (Fig. 1) has been replaced by massive γ (γ_m).

The microstructure of the alloy water quenched from 1320 °C is shown in Fig. 3(c). Only a few massive γ distribute on the bright α_2 matrix. Massive γ shows cluster appearance and precipitates along the α_2 grain boundary. At the same time microcracks are found. The α_2 matrix comes from the diffusionless transformation of α phase.

The γ_f appears to be a variant of a massive transformation. Mixed morphologies of massive and feathery needle γ may be related to the different stages during the quenching process in which cooling rates may change greatly. It is believed that, at the highest cooling rate during the initial quenching, the driving force is high enough to make the γ phase nucleate and grow rapidly in a massive fashion within the prior larger α grains.

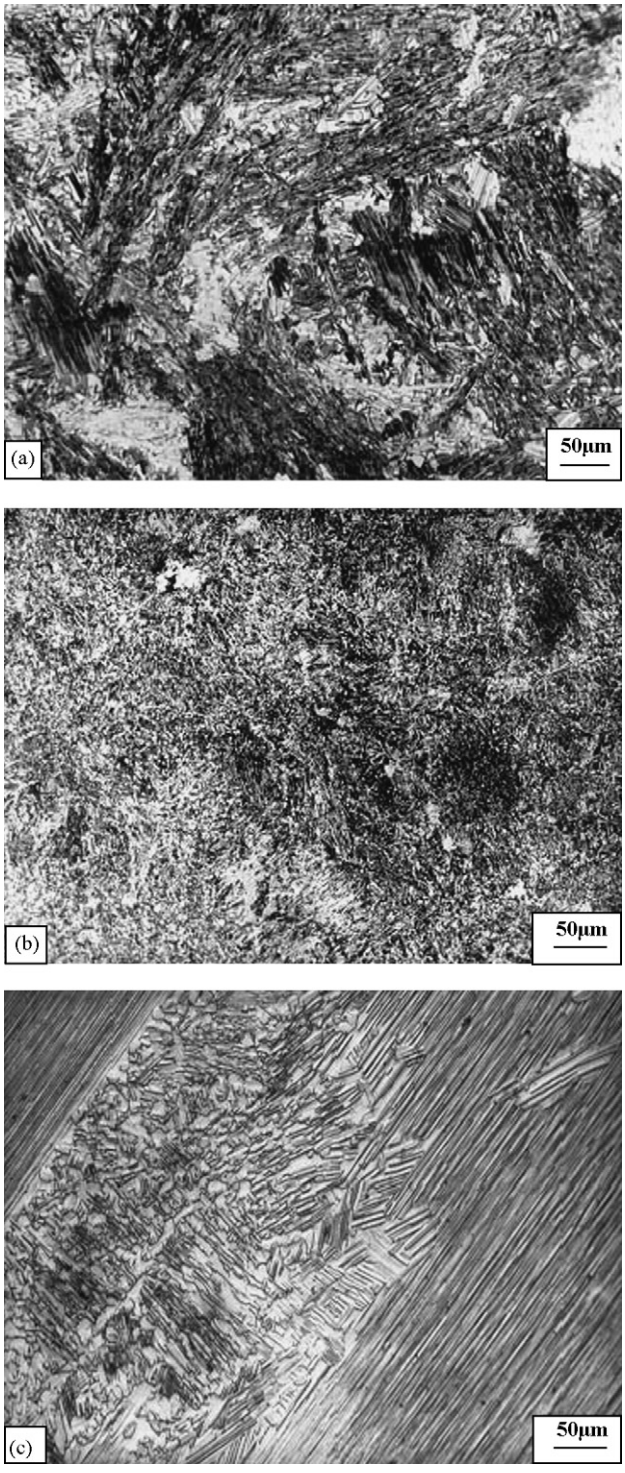

Fig. 4. The microstructures of the samples which were cooled from 1320 °C by (a) AC, (b) OQ and (c) WQ respectively and then aged at 1250 °C for 2 h.

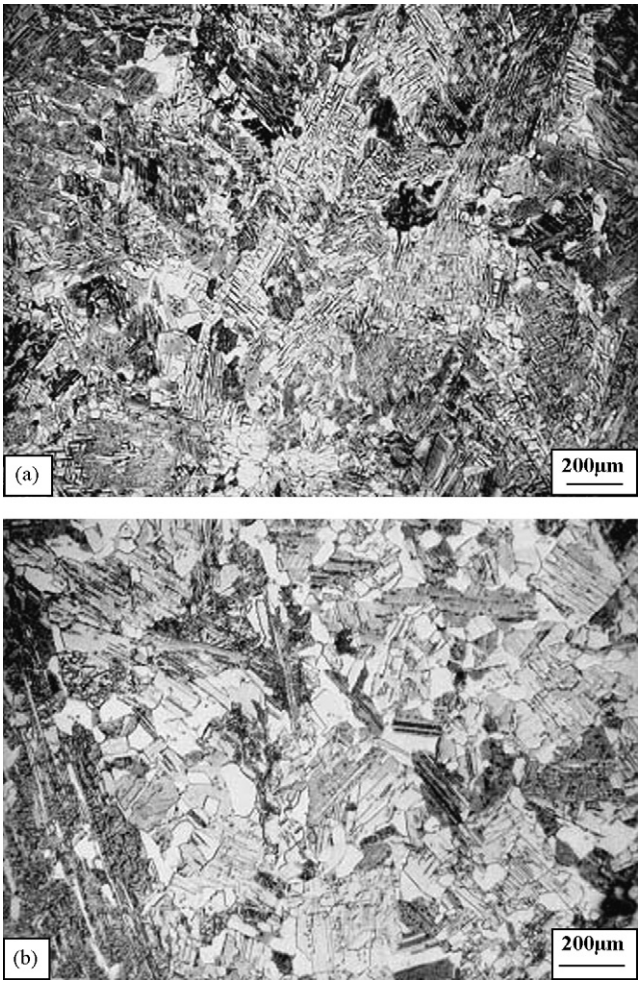

Fig. 5. Coarsened microstructure of oil quenched samples after aging at 1250 °C for (a) 9 h and (b) 60 h.

Later, with decrease of temperature and cooling rate, the driving force for formation of incoherent massive γ decreases and certain orientations may be needed to reduce the nucleation energy. Therefore, at the front of the existing different massive γ domains, some oriented γ domains can grow preferentially and give rise to feathery needle morphology.

After cooled from α single phase field, all samples were aged at 1250 °C for 2 h, and then air cooled to room temperature. It can be found that the microstructures of these samples have changed by decomposing α and γ_m phase. A feather like microstructure (Fig. 4(a)) is kept in air-cooled samples. A fine and uniform microstructure is formed in oil-quenched samples, as shown in Fig. 4(b), and a near fully lamellar microstructure (Fig. 4(c)) is formed in water-quenched samples.

While the air cooled samples were aged at 1250 °C, the α-laths grew along the γ-plate interfaces in γ_{pf} regions, and α particles and plates precipitated in γ_{sf} areas. Two hours late,

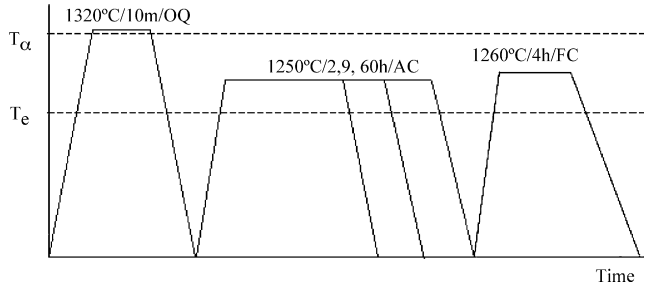

Fig. 6. The schematic chart of heat-treatment process for the control of grain size.

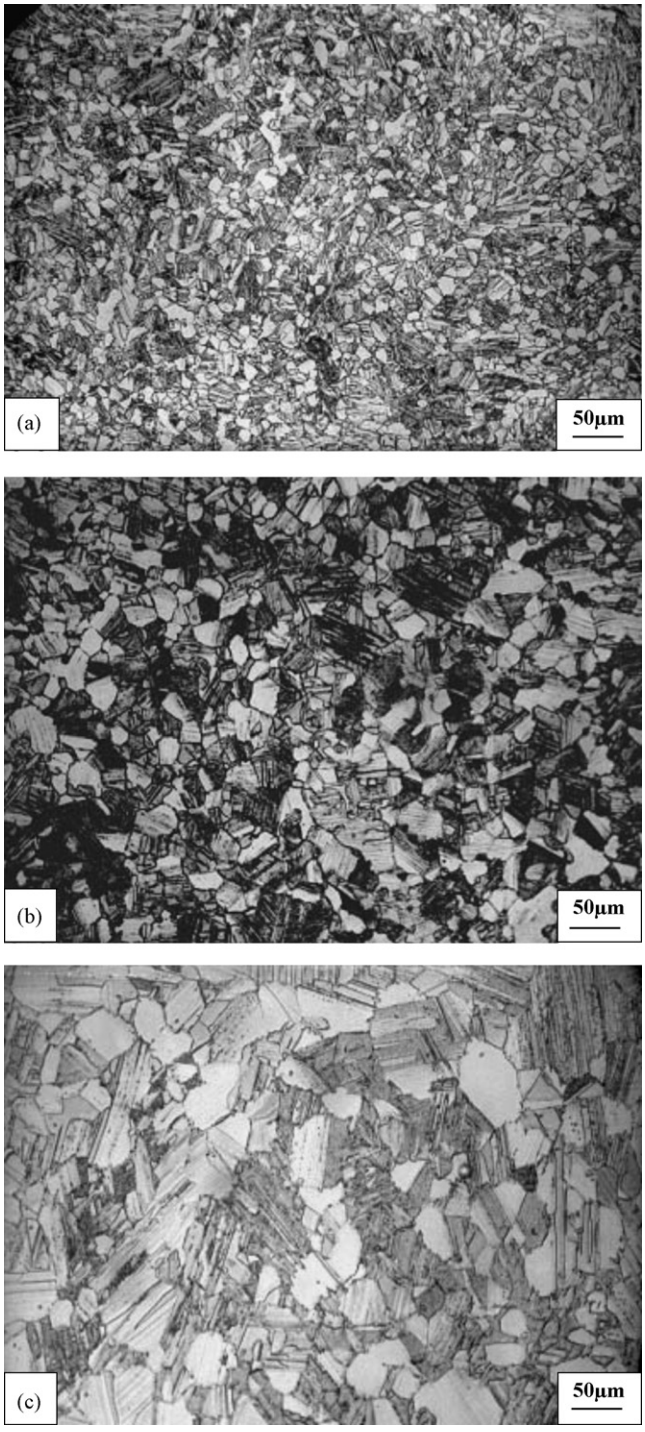

Fig. 7. The microstructure with the same lamellar grain volume fraction and different grain sizes. The grain size are (a) 16 μm, (b) 29 μm and (c) 53 μm respectively.

the lathed and the particles grew up, but the microstructure still had the feather like pattern and the original α grain boundaries still existed. The microstructure are composed of fine equiaxed γ grain, fine lathes, and lamellar microstructure, as shown in Fig. 4(a).

According to previous study [21], many defects have been observed within the non-equilibrium massive γ (γ_m) grains, such as antiphase boundaries (APBs), stacking faults, microtwins and dislocations. While the samples with γ_m microstructure are aged at α + γ two phases field, α-phase is observed to nucleate at these defects, in the shape of particles and dominantly as plate, as Fig. 4(b) shows. The α-precipitates satisfies the orientation relationship $\{0\,0\,0\,1\}_\alpha // \{1\,1\,1\}_\gamma$ and $\langle 1\,1\,\bar{2}\,0\rangle_\alpha // \langle 1\,\bar{1}\,0\rangle_\gamma$. Because the γ matrix has four equivalent $\{1\,1\,1\}$ planes, the α-plates are widely observed to lie in two or three different orientations. As a result, the original massive γ is replaced by a fine uniform microstructure. None obvious grain boundaries are found.

While the water-quenched samples are aged at α + γ two-phase region, the γ plates precipitate from α phase and satisfy the same orientation relationship as that of the α plate's precipitation in γ phase, i.e., $\{1\,1\,1\}_\gamma // \{0\,0\,0\,1\}_\alpha$ and $\langle 1\,\bar{1}\,0\rangle_\gamma // \langle 1\,1\,\bar{2}\,0\rangle_\alpha$. But different from γ matrix, there is only one $\{0\,0\,0\,1\}$ plane in α matrix, which leads to the formation of γ/α lamellar microstructure. When this lamellar microstructure is air cooled down to room temperature after its formation, however, the ordering of α to α_2 has happened, which produces the γ/α_2 lamellar microstructure at room temperature [23]. And a coexisting fine equiaxal γ grains and fine lathes substitute the original massive gamma microstructure. A near fully lamellar microstructure is formed, as Fig. 4(c) shows.

It can be seen from Fig. 4(a) that the grain boundaries will be kept in the air-cooled samples; the large lamellar grain and the crack is easy to form in the water-quenched samples. Comparatively, γ_m conduced by oil quenching is a fine and uniform microstructure, the decomposition of γ_m in the subsequent aging treatment made it even more fine and uniform. It is an ideal intermediate microstructure for the subsequent heat treatment and the massive transformation also appears to be a suitable pretreatment for microstructure controlling.

At 1250 °C, Ti–46.5Al–2.0Cr–1.5Nb–1.0V lies in the middle of α + γ phases field according to phase graph. At this temperature, the interaction of α phase and γ phase makes them grow slowly and keep the balance of two phases. It gives us opportunity to control the grain size of the alloys. Fig. 5(a) and Fig. 5(b) are microstructures of the samples which were oil quenched from 1320 °C and then annealed at 1250 °C for 9 and 60 h, respectively. As the aging time extends, the plates thicken, the particles grow, and the microstructures become coarse.

3.2. The control of grain size

Based on the microstructures that is shown in Fig. 5, microstructures with same volume fraction of lamellar grain

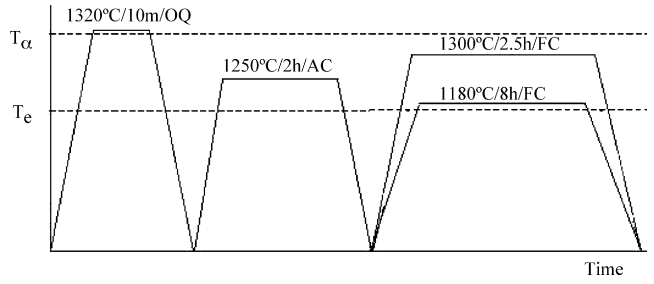

Fig. 8. The schematic chart of heat-treatment process for the control of lamellar grain volume fraction.

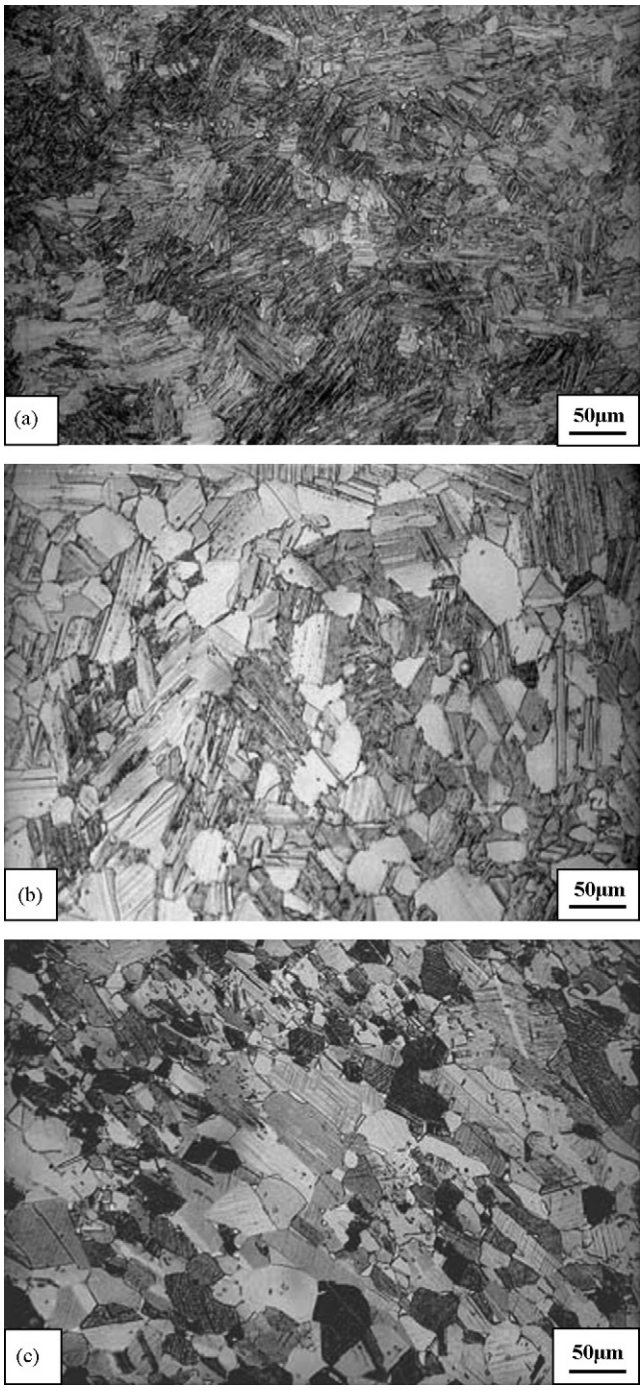

Fig. 9. The microstructure with the same grain size and different VFLG. The VFLG are (a) 6%, (b) 60% and (c) 95% respectively.

(VFLG) and different grain sizes can be obtained by subsequent heat treatment. The heat-treatment processes is shown in Fig. 6 and the microstructures are shown in Fig. 7. Because the aging temperature was same, VFLG of these microstructures were kept at about 60%. At the same time, grain sizes of 16, 29 and 51 μm were obtained by the change of aging time. The difference of grain size was distinguished by the control of a constant lamellar-grain fraction.

3.3. The control of lamellar-grain fractions

According to lever rule, the amount of α phase varies with the change of aging temperature in α + γ phase region, and the VLFG will be decided by the volume fraction of α phase while the samples are furnace cooled from aging temperature to room temperature. So different aging temperatures are employed in this paper to control the volume fraction of lamellar grain in the subsequent heat treatment, as Fig. 8 shows. 1300 °C is selected as aging temperature to obtain nearly lamellar microstructure, and 1180 °C is selected to obtain near γ microstructure. Different from 1250 °C, in these two temperature, one of the two phases turns to be dominant microstructure. The growth rate of grains increases and the grains grow faster at higher annealing temperature. In order to keep the same grain size of these microstructures, different aging times are adopted.

Metallograph are shown in Fig. 9 for three different lamella-grain fraction microstructures. It can be seen that the grain sizes of the three specimens are all about 50 μm, despite the fact that the lamellar-grain fractions are 95%, 63% and 6%, respectively. Through grain size control, the difference of lamellar-grain fraction can be obtained at the same time. Figs. 7(c) and 9(b) are the same micrographs because this structure is part of both series.

By now, the two microstructure parameters of Ti–46.5Al–2.0Cr–1.5Nb–1.0V DP microstructure have been distinguished from each other. This gives the opportunity to indpendently study the effect of each microstructure parameter on the mechanical behavior of the alloys.

4. Conclusions

Microstructure control is realized by the combination of thermodynamics and kinetics. It is hard to control the microstructure of as-HIPed TiAl alloys ingots if only the thermodynamics method is employed.

When the Ti–46.5Al–2.0Cr–1.5Nb–1.0V alloys are cooled at different cooling rates (AC, OQ and WQ) from 1320 °C, the microstructures of alloy vary from a feather like microstructure through a massive microstructure and finally to a nearly α_2 microstructure with increasing cooling rate.

While the alloys are oil quenched from 1320 °C and then annealed at 1250 °C, a fine uniform microstructure is formed. The interaction of α phase and γ phase makes them grow at same rate. With the increasing aging time, the microstructure turns to be coarser. These intermediate microstructures give us the opportunity for microstructural control.

The volume fractions of lamellar grain are controlled by different aging temperatures in the subsequent heat treatment. With the increase of annealing temperature, VFLG increases. At the same time, the grain sizes of different duplex microstructure are controlled by different annealing time.

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