Grain refinement of a TiAl alloy by heat treatment through near gamma transformation

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Casting of TiAl alloys is receiving more and more research because of its relatively low cost. One problem with this technology is that the coarse microstructure developed during solidification is harmful to material properties. Thus it is essential to seek an approach to refining the cast microstructure and this approach may also be applicable to cast components. In this study, a novel heat treatment route is proposed to obtain a fine fully lamellar structure from a cast TiAl alloy with a grain size of 1000 μ m. This route consists of three steps, namely pretreatment to have a feathery γ structure, annealing in the $\alpha + \gamma$ region to have a fine near gamma structure and solution treatment to develop a fully lamellar structure with a grain size of ~30 μ m. © 2001 Kluwer Academic Publishers

1. Introduction

A great deal of research has been conducted on TiAl based alloys as they are potential materials for high temperature applications. This is related to their promising properties such as low density, high specific modulus and strength, high melting temperature and acceptable oxidation resistance [1–3]. From the viewpoint of application, casting has received considerable research because it is a simpler processing method and thus has a lower cost, compared with other approaches such as powder metallurgy. But how to transform the coarse cast microstructure to a fine fully lamellar (FFL) structure which is known to have an excellent combination of strength and ductility [4], is a key issue.

Several different methods have been proposed to produce a FFL structure merely by heat treatment [5–8]. They are concerned with different phase transformations including massive transformation [5, 6], discontinuous coarsening (DC) [7] and near gamma transformation [8]. All these transformations result in refinement. By the third method, the coarse microstructure is converted to a near gamma (NG) structure first and then heated to the single α field.

An NG structure may be obtained by thermomechanical processing or by long-term annealing at a temperature slightly higher than the eutectoid temperature of $\alpha \rightarrow \alpha_2 + \gamma$ (T_e). By the former method, an equiaxed structure may be obtained by double forging plus heat treatment (see [9] for an example on a TiAl based alloy with a composition of Ti-47.5Al-1.8Nb-1.9Cr-1.0Mo).

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Because of the involvement of mechanical processes, this technique is complex and thus costy in a commercial sense. For the latter method to be effective, the alloy has to be annealed for a very long time (longer than a few days), depending on the stability of the lamellar structure and the composition of the alloy. For instance, Zhang *et al.* [10] produced a NG structure by cyclic heat treatment around T_e for 168 hrs and Pu *et al.* [11] annealed the alloy of Ti-46.6Al-2.3V-0.9Cr for more than 50 hrs to complete the transformation. In this study we demonstrate an alternative method for obtaining a fine NG structure and refining the cast microstructure to a FFL one in a relatively short period.

2. Experimental

The alloy used in this study had a composition of Ti-46Al-2Cr-2Nb (at.%). It was prepared by vacuum induction melting in a water-cooled copper crucible. To minimize compositional segregation and inhomogeneity, electromagnetic stirring was imposed and each alloy was melted at least twice. The ingots were hot isostatically pressed (HIP) at 1250° C/175 MPa for 4 hours to eliminate casting flaws. Specimens with a dimension of $10 \text{ mm} \times 10 \text{ mm} \times 10 \text{ mm}$ were cut from the ingot using electrical discharge machine (EDM). Heat treatments were conducted in a SiC high temperature furnace with an accuracy within $\pm 2^{\circ}$ C. The heat treatment route is shown in Fig. 1. A sample was pretreated in the single α phase region and quenched in oil (OQ) (step A).



Figure 1 Three-step heat treatment route. T_{α} : α transus temperature, T_e : eutectoid temperature, OQ: quenching in oil and AC: cooling in air.

It was then annealed at a temperature slightly above T_e and OQ (step B). Finally the sample was heated to the single α region again and cooled in air (AC) (step C).

After heat treatment, the sample was grounded, polished and etched with Kroll's reagent. The microstructures were studied using optical and scanning electron microscopes. The phase distribution was investigated in the backscattered electron (BSE) mode and composition analyzed using an energy-dispersive spectrometer (EDS).

3. Results

Fig. 2 shows the coarse columnar microstructure with a width of ~1 mm and a length about 5–10 mm. These columnar grains contained alternate γ and α_2 plates, exhibiting a lamellar structure. The lamellar orientation is nearly perpendicular to the extension of columnar grains. This is due to the fact that the preferred growth direction of α phase is (0001) and the habit plane for the precipitation of γ plates is (0001). At the boundaries of columnar grains there exist some equiaxed γ grains which probably formed during HIPing.

Fig. 3 shows the microstructure after step A. After this treatment the original columnar grains were replaced by feathery γ (γ_f). Every γ_f shows a divergent



Figure 2 Columnar microstructure of the cast TiAl alloy.



Figure 3 The feathery structure formed in heat treatment A.

and bunched configuration. There is no clear boundary between individual γ_f variants.

Fig. 4a shows the microstructure after step B. As can be seen, the microstructure is composed of mainly



Figure 4 The fine near gamma microstructure after annealing the γ_f in the $\alpha + \gamma$ region. (a) Optical image, (b) BSE image. Single arrow: γ phase, double arrows: α_2 phase.



Figure 5 Fine fully lamellar structure formed after solution-treatment of the fine near gamma structure.

fine equiaxed grains with a grain size of $\sim 10 \ \mu$ m, and among the equiaxed grains there exists a platelike phase. This microstructure is further shown by BSE imaging in Fig. 4b. The compositions of equiaxed grains and black plates were determined by EDS. It was found that the concentration of Ti in the equiaxed grains is close to that of Al (Ti: 49.25, Al: 46.29), but the dark plates and grains are enriched with Ti (Ti: 57.19, Al: 37.78).

The microstructure after step C is illustrated in Fig. 5, which appears fully lamellar. Experiments showed that the grain size strongly depended upon the holding temperature and time used. As expected, a higher temperature and longer time led to larger sizes. By controlling these two variables, an average grain size of 30 μ m could be obtained (Fig. 5). This size is larger than that of the NG structure (Fig. 4a), indicating slight grain growth in the final heat treatment.

4. Discussion

The heat treatment following the route shown in Fig. 1 is for obtaining a FFL microstructure. The purpose of the first step is to destroy the cast coarse columnar microstructure and obtain the $\gamma_{\rm f}$. Previous TEM studies showed that γ_f structure is comprised mainly of misaligned γ laths, and between them some elongated or equiaxed γ grains, and few retained α_2 phase [12, 13]. When the cast microstructure was heated into the single α region, recrystallization took place and the columnar microstructure turned to equiaxed α grains. In the course of quenching, γ phase nucleated at the defects in the α matrix or grain boundaries and grew quickly. When the phase γ reached a critical size, the habit plane for the precipitation of this phase would shift lightly by a small degree probably due to the action of phase transformation stress. This may be the reason for that the γ phase showed a divergent feathery configuration. Nevertheless the detailed mechanism of formation of $\gamma_{\rm f}$ needs further studies.

Based on the Ti-Al phase diagram and heat treatment [14, 15], TiAl (γ) is the dominant phase in equilibrium at 1200°C. So during the annealing at 1200°C in the $\alpha + \gamma$ phase field (step B), the $\gamma_{\rm f}$ made up of misaligned γ laths which formed through unequilibrium

precipitation was unstable and thus underwent transformation to a more stable equiaxed γ structure. According to BSE and EDS analyses, phase determination is straightforward; the equiaxed matrix (grey and black grains in Fig. 4a, bright grains in Fig. 4b) is γ phase, and the other phase in the matrix (bright plates and grains in Fig. 4a, but dark ones in Fig. 4b) is α_2 . It is clear that the microstructure consists of γ phase with some α_2 phase.

If the as-cast microstructure was directly annealed in the $\alpha + \gamma$ region, the NG microstructure (shown in Fig. 4a) could not be obtained in a short time because the cast lamellar structure is very stable, and its degeneration is very time-consuming. Previous studies [16–20] have shown that the degeneration of lamellar structure in which discontinuous coarsening (DC) and continuous coarsening (CC) are involved is a slow process and takes a long time to complete. Additionally, after degeneration the grain size is still large (usually larger than 100 μ m). However, such degeneration by DC and CC was avoided in this study. Instead, the lamellar structure was changed to γ_f by pretreatment first, and then $\gamma_{\rm f}$ to an FNG structure (~10 μ m) by annealing. Consequently, the whole process is shorter and final structure is finer.

To transform the FNG structure to an FFL one, it is necessary to perform heat treatment in the single α region. During heating, the α phase would precipitate within the γ grain or at the grain boundary first [21]. During the subsequent holding, the whole γ grain could transform into an α grain, and grain growth might occur. During cooling a lamellar structure formed within every α grain as a result of precipitation of γ plates.

5. Conclusions

This study shows that a fine fully lamellar (FFL) structure (~30 μ m) could be obtained by heat treating a coarse cast lamellar structure of TiAl alloys. No mechanical processing such as forging or extrusion was required. The heat treatment includes three steps: (1) pretreatment to obtain a feathery γ structure (γ_f), (2) annealing to change the γ_f to a fine near gamma (FNG) structure, and (3) solution treatment to convert the FNG structure to a FFL one. The present approach for developing NG structure is different from previous one using direct annealing of the cast structure, with an advantage of being able to have a finer NG and thus a finer lamellar structure.

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