A NEW HEAT TREATMENT PROCESSING FOR TiAl BASED ALLOY①

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ABSTRACT Owing to the limitation of the normal thermomechanical treatment on TiAl based alloy, a double-temperature heat treatment technology for hot forged TiAl based alloy was suggested. The effect of the new technology on microstructure and tensile properties of the alloy at ambient temperature was also studied. By TEM analysis, the mechanism that the double-temperature heat treatment improves the homogeneity of the microstructure in TiAl based alloy was discussed.

Key words TiAl based alloy microstructure mechanical property heat treatment

1 INTRODUCTION

Having the merits of low density and excellent elevated temperature properties, TiAl based alloy is regarded as an ideal high temperature structure material which has the promising application in aerospace. However, owing to poor thermal deformability[1], brittleness at ambient temperature[2] and insufficient oxidation resistance above 850 ℃[3] etc, the practical application of this alloy is hindered. Its brittleness at ambient temperature is more severe . Studies[4-6] showed that, the ambient temperature mechanical properties of TiAl based alloy were significantly influenced by its microstructure, and the specimen with fine, homogeneous duplex structure demonstrated excellent ambient temperature ductility[7-9]. However, the coarse cylindrical α_2/γ lamellar colonies in the as-cast microstructure of TiAl based alloy can be disintegrated only through hot pressing deformation. The lamellar colonies of TiAl based alloy exhibit anisotropy in mechanical properties[10] and in deformation substructure[11-13], and the zigzag boundaries[14] restrict the rotation of grains, therefore, the deformation substructures of all the α_2/γ lamellar colonies exhibit monotonity during the thermal deformation process. As a result, several coarse lamellae colonies still remain perfectly lamellae after thermal deformation process[15]. Moreover, because of high rheological stress in TiAl based alloy and high friction between the specimen and punch, the distribution of internal stress is heterogeneous in the specimen. Stress may be very low in some area in which thermal deformation characteristics experienced by material are not apparent, and coarse lamellae colonies still exist[14]. Owing to the complexity of the thermal deformation process of TiAl based alloy, several indestructed coarse lamellae colonies always remain in the deformed specimen[17]. Those lamellar colonies exhibit microstructural thermal-stability when annealed at high temperature and can not be fined during subsequent annealing in two-phase field. Therefore, in order to acquire fine, homogeneous thermal-deformation structure for further study, specimens should be selectively cut from the ingot[18]. Moreover, due to low thermoplasticity of TiAl based alloy, a large amount of deep cracks come out on the periphery and upper and lower surface of the ingot, and homogeneous,

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thermal deformation-microstructure only partially exist. Therefore the quality of the ingot is very poor, the selected specimens always demonstrate poor mechanical properties because of containing some coarse lamellar colonies.

This article concentrates the investigation on the effects of double-temperature heat treatment process on the microstructure and ambient temperature mechanical properties of randomly selected specimens from TiAl based alloy ingot.

2 EXPERIMENTAL

The alloy with nominal composition (in weight fraction) of Ti-33Al-3Cr-0.5Mo was prepared with consumable arc melting technique in argon atmosphere. In order to reduce composition inhomogeneity, the alloy ingot was remelted. After homogeneously annealing at 1 050 ℃ for 48 h, the ingot was spark-eroded into dia. 50 mm × 100 mm cylindrical specimen, which was then HIP-ed (1 250 ℃, 170 MPa, 4 h). Plastic deformation through hot press treatment was conducted under isothermally forging machine, process parameters were: at 1 040 ℃, 3×10^{-4} s^{-1}, 82%. After the crack parts on the ingot periphery were cut off, specimens were selected randomly from the remain parts for heat treatment and mechanical property tests. Ambient temperature tensile test was conducted on CSS-112 type electronic versatile test machine. The effective dimensions of cylindrical tensile specimen are dia. 3 mm × 18 mm. Fractograph analysis was conducted under scanning electron microscope. The metallographic specimens after heat treatment were prepared in a standard fashion and etched with the Kroll′s solution. Microstructural-metallographic analysis were conducted under Leica-Quantimet 520 type optical microscope and morphology analysis instrument, and scanning electron microscope. The foils for transmission electron microscope were prepared by twin-jet technique using a solution consisting of 70 ml alcohol, 120 ml methanol, 100 ml butane-1-01 and 80 ml perchloric, at a voltage of ~45 V, a current of 7 to 10 mA, and temperature of − 40 ℃. Observation of the foils was conducted in a CM-12 transmission electron microscope operated at 120 kV.

3 RESULTS

Table 1 describes the mechanical properties of the alloy at ambient temperature which was normally heat treated according to the route shown in Fig. 1. The experiment data show that, Ti-33Al-3Cr-0.5Mo alloy exhibits low ambient temperature mechanical properties, especially poor ambient temperature ductility.

Table 1 The mechanical properties at ambient temperature of Ti-33Al-3Cr-0.5Mo alloy after normal heat treatment

Sample No.	Heat-treatment processing	Processing type	$\sigma_{0.2}$ /MPa	σ_b /MPA	δ /%
1	900 ℃, 24 h, A.C.	I	390	428	1.3
2	1 000 ℃, 24 h, A.C.	I	453	480	1.2
3	1 180 ℃, 4 h $\xrightarrow{F.C.}$ 950 ℃, 6 h, A.C.	II	460	519	1.2
4	1 250 ℃, 4 h $\xrightarrow{F.C.}$ 950 ℃, 6 h, A.C.	II	510	537	1.8
5	1 280 ℃, 4 h $\xrightarrow{F.C.}$ 950 ℃, 6 h, A.C.	II	521	570	1.5
6	1 380 ℃, 2 h, A.C. + 950 ℃, 6 h, A.C.	III	621	746	1.3

A.C. —air cooling; F.C. — Furnace cooling

Fig. 1 Scheme of normal heat-treatment processing for TiAl based alloy sample IF′ed

Fig. 2 illustrates the microstructure of specimens normally heat-treated. Indestructed coarse lamellae colonies still remain when heated in two-phase field as shown in Fig. 2(a). Chemical composition EDAX analysis of the microstructure in selected area is shown in Fig. 2(b), no apparent difference between the coarse lamellae colony and the lamellar colony in fine duplex microstructure thereabout was found. TEM analysis shows that, coarse lamellae colony still consists of α_2 lathes and γ lathes, strict orientational relation exists between adjacent lathes, as shown in Fig. 3. Fig. 4 describes the SEM fractograph of tensile specimen after annealed in two-phase field. The fracture of coarse colony can be found in the fractograph, and the large amount of secondary cracks also can be detected around these lamellar colonies boundary.

When randomly selecting specimens from TiAl based alloy ingot, it exhibits inhomogeneous microstructure and poor mechanical properties at ambient temperature. For this reason, a new technique —double-temperature heat treatment is presented, as shown in Fig. 5. Table 2 illustrates the ambient temperature tensile property of Ti-33Al-3Cr-0.5Mo alloy treated by this new technique.

Compared with the experiment data in Table 1, the ambient temperature mechanical properties of TiAl based alloy ingot after double-

Fig. 2 Optical micrographes of Ti-33Al-3Cr-0.5Mo alloy normally heat treatd

(a) —900 ℃; (b) —1 250 ℃ $\xrightarrow{\text{F. C.}}$ 950 ℃, 6 h

Fig. 4 Fractograph by SEM analysis for Ti-33Al-3Cr-0.5Mo alloy ingot heat-treated at

1 250 ℃, 4 h $\xrightarrow{\text{F. C.}}$ 950 ℃, 6 h

Fig. 5 Scheme of double-temperature heat-treatment processing

Table 2 The effects of double-temperature heat-treatment types on mechanical properties of Ti-33Al-3Cr-0.5Mo

Sample No.	Heat-treatment procesing	Heat-treatment type	$\sigma_{0.2}$ /MPa	σ_b /MPA	δ/%
1	1310 ℃, 4h, A.C + 1250 ℃, 4h, A.C + 950 ℃, 6h, A.C.	Ⅰ	521	583	2.1
2	1310 ℃, 4h, $\xrightarrow{F.C.}$ 1250 ℃, 4h, A.C. + 950 ℃, 4h, A.C.	Ⅱ	590	673	2.4
3	1280 ℃, 4h, $\xrightarrow{F.C.}$ 1250 ℃, 4h, A.C. + 950 ℃, 6h, A.C.	Ⅲ	543	657	2.8

temperature heat treatment are improved. Fig. 6 shows two optical metallographs for the microstructure of TiAl based alloy ingot after various double-temperature heat treatment. After Ⅰ-type double-temperature heat treatment, microstructure of mixed grains is obtained —fine equaxial γ grains or fine lamellae colonies distributing in coarse equaxial lamellae colonies, as shown in Fig. 6(a); while after Ⅱ-type double-temperature heat-treatment, fine, homogeneous duplex microstructure is obtained, as shown in Fig. 6(b). In this article, the process parameters of Ⅱ-type double temperature are further studied, and their experiment results are shown in Table 3, from which it can be seen that variation of the heating time at the first annealing temperature in double-temperature heat treatment influences the ambient temperature tensile property, i. e. diminishing the first annealing time will improve the ambient temperature mechanical properties, especially the ductility of TiAl based alloy. In order to investigate how the Ⅱ-type double-temperature heat treatment influences the ambient temperature mechanical properties of the alloy, their microstructures are analyzed. The results are as follows: when heated at 1310 ℃ for 4, 3, 2, 1 and 0.5h, the mean grain diameters of duplex structure in the finally heat-treated specimens are respectively 48, 35, 28, 25 and 18 μm, part of the optical metallographs are shown in Fig. 7.

Fig. 6 Micrographes of Ti-33Al-3Cr-0.5Mo alloy heat-treated at double-temperature

(a) —1310 ℃, 4h, A.C. + 1250 ℃, 4h;

(b) —1310 ℃, 4h, $\xrightarrow{F.C.}$ 1250 ℃, 4h, A.C. + 950 ℃, 6h, A.C.

4 DISCUSSION

One largely hot forging plasticity deformation can not disintegrate all the coarse lamellae colonies in TiAl based alloy. When annealing in two phase field, owing to in-situ order → disorder phase transformation of α_2, lamellar colonies with perfect α_2/γ lamellae structure can trans-

Table 3 The effects of Ⅱ type heat-treatment processing on mechanical properties of Ti-33Al-3Cr-0.5Mo alloy at ambient temperature

Sample No.	Heat treatment processing	$\sigma_{0.2}$ /MPa	σ_b /MPA	δ /%
1	1310 ℃, 3h $\xrightarrow{F.C.}$ 1250 ℃, 4h $\xrightarrow{F.C.}$ 950 ℃, 6h, A.C.	432	577	2.6
2	1310 ℃, 2h $\xrightarrow{F.C.}$ 1250 ℃, 4h $\xrightarrow{F.C.}$ 950 ℃, 6h, A.C.	403	507	2.8
3	1310 ℃, 1h $\xrightarrow{F.C.}$ 1250 ℃, 4h $\xrightarrow{F.C.}$ 950 ℃, 6h, A.C.	426	603	3.3
4	1310 ℃, 0.5h $\xrightarrow{F.C.}$ 1250 ℃, 4h $\xrightarrow{F.C.}$ 950 ℃, 6h, A.C.	429	572	3.2

form to α/γ lamellar structure at elevated temperature, which inherit the original α_2/γ or γ/γ twin interface relation. Due to strict orientation relation on the lathes boundaries, it is very difficult for nucleation. Additionally, the strict orientation relation restricts the boundary migration when lathes grow widely and equiaxially (as shown in Fig. 8). Therefore, the coarse α_2/γ lamellae colonies remaining after thermal deformation exhibit high microstructure thermal-stability during subsequent annealing in two-phase field. Poor mechanical properties at ambient temperature are mainly due to coarse colonies in specimens, as shown in Fig. 2 and Table 1. However, the ambient temperature mechanical properties of TiAl based alloy are greatly improved through appropriate double-temperature heat treatment, as illustrated in Table 2 and Table 3, owing to having obtained a fine, homogeneous duplex microstructure (Fig. 6 and Fig. 7). Fig. 9 describes how II-type double-temperature heat treatment can refine the microstructure of TiAl based alloy specimen hot-forged. After thermoplastic deformation, a large amount alloys of lattic defects are generated in TiAl based alloy, keeping high internal energy store. When heating in monophase field, a large number of α nucleuses come into forming. Without the restriction on new crystal boundary migration by lathes boundaries when heating in two-phase field, α phase nucleuses grow equaxially. Because of high nucleation rate, the equiaxial

Fig. 7 Micrographes of TiAl based alloy after double-temperature heat-treatment

(a) —1310 ℃, 3 h $\xrightarrow{\text{F. C.}}$ 1250 ℃, 4 h, A. C.

(b) —1310 ℃, 0.5 h $\xrightarrow{\text{F. C.}}$ 1250 ℃, 4 h, A. C.

ig. 8 TEM micrograph of Ti-33Al-3Cr-0.5Mo alloy ingot IF′ ed after annealing of heated at $\alpha+\gamma$ two phase field

Fig. 9 Scheme of microstructure transformation after annealing at type II processing double-temperature

grains are not so large, as shown in Fig. 9(a). When cooling in furnace and retaining at constant temperature in two phase field, γ phase will precipitate. The formation of γ nucleuses is homogeneous, and the growth of them is equiaxial, as shown in Fig. 9(b). At this time, the microstructure is characterized by dispersion of finely equiaxial γ phase grains in coarsely equiaxial α phase grains. Subsequently, when cooling from $\alpha+\gamma$ two phase field to room temperature, the transformation of $\alpha \rightarrow \alpha_2+\gamma(\alpha_2/\gamma)$ occurs. Main steps are $\alpha \rightarrow \alpha+\alpha^{SF} \rightarrow \gamma_2+\alpha \rightarrow \gamma+\alpha \rightarrow \gamma+\alpha_P(\alpha_2/\gamma)$, i.e. during $\alpha \rightarrow \alpha_2+\gamma$ transformation process, γ phase acts as an antecede phase, then $\alpha \rightarrow \alpha_2$ ordered transformation occurs at T_e, thus, α_2/γ lamellar colony is obtained. In order to decrease nucleation energy, the γ lathes formed in α_2/γ lamellar colony should adhere to initially formed equiaxial γ grains, i.e. nucleation locations are provided on the surface of the initially formed equiaxial γ grains. According to nucleation theory, certain orientation relation should be acquired between γ lathes and boundaries of initial γ grains, herein the twin relation is {111}<110>, which has been proved by expriments, as shown in Fig. 10. In α_2/γ lamellar colony, lath B of γ phase adheres to an equiaxial grain A of γ Phase, nucleating and growing up. As also shown in Fig. 10, the selected area electron diffraction pattern of B/A interface is $[011]_A \parallel [0\overline{1}\overline{1}]_B$, twin face is (111). In an original α grain there exist many γ equiaxial grains whose surfaces are composed of many crystal faces with various orientation. Hence, many α/γ colonies with various lamellae orientation come into forming in an original α grain. When cooling to room temperature, ordered transformation of α phase occurs. As a result, through II-type double-temperature heat treatment, the fine, homogeneous duplex microstructure is obtained, as shown in Fig. 9(c). The microstructure analyzed by SEM is also shown in Fig. 11 that α_2/γ colonies nucleate and grow up around equiaxially γ phase grains. When diminishing heating time at first temperature, compositional heterogeneity in α grain gets more and more, and the nucleation of γ grains is easily enhanced when cooling from α monophase field to $\alpha+\gamma$ two-phase field and retaining at a contant temperature, as a result, the number of γ grains increases. Moreover, due to shorter heating time, the original α phase grain is fine, and favorable for acquiring fine duplex microstructure. However, through I-type double-temperature heat treatment, α_2/γ lamellar colonies are firstly obtained, as shown in Fig. 12(a). When reheating to $\alpha+\gamma$ two-phase field, $\alpha_2 \rightarrow \alpha$ in-situ phase transformation occurs and α/γ lamellar colonies thus be generated. Moreover, the renucleation and growth of both α grains and γ grains happen in some local area, the microstructure is shown in Fig. 12(b).

Fig. 11 SEM BS micrograph of TiAl based alloy sample double-temperature heat-treated

When cooling to room temperature, $\alpha \rightarrow \alpha_2$ in-situ ordered transformation occurs in α/γ lamellar colonies, hence α_2/γ lamellar colonies are obtained, and finally a microstructure of mixed grains is generated, as shown in Fig. 12(c).

Fig. 12 Scheme of microstructure transformation after annealing at double-temperature in type Ⅰ processing

5 CONCLUSIONS

(1) After normal heat treatment, the specimen, which is randomly selected from TiAl based alloy ingot hot-forged, exhibits inhomogeneous microstructure. In the microstructure, coarse α_2/γ lamellae colonies can be easily found, which has been proved to be inherited from as-cast microstructure. The alloy specimens exhibit poor mechanical properties at the ambient temperature.

(2) Through appropriate double-temperature heat treatment, the fine and homogeneous duplex microstructure can be acquired from TiAl based alloy ingot. The ambient temperature mechanical properties of the specimens are significantly improved.

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