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Pilot processing and microstructure control of high Nb containing TiAl alloy

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Abstract

The investigation of high Nb containing TiAl alloy so far was performed using small heats. In this study, the microstructure control of high Nb containing TiAl alloy was investigated using relatively large forged pancake. The canned forging pancake was successfully gained. The effect of processing and heat treatment on the microstructure was studied using X-ray diffraction, optical microscopy and scanning electron microscopy. Results show that the pancake with a duplex microstructure consists of lamellar colonies, equiaxed γ phase with the grain size of about 20–30 μm and the β(B2) phase at grain boundaries. Energy dispersive spectroscopy analysis shows that the β(B2) phase is enriched in Nb and W. The β(B2) phase was eliminated by heat treatment in $\alpha+\gamma$ phase region. It is confirmed that the existence of β(B2) phase is due to the segregation of Nb and W. The homogenous near gamma microstructure with the grain size of about 20 μm, and homogeneous refined fully lamellar microstructure with a colony size of about 20 μm was obtained by the special heat treatments.

Keywords: A. Titanium aluminides, based on TiAl; C. Heat treatment; C. Isothermal forging; D. Microstructure

1. Introduction

TiAl alloys have attracted considerable attention recently owing to their attractive properties such as low density, excellent high temperature strength and good oxidation resistance [1]. However, the practical application of this material is hindered due to its poor hot deformability, and brittleness at ambient temperature [2]. Hot working operation is an efficient way to develop a fine, uniform microstructure suitable for subsequent processing or final heat treatment for the desired microstructure. Therefore, great effort has been devoted to establishing hot workability indices for a variety of conventional TiAl alloys [3].

High Nb containing TiAl based alloy developed by Chen is the first example for the development of high performance TiAl alloy exhibiting very high strength at both room temperature and high temperatures [4–6]. The creep strength retention is 60–100 °C higher than that of conventional TiAl alloy [7]. Effects of microstructure on properties for the high Nb containing TiAl alloy are similar to that for the conventional TiAl. However, investigation of high Nb containing TiAl alloy so far was performed using small samples. The hot workability and microstructure control of as-forged pancakes for an industrial scale of the alloys are poorly understood. In this study, the hot forging processing and the microstructure control of high Nb containing TiAl alloy were investigated using a relatively large forged pancake.

2. Experimental

The alloy with nominal composition Ti–45Al–9(Nb, W, B, Y) (at.%) was prepared using consumable electrode arc melting technique in an argon atmosphere, and remelted in a vacuum-skull melting furnace in order to reduce composition heterogeneity. The ingot was hot isostatic pressed (HIP) under an argon pressure of 140 MPa to eliminate casting porosity. The forging billet having a diameter of 115 mm and height of 245 mm was machined from the ingot. The canned billet was heated to $\alpha+\gamma$ phase region and hot forged to a reduction of the height of 75% using a nominal strain rate of $1.0\times10^{-4}\ s^{-1}$. In order to investigate

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Table 1
Heat treatments and the corresponding microstructure

Type	Initial samples	Heat treatment	Microstructure
HT1	As-forged	1310 °C/30 min/FC	DP, GS = 20 μm
HT2	After HT1	1180 °C/50 h/FC	NG, GS = 20 μm
HT3	After HT1	1345 °C/1 h transfer to 900 °C/2 h/AC	FL, heterogeneous
		1345 °C/2 h transfer to 900 °C/2 h/AC	FL, heterogeneous
HT4	After HT1	1320 °C/1 h transfer to 1345 °C/30 min transfer to 900 °C/2 h/AC	FL, CS = 20 μm
		1320 °C/1 h transfer to 1345 °C/1 h transfer to 900 °C/2 h/AC	FL, heterogeneous
HT5	After HT2	1345 °C/30 h transfer to 900 °C/2 h/AC	FL, CS = 15 μm
		1345 °C/1 h transfer to 900 °C/2 h/AC	FL, CS = 30 μm

AC, air cooling; FC, furnace cooling; GS, grain size; CS, colony size.

the microstructure evolution and phase transformations, a series of heat treatments were conducted for the as-forged pancake. Five types of the heat treatments are described in Table 1. The samples taken from the as-forged pancake were approximately 10×10×3 mm.

The microstructures of as-forged and heat-treated samples were studied by scanning electron microscopy (SEM) using backscattered electron imaging (BSE). A line-intercept method was used to measure the average grain size. All the samples were ground and mechanically polished. Metallographic samples were etched with Kroll's reagent. Phase analysis was performed before and after some heat treatments by X-ray diffraction technique (XRD). Measurements were carried out at room temperature with a Philips APD-10 diffractometer, using Cu Kα radiation for an angle range of 20–90° (2θ).

3. Results

3.1. Appearance and microstructure of the pancake

The appearance of can-forged pancake of Ti–45Al–9(Nb, W, B, Y) alloy is shown in Fig. 1. The deformed pancake has a regular shape without macro-cracks on the surface and no oxidization scale stripped. Metallographic examination shows that after canned forging, the majority zone of the pancake has homogenous duplex microstructure (DP), with fine grains (about 20–30 μm), while the surface zone has a fine, DP and curved coarse lamellar colonies (Fig. 2). BSE micrograph shows that the majority zone is composed of three phases with different contrast. They are lamellar colony, equiaxed γ and irregular grey phase with a volume fraction of about 15% (Fig. 3). Some bright particles enriched in Y and ribbons enriched in B are also seen in Fig. 3. X-ray diffraction spectra of the material confirm the presence of the three phases, namely γ, β(B2) and α_2. Energy dispersive spectroscopy analysis reveals that the grey phase contains higher Ti, W and Nb than the nominal composition and is identified as β(B2) phase (Table 2).

3.2. Microstructure evolution after different heat treatments

After HT1, microstructure of the samples revealed by SEM using BSE shows DP with grain size below 20 μm, similar to that in the as-forged material except the absence of the β(B2) phase. Therefore, the β(B2) phase is not stable and can be removed by the heat treatment mentioned above (Fig. 4). Near gamma (NG) microstructure is obtained after homogenization at 1180 °C, namely HT2, as shown in Fig. 5. The grain size is about 20 μm. The three types of heat treatments, namely TH3, HT4 and TH5, lead to different fully lamellar (FL) microstructure. There is a significant difference in grain size and morphology among the samples treated by the three kinds of processing. The samples heated to 1345 °C for 1 and 2 h and then held at 900 °C for 2 h followed by air cooling have FL microstructure. However, the grain size distribution is quite inhomogeneous, as shown in Fig. 6. Coarse lamellar grains, with stripes of fine grains form a non-uniform banded microstructure. Increasing the holding time at 1345 °C results in more heterogeneous microstructure. By holding at 1320 °C for 1 h and decreasing holding time at 1345 °C to 30 min refined FL microstructure is obtained (Fig. 7). Increasing holding

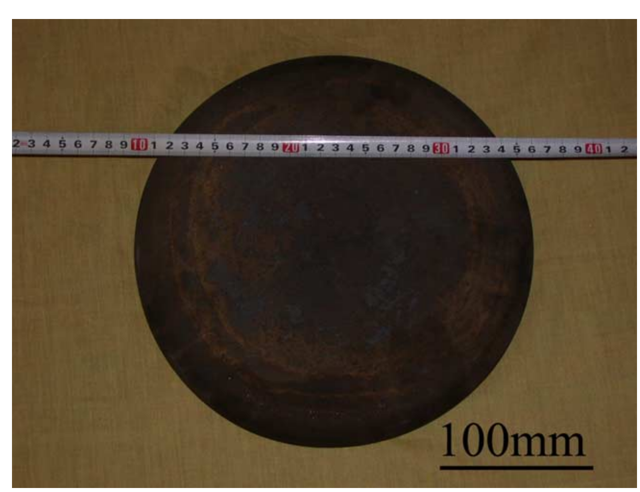

Fig. 1. Pancake of Ti–45Al–9(Nb, W, B, Y) alloy obtained by canned forging to deformation of 75% (Φ266×34 mm).

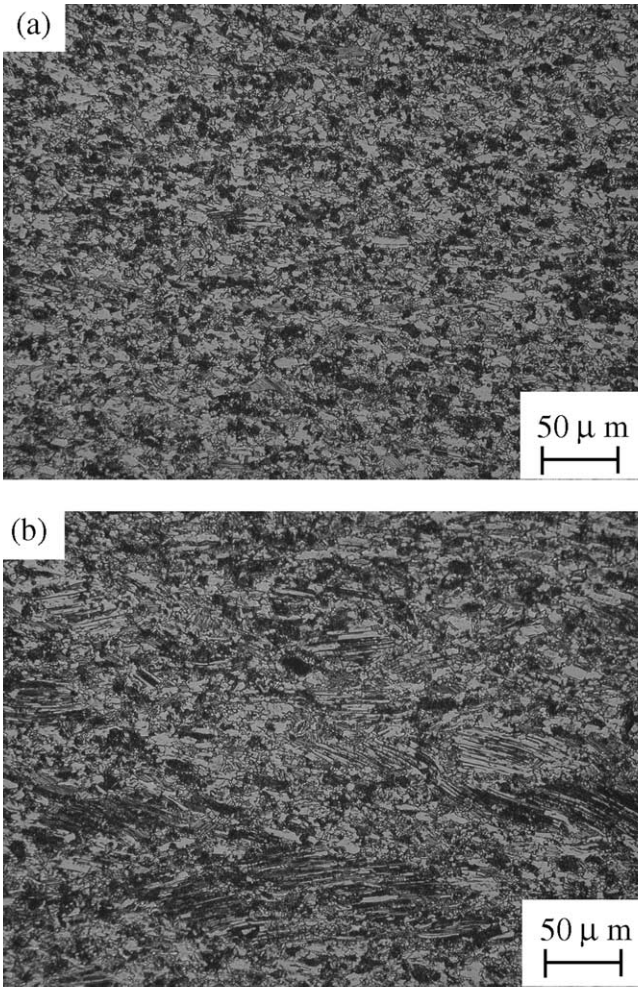

Fig. 2. Optical micrographs of the pancake for Ti–45Al–9(Nb, W, B, Y) alloy: (a) central zone; (b) near surface zone.

Table 2
Composition of the β(B2) phase (at.%)

	Ti	Al	Nb	W
Nominal composition	46	45	<9	<0.5
Actual composition	54.8	35.6	9.55	1.05

time at 1345 °C also leads to heterogeneous microstructure for this kind of heat treatment. Uniform FL microstructure with different grain size can be obtained by holding at 1345 °C for different time followed by holding at 900 °C for 2 h and air cooling for starting NG microstructure. The fine FL microstructure with a grain size of about 15 μm shown in Fig. 8 is obtained in a sample with a NG microstructure after holding at 1345 °C for 30 min.

4. Discussion

During the forging process, a large amount of strain energy can be imparted, which is certainly beneficial to homogeneous dynamic recrystallization. Therefore, fine, homogeneous DP microstructures have been developed in the majority zone of the pancake through dynamic recrystallization. However, the deformation of the upper and lower part of the ingot is smaller compared with the central zone due to the friction between specimen and dies, and the microstructure in the near surface zone is striped structure consisting of refined DP and retained lamellar structure.

At room temperature, Ti–45Al–9(Nb, W, B, Y) alloy should have an $\alpha_2+\gamma$ dual-phase structure according to the equilibrium phase diagram constructed by our group [6]. The diagram shows that during the solidification, the single β phase will transform consecutively to an $\alpha+\beta$ mixture, α

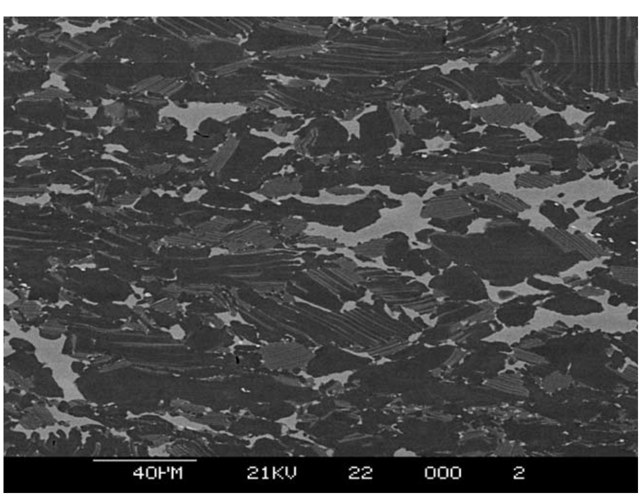

Fig. 3. Back scattered-electron micrograph of central zone of the as-forged pancake of Ti–45Al–9(Nb, W, B, Y) alloy with a microstructure consisting of γ/α_2 lamellae, equiaxed γ grains and some grey colonies of B2 at grain boundaries.

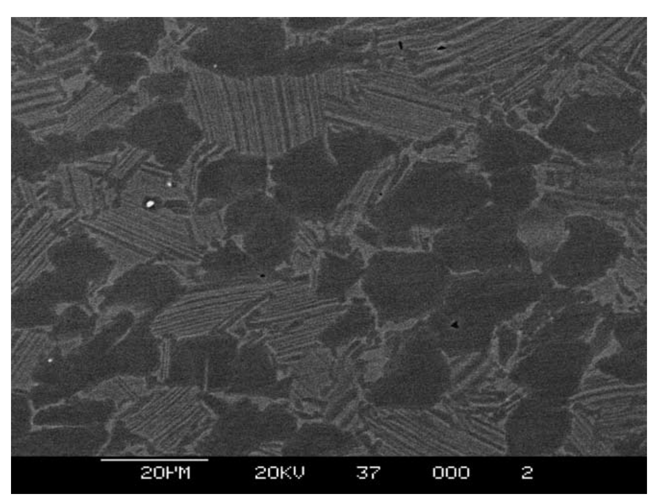

Fig. 4. Back scattered-electron micrograph of central zone of the pancake of Ti–45Al–9(Nb, W, B, Y) alloy with the B_2 phase eliminated after annealing at 1310 °C for 30 min.

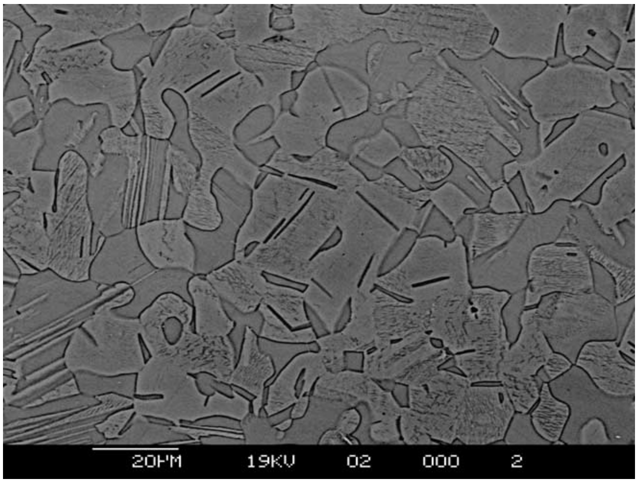

Fig. 5. NG microstructure obtained by heat treatment at 1180 °C for 50 h followed by furnace cooling.

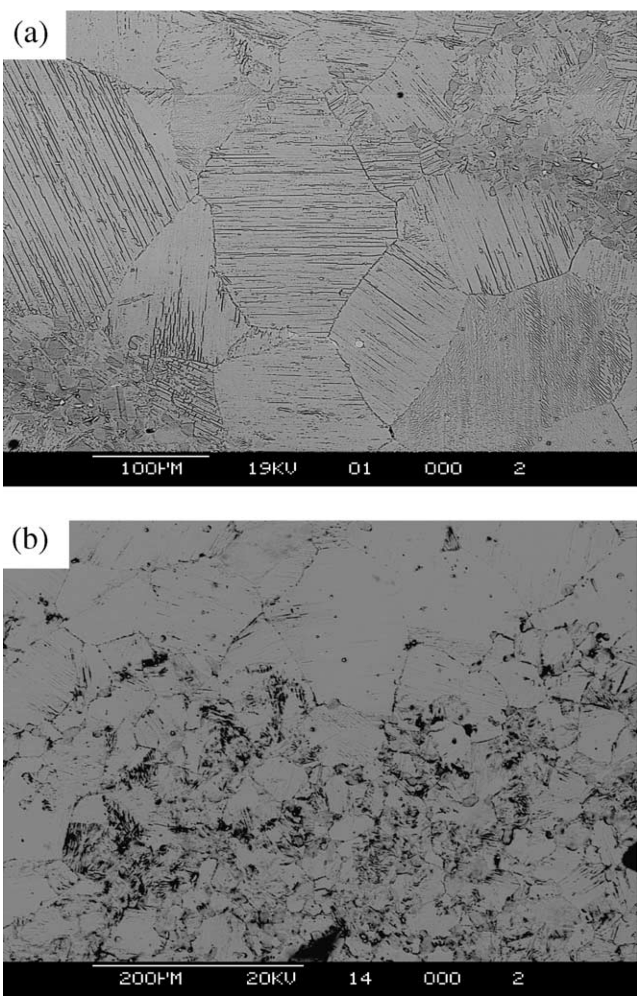

Fig. 6. Back scattered-electron micrographs showing heterogeneity of FL microstructure of the samples after heat treatment at 1345 °C for 1 and 2 h followed by holding at 900 °C for 2 h and air cooling: (a) holding at 1345 °C for 1 h; (b) holding at 1345 °C for 2 h.

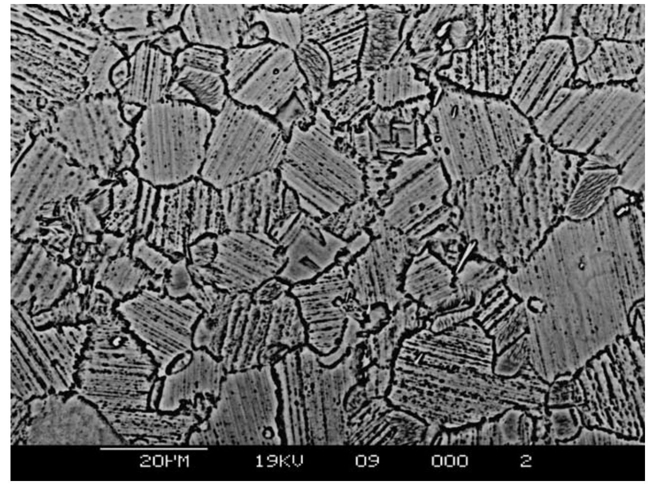

Fig. 7. Back scattered-electron micrograph of Ti–45A–9(Nb, W, B, Y) alloy obtained by heating at 1320 °C for 1 h, followed by heat treatment at 1345 °C for 30 min, holding at 900 °C for 2 h and air cooling.

single phase, $\alpha+\gamma$ mixture and $\alpha_2+\gamma$ mixture. But the solidification segregation of Nb and W results in existence of the $\beta(B_2)$ phase in the as-forged pancake, even though the HIP and forging are carried out [8]. After HT1 the $\beta(B_2)$ phase disappears. It can be concluded that composition homogeneity can only be realized by increasing the diffusion rate of the atoms. This is attributed to the high diffusion rate of W, Nb atoms activated by energy introduced by high temperature annealing.

For TiAl alloy microstructures that can be developed upon heat treatments are classified into NG, DP, near-lamellar (NL) and FL [9]. Various factors affect the microstructure in γ/α_2 alloy, such as cooling rate, solution treating temperature and time, initial microstructure and composition [10]. For the alloy studied, because of the high Nb addition, the eutectoid temperature (T_e) and α transus

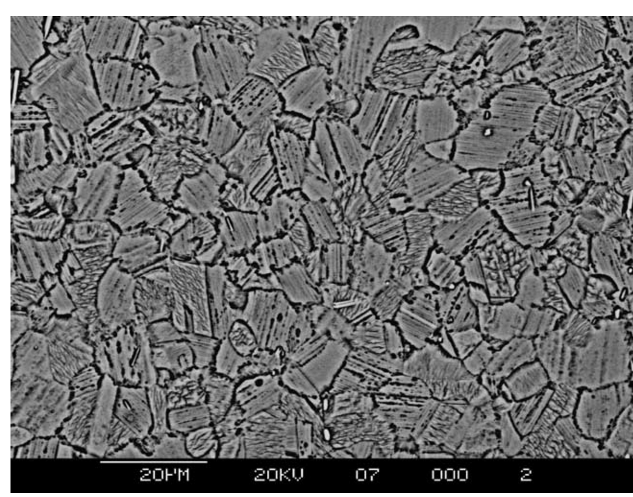

Fig. 8. Back scattered-electron micrograph of the specimen obtained by heat treatment at 1345 °C for 1 h, followed by holding at 900 °C for 2 h and air cooling for Ti–45Al–9(Nb, W, B, Y) alloy with NG microstructure. The homogenous FL microstructure with colony size of about 15 µm was shown.

temperature (T_α) are 1170 and 1330 °C, respectively [6]. The temperature of 1180 °C is just above T_e of Ti–45Al–9(Nb, W, B, Y). After holding at this temperature for a long time, the microstructures are γ and a few α. Therefore, NG microstructure forms upon cooling via the $\alpha \rightarrow \alpha_2/\gamma$ lamellar reaction and high temperature γ phase retention. The initial microstructures before heat treatment affect the colony size of the FL, especially the grain size and the deformation texture. Solution treatment at 1345 °C just above T_a, followed by holding at 900 °C for 2 h and air cooling, results in striped FL microstructure of coarse colony and fine colony. This is likely attributed to the texture produced during the casting or forging [11]. But holding shorter time at this temperature, α transformation cannot finish. By holding at 1320 °C just below T_a for 1 h, the microstructure consists of relatively fine α grains as the major constituent and fine gamma grains as minor, then raising to 1345 °C for a shorter time, the remained gamma grains transformed to α without coarsening of the existing α. Thus, the homogenous FL microstructure with finer colony size is obtained. But by this means, the uniform FL with larger colony size cannot be obtained due to the heterogeneous growth of the α grains because of the texture. For initial microstructure of NG, after holding at 1345 °C, the homogenous α phase is obtained, thus subsequent homogenous FL microstructure is obtained. The grain size of α phase and the colony size increases with increase of the holding time. As a result, the colony size of the FL microstructure can be controlled by this kind of heat treatment.

5. Conclusions

(1) In $\alpha+\gamma$ phase region, high Nb containing TiAl based alloy has good workability. High-quality pancake can be produced by canned forging of the ingot.

(2) Microstructure in the major part of the pancake is homogenous with a DP microstructure containing equiaxed γ and fine α_2/γ lamellae with a grain size of about 20–30 μm, while in the near surface zone is a fine-grained DP and retained lamellar colony. Due to solidification segregation, $\beta(B_2)$ phase exists at the grain boundaries and triple points of the grain boundaries. The $\beta(B_2)$ phase can be eliminated by annealing in $\alpha+\gamma$ phase region.

(3) Different microstructures can be obtained after various heat treatments for the as-forged pancake. NG microstructure is obtained by heat treatment at 1180 °C for 50 h. Heat treatment at 1345 °C followed by stabilization at 900 °C and air cooing leads to FL microstructure. Homogeneity of the microstructure is affected by the initial microstructure and processing. Fine FL obtained by heat treatment at 1320 °C for 1 h, followed by heat treatment at 1345 °C for 30 min is more homogenous than that obtained by heat treatment at 1345 °C directly. For the samples with NG microstructure heat treatment in the α phase region results in homogenous FL microstructure, and the colony size can be controlled by changing the holding time in the α phase region.

Acknowledgements

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