MICROSTRUCTURAL EVOLUTION OF TiAl-INTERMETALLIC ALLOYS CONTAINING TUNGSTEN AND BORON

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ABSTRACT

TiAl alloys have been considered as promising candidates for structural-materials applications at around 800°C [1]. In this work, new TiAl alloys, containing tungsten (W) and boron (B), have been developed. Using the scanning-electron microscopy (SEM), the effects of W and B on the microstructural evolution of TiAl alloys, including the colony size and lamellar spacing, were analyzed. It is important to point out that fine uniform microstructures (with the colony size smaller than 50 μm) can be conveniently developed after hot-isostatic pressing (HIP) the as-cast alloys at 1,250°C and 150 MPa for 4 h without the deformation process. It was found that tungsten prefers to react with boron to form borides, and disperses mainly along grain boundaries, and occasionally inside grains. With the increase of the tungsten content, the microstructure can be further refined. Heat treatments at temperatures ranging from 900°C to 1,310°C were conducted. The addition of tungsten can restrain the grain coarsening and stabilize the microstructure up to 1,280°C by hindering the migration of grain boundaries at high temperatures. It is also noteworthy that the β phase, a high-temperature ductile phase [2], forms when the tungsten content exceeds 0.4 atomic percent (at.%). The α-phase transus temperature, T_α, has been determined

through differential-thermal analyses (DTA) and further proved by the investigation of the microstructural changes during various heat treatments. Different microstructures meeting desirable needs can be developed through heat treatments beyond and below the α-phase transus temperature. The research is supported by the Fossil Energy Materials Program, with Dr. R. Judkins and Dr. J. Zollar as program managers, under the contact number of 11X-SP173V, and the National Science Foundation Combined Research-Curriculum Development Program, with Ms. Mary Poats as the program director, under the contract number of EEC-0203415.
Key words: TiAl alloy; microstructural evolution; alloying effect

INTRODUCTION

Two-phase TiAl alloys have been receiving considerable attentions because of their attractive properties, such as the low density, excellent high-temperature strength, and good oxidation resistance. However, TiAl alloys are quite brittle at room temperature and have relatively low fracture toughnesses [3]. The mechanical properties of the TiAl-based alloys with lamellar structures depend on three factors: the colony size, interlamellar spacing, and alloying addition. The tensile elongation at room temperature is strongly dependent on the lamellar colony size, showing the increased ductility with decreasing the colony size. The strength at room and elevated temperatures is sensitive to the interlamellar spacing, exhibiting the increased strength with decreasing the interlamellar spacing [4].

The as-cast TiAl-based alloys are usually coarse in the grain size. A refinement in the microstructures is necessary to meet the desirable applications. Additional elements, such as W, B etc., added into the base alloy can help refine the grain size [5-7]. Related heat treatments can facilitate the development of the desirable microstructures. In general, the fully lamellar (FL) structure has a better overall property than the other microstructures. But it has the low room-temperature tensile ductility. The duplex structure (DP) tends to have the good room-temperature tensile ductility, but its fracture toughness is low. This dilemma has not been satisfactorily solved recently. For the DP structure, it is hard to enhance its fracture toughness and creep resistance. On the other hand, the room-temperature ductility can be enhanced by refining the grain size of the FL structure. A fine fully lamellar microstructure has both good ductilities and fracture strengths, and the fracture-toughness and creep resistance are also better than the duplex microstructure [8].

For deformed TiAl-based alloys, the most optimal combination of the "composition-microstructure-property" can be obtained by different kinds of deformation techniques for the microstructural control, and by additional heat treatments for the microstructural rearrangement [9]. Some deformation methods, such as isothermal forging, may initiate small cracks [10]. Since most deformation techniques are costly, optimal solutions are in the search.

With the understanding of the relationship among the fabrication technique, the microstructure, and the related mechanical properties, optimal processing methods can be developed so as to control the microstructures of the TiAl intermetallics, and to improve the associated mechanical behaviors.

In this research, a refinement in the colony size of the TiAl-based alloy is obtained by alloying, and heat treatments. An investigation regarding the effect of the tungsten addition on the phase formation, the microstructural features, and the stability of both lamellar structures and β phases is investigated.

EXPERIMENTAL PRODEDURES

Reducing the Al concentration is effective in strengthening TiAl alloys. However, the toughness deteriorates, if the Al concentration is reduced too much. More than 45 at.% Al is required in order to secure toughness. The strength of the TiAl alloy is improved with the increase of the Nb concentration, but the elongation decreases. The oxidation resistance of TiAl-Nb alloys is improved up to 7.5 at.% Nb. It is accordingly concluded that 7.5 at.% is an optimal concentration of Nb. The B addition alone refines the lamellar structure, but causes fragmental and discontinuous α_2 lamellae. In contrast, the W + B addition refines the lamellar structure and produces more uniform and continuous α_2 lamellae [4-8]. In this research, the base TiAl intermetallic alloy is Ti-45at.%Al-7at.%Nb-χat.%W-0.15at.%B (χ = 0, 0.2, 0.4, 0.7, 1.1, and 1.5). TiAl-based samples fabricated at the Oak Ridge National Laboratory (ORNL) by Dr. C. T. Liu are labeled as:

#52 Ti-45at.%Al-7at.%Nb-0.2at.%W-0.15at.%B
#53 Ti-45at.%Al-7at.%Nb-0.4at.%W-0.15at.%B
#54 Ti-45at.%Al-7at.%Nb-0.7at.%W-0.15at.%B
#55 Ti-45at.%Al-7at.%Nb-0.15at.%B
#56 Ti-45at.%Al-7at.%Nb-1.1at.%W-0.15at.%B
#57 Ti-45at.%Al-7at.%Nb-1.5at.%W-0.15at.%B

All alloys were prepared by arc melting and drop casting, using pure Ti, Al, W, and Nb metal lumps together with the semiconductor-grade boron. Alloy buttons were melted at least 5 times, and, then, drop cast into a copper mold with an ingot size of 12.7 x 12.7 x 76.2 mm.

The TiAl-based sample alloys are hot-isostatic pressed (HIPed) at 1,250°C, for 4 h at a pressure of 150 MPa, and, then, heat-treated at 1,250°C for 16 h for the homogenization. The heat-treatment temperatures for the sample alloys have been scheduled as: at 900°C for 360 h; at 1,265°C for 18 h; at 1,280°C for 14 h; at 1,295°C for 10 h; and at 1,310°C for 5 h. Most alloy samples were encapsulated in quartz

tubes at a low inert-gas pressure. A small group of samples were heat-treated in a vacuum furnace (10^{-6} torr). Some specimens were cooled in air after heat treatments, and others were quenched by immersing capsules in water.

Specimens were prepared for the optical microscopy by conventional grinding and polishing, using a 0.5 μm diamond paste, followed by etching with the Kroll solution, 1%HNO_3+1%HF +98%H_2O (vol.%). Metallography was done on the REICHERT MeF3A optical microscope. The scanning-electron microscopy (SEM) is used for more detailed analyses of the specimens. The preparation of the specimens for SEM is the same as that for the metallography. For the backscattered-electron detection, specimens do not need any etching. For the secondary-electron detection, a Kroll solution is used for etching. JSM-5600LV is employed for the SEM analyses, with an acceleration voltage of 20 KV. The α-phase transus temperature, T_α, has been determined through differential-thermal analyses (DTA) and further proved by the investigation of the microstructural changes during various heat treatments.

RESULTS AND DISCUSSION

1. Effect of HIPing and homogenization on the microstructures of the as-cast alloys

The Ti-Al-Nb-W-B alloys are kept at a high temperature (1,250°C) and high pressure (150 MPa) during HIPing. The diffusion process takes place, and the defects, such as porosities in the as-cast material, are eliminated. Comparing Figs. 1 and 2, before and after the HIPing and homogenization, respectively, the microstructure of the Ti-Al-Nb-W-B alloy has changed greatly. The lamellar structure after the treatments is more continuous, and the colony size of the lamellae refines. As-cast microstructures are non-uniform, and Nb and W are richly dispersed in the α_2 phase, the β_2 phase, and the borides. During the HIPping and homogenization, the α phase preferentially nucleates at the metastable lamellae-colony boundaries, thus refining the as-cast coarse microstructure. After the homogenization, the Ti-Al-Nb-W-B alloy is fast cooled. The high-temperature α phase turns into a lamellar structure, and this trend made the inhomogeneous as-cast microstructure develop into a fine lamellar structure.

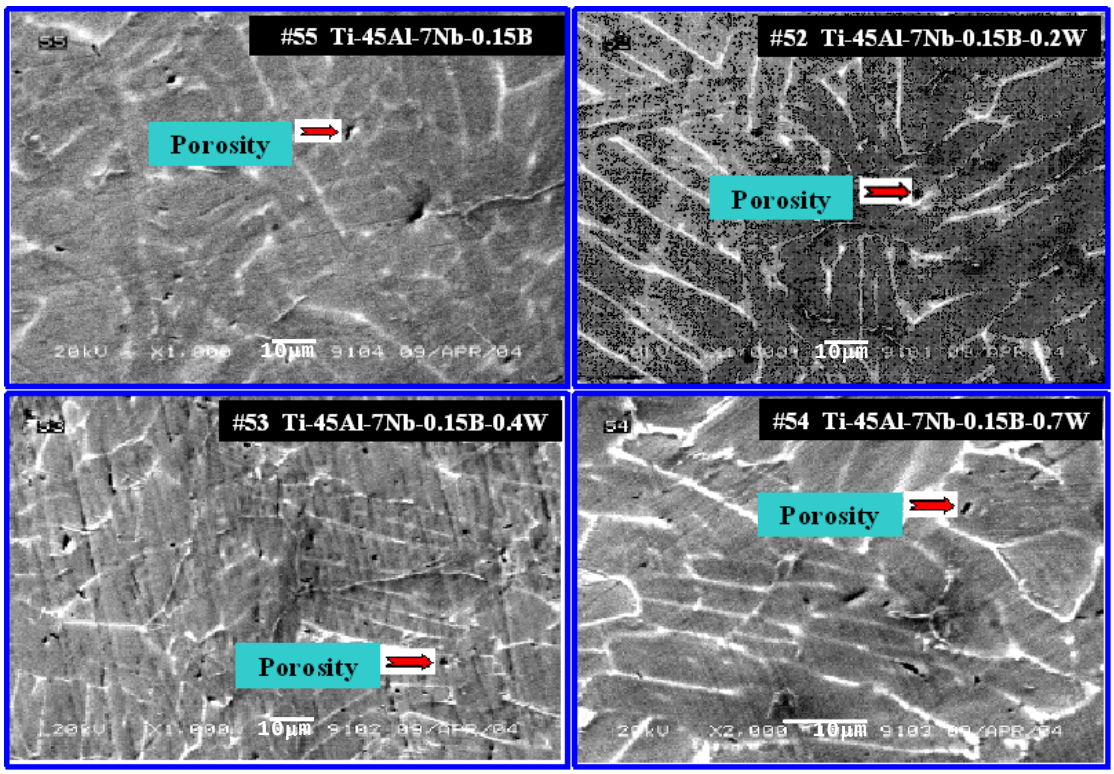

Figure 1 Backscattered-electron (BSE) images of the as-cast alloys

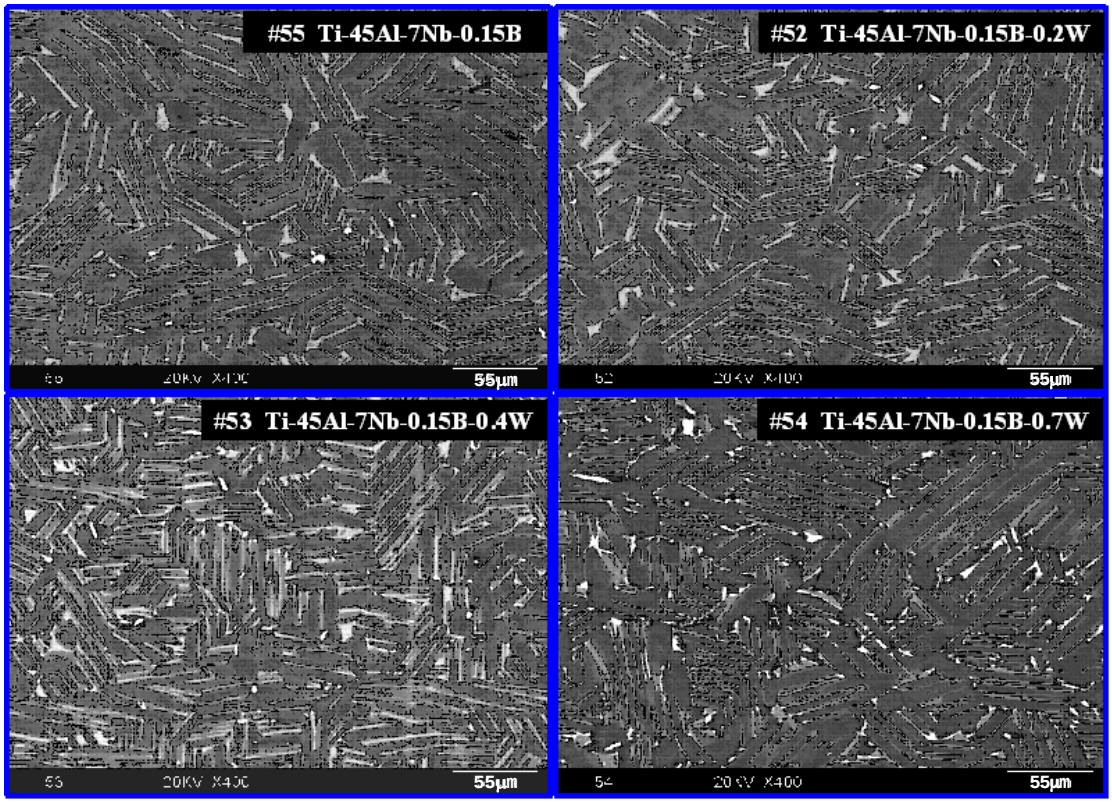

Figure 2 BSE microstructures of the Ti-Al-Nb-W-B alloys after the HIPing and homogenization

2. Effect of heat treatments on the microstructures of the alloys

The microstructures of the as-cast Ti-Al-Nb-W-B alloy are developed in a non-equilibrium condition within a γ matrix. The heat-treatment temperatures for the TiAl alloys have been scheduled as: 900°C for 360 h; 1,265°C for 18 h; 1,280°C for 14 h; 1,295°C for 10 h; and 1,310°C for 5 h. According to the binary phase diagram of the Ti-Al alloy, above the eutectoid temperature (T_e, approximately 1,175°C), as the heating temperature increases, the fraction of the γ phase tends to decrease, while the amount of the α phase increases in the Ti-45 at.% Al alloy. Following heat-treating the alloys below T_e, at 900°C for 360 h, and after the redistribution of the elements, the microstructure of the alloy is in an equilibrium condition. The microstructure of the alloy contains a blocky γ phase and α_2/γ lamellae, with the γ phase as the matrix. There is not much change in the grain size, shown in Figure 3.

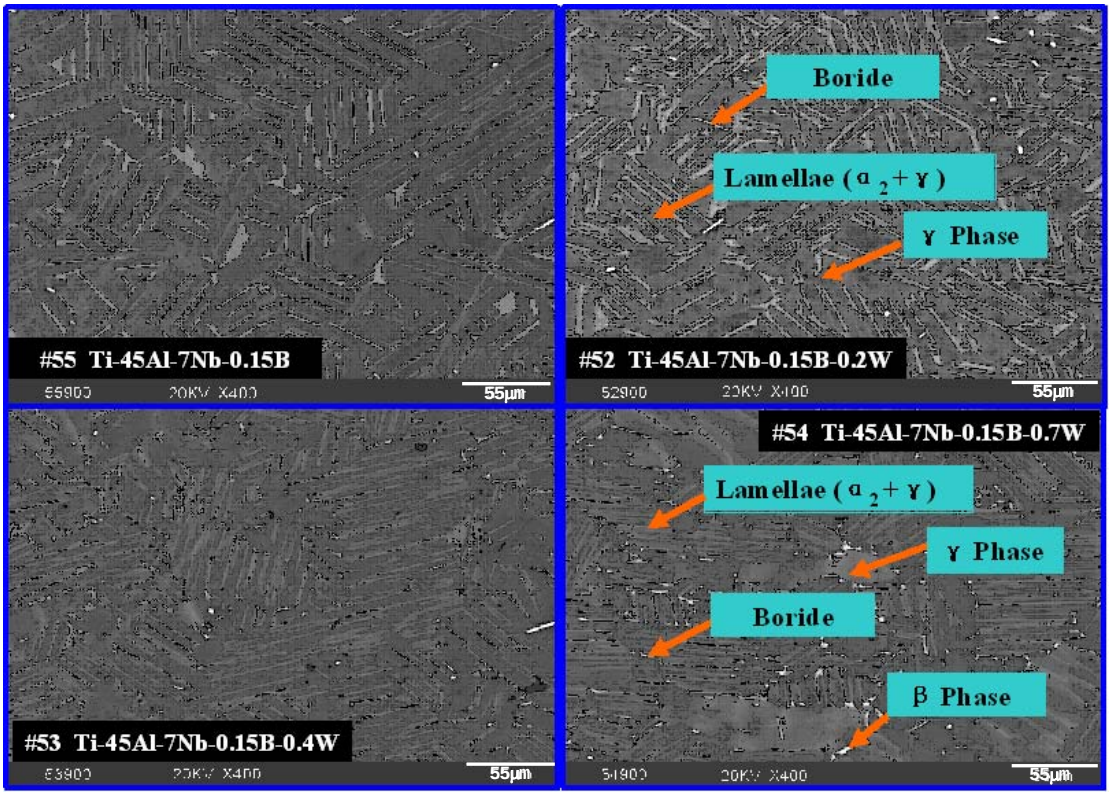

Figure 3 Microstructures of the Ti-Al-Nb-W-B alloys heat treated at 900°C / 360 h

After heat-treating the alloy above T_e, at 1,265°C for 18 h, the matrix of the alloy tends to change from the γ matrix to α matrix. The microstructure of the alloy is made up of the blocky γ phase and α_2/γ lamellae. The grain size does not change much in the alloy, but the interlamellar spacing tends to increase (Figure 4).

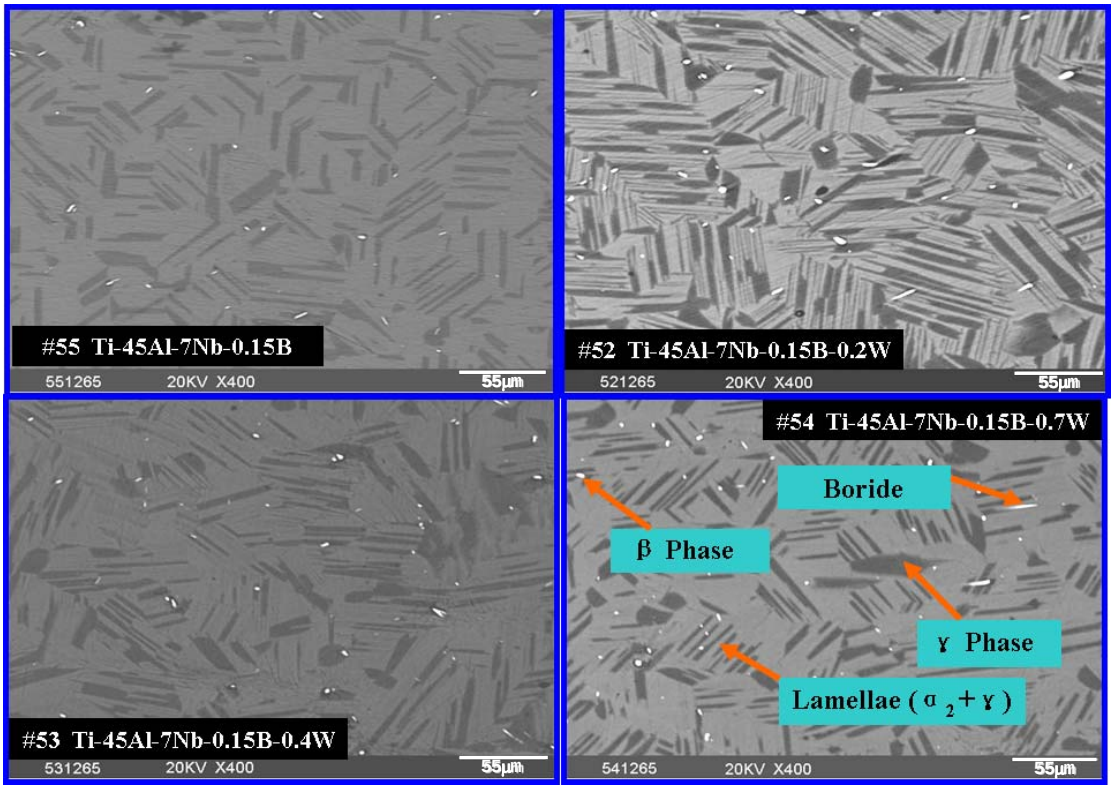

Figure 4 Microstructures of the Ti-Al-Nb-W-B alloys heat treated at 1,265°C / 18 h

After heat-treating the alloy at 1,280°C for 14 h, the amount of the γ phase starts to diminish. The grain size grows, and the interlamellar spacing increases (Figure 5).

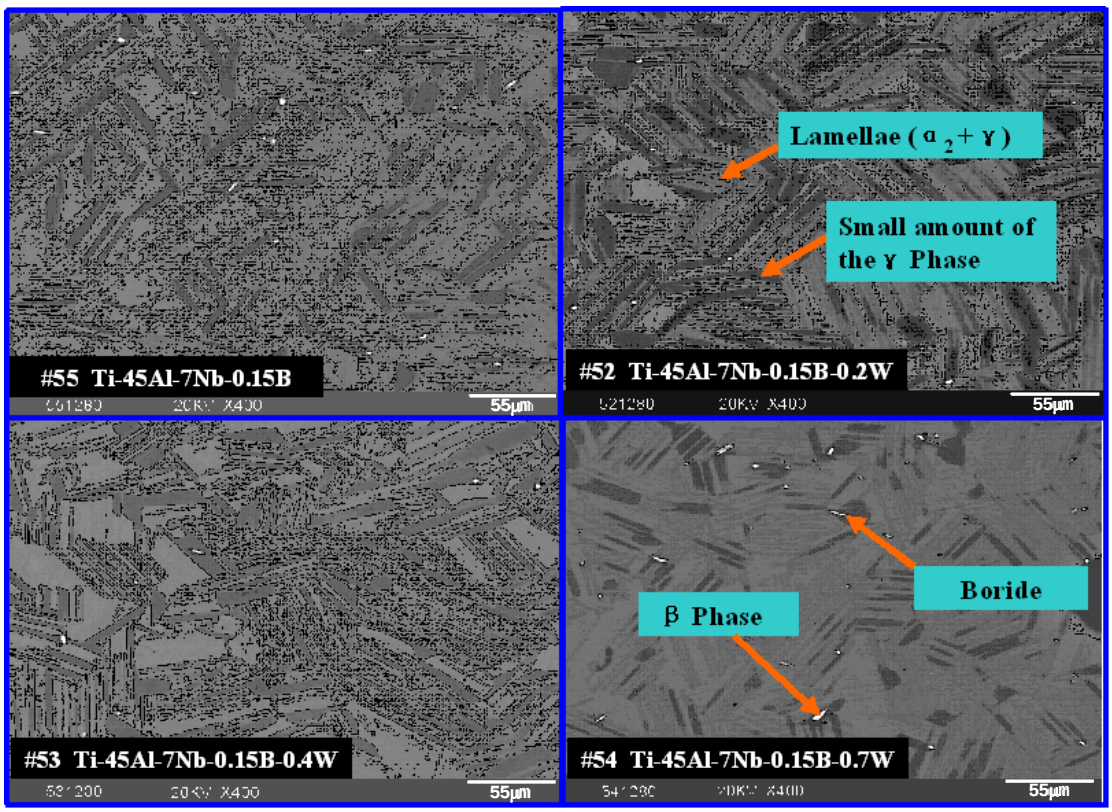

Figure 5 Microstructures of the Ti-Al-Nb-W-B alloys heat treated at 1,280°C / 14 h

After heat-treating the alloy at 1,295°C for 10 h and at 1,310°C for 5 h (Figs. 6 and 7, respectively), the microstructure of the alloy is composed of only a fully lamellar structure. No primary γ phase is found in the alloy. The grain size tends to grow fast. This is because when the heat-treatment temperature exceeds the α-phase transus temperature, T_α, the alloy enters the single-α phase region. Since the reaction, γ→α, takes place, almost all of the γ phase transforms into the α phase, and the α crystals tend to grow rapidly. During cooling, the reactions, such as α→α+γ, and $\alpha \rightarrow \alpha_2 + \gamma$, occur. The microstructure of the alloy, being heat treated at a temperature above T_α, depends mainly on the cooling rate of the alloy. When the alloy is being cooled very fast, the phase transformation has been suppressed, and no γ phase will form. The microstructure of the alloy is composed of the supersaturated α phase, which later on will tend to decompose into martensitic structures. If the alloy is cooled down at a medium pace, fine fully α_2/γ lamellae will be formed. When the alloy is cooled down slowly, the reaction, $\alpha \rightarrow \alpha_2 + \gamma$, takes place completely, the α_2/γ lamellae is equiaxed, and a fully lamellar structure will be formed.

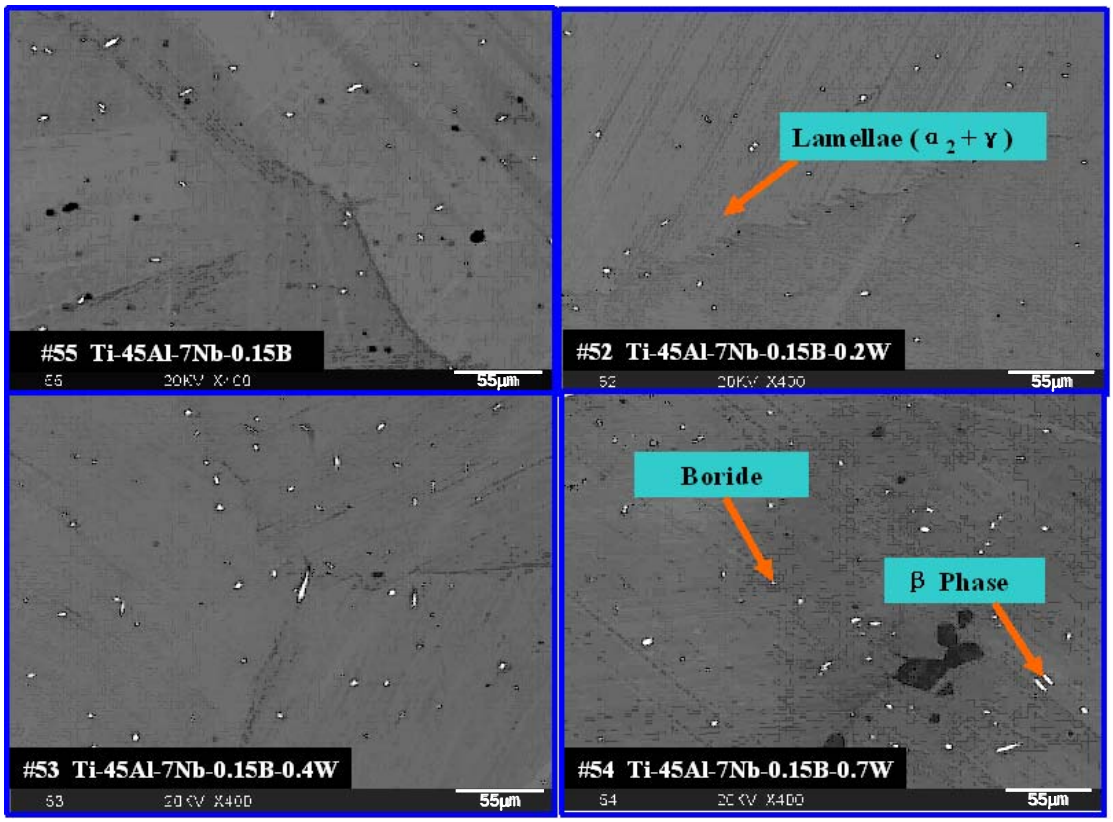

Figure 6 Microstructures of the Ti-Al-Nb-W-B alloys heat treated at 1,295°C / 10 h

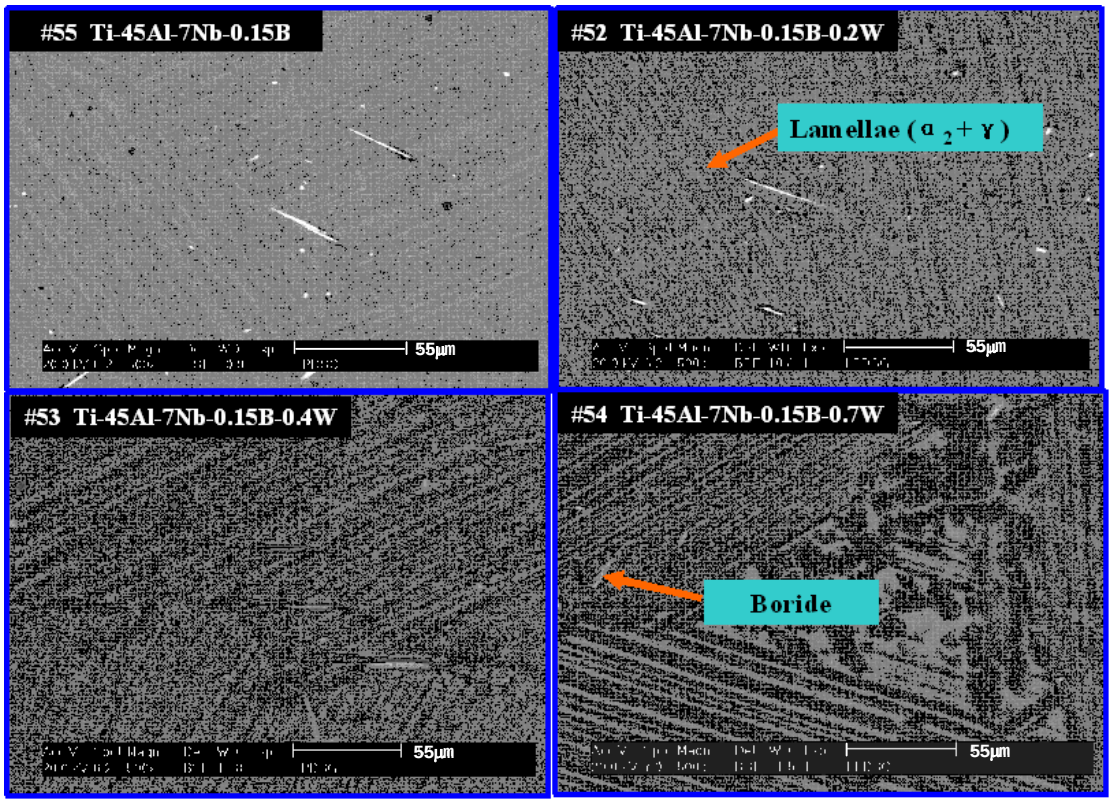

Figure 7 Microstructures of the Ti-Al-Nb-W-B alloys heat treated at 1,310°C / 5 h

3. Determination of the α-phase transus temperature

From the differential-thermal analyses (Fig. 8), the Ti-Al-Nb-W-B alloy shows an endothermic reaction in the temperature range of approximately 1,280°C-1,295°C, and the peak appears at a temperature around 1,290°C. This reaction indicates that, a phase transformation occurs at 1,290°C in the Ti-Al-Nb-W-B alloys. Comparing the microstructures in Figs. 3 to 7, obtained from different heat-treatment temperatures (900°C, 1,265°C, 1,280°C, 1,295°C, 1,310°C), the grain size does not change much for the 900°C, 1,265°C, and 1,280°C heat treatments, but it coarsens significantly at temperatures above 1,280^{0}C. Apparently, there is a phase transformation in the Ti-Al-Nb-W-B alloy at a temperature above 1,280°C and below 1,295°C, which involves $\alpha+\gamma\rightarrow\alpha$. Based on the results from the heat treatments and differential-thermal analyses, the α-phase transus temperature, T_α, of the Ti-Al-Nb-W-B alloy is equal to approximately 1,290 ± 5°C.

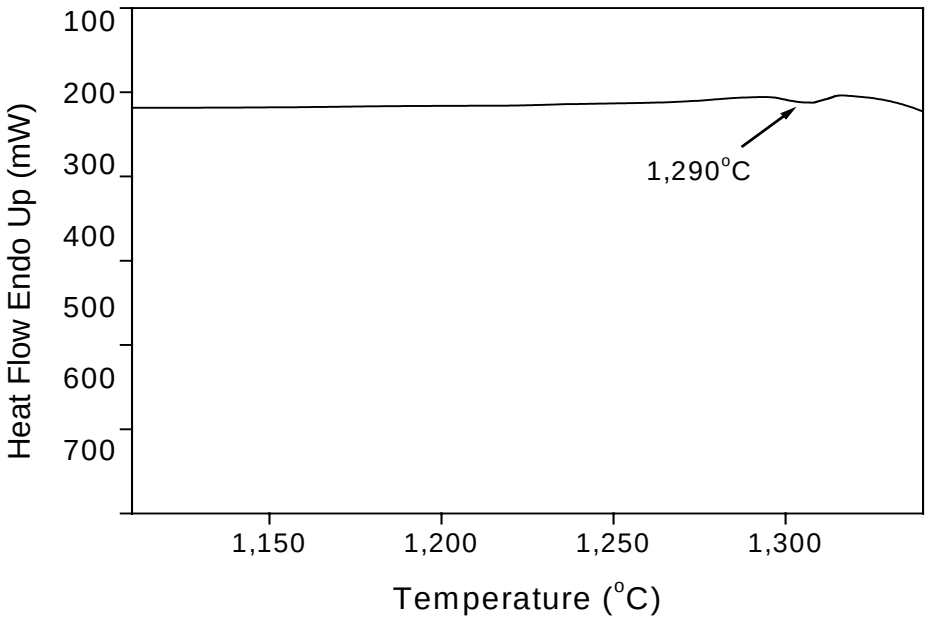

Figure 8 Differential-thermal analyses

4. Effect of W on the microstructures of the alloys

As the content of W differs, the equilibrium structure of the TiAl-based alloys varies. With the content of W increasing from 0 at.% to 0.2 at.%, 0.4 at.%, and 0.7 at.%, the colony sizes are 70 μm, 50 μm, 32 μm, and 25 μm, respectively; and the interlamellar spacings are 2.46 μm, 2.35 μm, 2.28 μm, and 2.08 μm, respectively. With the addition of W, the microstructure of the Ti-Al-Nb-W-B alloy refines. When the content of W increases, the grain size and the interlamellar spacing decrease. As the amount of W exceeds 0.4 at.%, the trend of the refinement slows down.

Table 1 shows the summary of heat treatments and tungsten contents on the microstructures of the TiAl alloys.

Table 1 Effect of heat treatments and tungsten contents on the microstructures of the TiAl alloys

W (at.%)	900^{0}C/ 360 h	1,265^{0}C/ 18 h	1,280^{0}C/ 14 h	T_α	1,295^{0}C/ 10 h	1,310^{0}C/ 5 h
0	γ, α_2+γ, boride	γ, α_2+γ, boride	γ, α_2+γ, boride		α_2+γ, boride	α_2+γ, boride
0.2	γ, α_2+γ, boride	γ, α_2+γ, boride	γ, α_2+γ, boride		α_2+γ, boride	α_2+γ, boride
0.4	γ, α_2+γ, boride	γ, α_2+γ, boride	γ, α_2+γ, boride		α_2+γ, boride	α_2+γ, boride
0.7	γ, α_2+γ, β, boride	γ, α_2+γ, β, boride	γ, α_2+γ, β, boride		α_2+γ, β, boride	α_2+γ, boride

CONCLUSIONS

1. Cellular structures and dendrites are formed in the as-cast Ti-Al-Nb-W-B alloys. This is because the addition of heavy metals, such as Nb and W, increased the undercooling of the liquid phase, which triggered the formation of the cellular structure and dendrite at the solid-liquid interface. Nb and W tend to segregate strongly at the interface of the cellular structure.
2. Porosities in the as-cast Ti-Al-Nb-W-B alloys can be generally eliminated by HIPing at 1,250°C. The cellular structures, dendrites, and macro-segregations of the alloys can also be eliminated after the HIPing and long-term homogenization at 1,250°C.
3. A small additional amount of W can refine the grain size of the Ti-Al-Nb-W-B alloys. The lamellar spacing also decreases with increasing the W concentration. But as the amount of W exceeds 0.4 at.%, its beneficial effect becomes small.
4. The addition of W promotes the formation of the β phase. When the amount of W exceeds 0.4 at.%, the β phase precipitates. The β phase in the blocky form mainly distributes along the grain boundary. Its composition is close to the α_2 phase, but with a less amount of Al.
5. From the results of microstructural changes related to different kinds of heat treatments, and differential-thermal analyses (DTA), the α-transus temperature, T_α, is estimated to be 1,290 ± 5°C.

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