Effects of Heat Treatment on Microstructure and Mechanical Properties of High Nb-TiAl Alloy Sheets (Postprint)

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Abstract

The effects of different heat treatment processes on the microstructure and mechanical properties of high Nb-TiAl alloy sheets were investigated. Sheets prepared by direct canned hot rolling of cast ingot material are primarily composed of residual coarsened lamellar colonies, recrystallized γ grains, and β phase distributed in bands along the rolling direction. Through different heat treatment processes, the residual lamellae and β phase can be eliminated, yielding typical duplex, near-lamellar, and fully-lamellar microstructures, respectively. Mechanical property tests at room and elevated temperatures were conducted on heat-treated sheets with duplex microstructure. The results demonstrate that after heat treatment, the room-temperature elongation of the hot-rolled sheets reaches 0.5%, with yield strength and tensile strength increasing to 646 MPa and 691 MPa, respectively. Compared with the as-cast condition, the room-temperature strength and ductility are improved. The material undergoes a ductile-to-brittle transition between 850–900°C, and the corresponding fracture mechanism transitions from brittle transgranular fracture to void nucleation and coalescence.

Full Text

Preamble

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Effect of Heat Treatment on Microstructure and Mechanical Properties of High Nb-TiAl Alloy Sheet

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Abstract
This study investigated the effect of heat treatment on the microstructure and mechanical properties of a high Nb-containing TiAl alloy sheet fabricated by directly hot-rolling a packed alloy ingot. The as-rolled sheet mainly consisted of remnant coarsened lamellar colonies, recrystallized γ grains, and strip-like β phase distributed along the rolling direction. Subsequent heat treatments eliminated the coarse lamellae and β phase, yielding various typical microstructures including duplex, near fully lamellar, and fully lamellar structures. Mechanical properties of the sheet with duplex microstructure were tested at room and elevated temperatures. The results showed that the strength and ductility at room temperature were improved after heat treatment. The brittle-ductile transition temperature ranged between 850-900℃, with the corresponding fracture mode transforming from brittle transgranular fracture to nucleation and coalescence of voids.

Key words metallic materials, high Nb-TiAl alloy, hot-pack rolling, heat treatment, duplex microstructure, mechanical property

Introduction

Titanium aluminide-based alloys are promising lightweight high-temperature structural materials due to their low density, excellent high-temperature strength, oxidation resistance, and creep properties. Although mechanical properties can be improved through alloying and microstructural control, conventional TiAl-based alloys are limited to service temperatures below 850°C. In contrast, high Nb-TiAl alloys, with the addition of high-melting-point Nb, exhibit increased melting points and ordering temperatures, enabling service temperatures of 900°C and above. Furthermore, high Nb-TiAl alloys demonstrate superior oxidation resistance while maintaining the advantages of low density, simple crystal structure, and microstructure-controlled property optimization.

After years of fundamental research, high Nb-TiAl alloys have entered the practical application stage, with sheet production and application representing a critical research direction. Currently, TiAl alloy sheets are primarily fabricated through ingot metallurgy and powder metallurgy methods, both of which involve complex processes and high equipment requirements. Consequently, developing a short-process, high-efficiency manufacturing route is essential. Our research group has developed a novel process for producing large-scale high Nb-TiAl alloy sheets through direct hot-pack rolling of as-cast ingots. The properties of hot-rolled sheets depend not only on alloy composition and processing parameters but also on subsequent heat treatment strategies. This paper focuses on the influence of heat treatment on the microstructure and mechanical properties of the rolled sheets.

1 Experimental Methods

The experimental alloy had a nominal composition of Ti-45Al-8.5Nb-0.2B-0.2W-0.02Y (at.%). The alloy ingot, weighing 2 tons, was prepared by plasma cold hearth melting. Rectangular blanks measuring 120 mm × 80 mm × 15 mm were directly cut from the ingot, sealed in specially designed stainless steel packs, and subjected to hot-pack rolling. The rolling temperature was 1280°C with a 30-minute preheating hold, an average reduction of 25% per pass, and 15-minute inter-pass reheating. After four rolling passes, the average sheet thickness was 4.5 mm, corresponding to a total deformation of 70%.

The critical phase transformation temperatures for the high Nb-TiAl alloy were determined to be: (1) the $\alpha \rightarrow \alpha+\gamma$ transition temperature at 1330°C; (2) the temperature where α and γ phases have equal volume fraction in the $\alpha+\gamma$ region at 1250°C; and (3) the eutectoid temperature at 1175°C. Square specimens (10 mm × 10 mm) were cut from the rolled sheet for heat treatment in a box furnace. Detailed heat treatment parameters are listed in .

Tensile specimens with a gauge section of 14 mm × 4 mm × 1.5 mm were cut from both the ingot and the sheet along the rolling direction. Room- and high-temperature tensile tests were conducted on a CM75105 universal testing machine at a strain rate of 5$×10^{-4}$ s^{-1}. For microstructural analysis, all observation planes were longitudinal sections of the sheet. Microstructures of as-cast, as-rolled, and heat-treated specimens were examined using backscattered electron imaging (BSE-SEM) on a Supra 55 scanning electron microscope. BSE-SEM samples were prepared by electrolytic polishing in a solution of 65% methanol, 30% n-butanol, and 5% perchloric acid (by volume). Phase identification was performed using an Inca X-Max energy dispersive spectrometer (EDS) and an APD-10 X-ray diffractometer (XRD, CuKα).

2 Results and Discussion

2.1 As-Cast Microstructure

Accurate determination of phase transformation temperatures is crucial for selecting appropriate heat treatment parameters. Based on differential thermal analysis and metallographic observations, the as-cast microstructure of the high

Nb-TiAl alloy is shown in [Figure 1: see original paper]. The as-cast structure exhibited a typical near lamellar morphology, consisting primarily of lamellar colonies (α_2+γ) with minor amounts of white-contrast β phase and black-contrast equiaxed γ grains distributed between lamellar colonies. Due to boron addition and the β solidification path, the lamellar colonies were relatively fine, approximately 150 m in size. Additionally, trace additions of B and Y resulted in small amounts of white particulate and needle-like borides, as well as minor Y_2O_3 particles, within colony boundaries and interiors. Higher magnification observation [Figure 1b: see original paper] revealed that β phase existed not only between lamellar colonies but also within them, arising from β segregation and α segregation, respectively. EDS microanalysis of the as-cast alloy confirmed that the β phase was enriched in Nb and W but depleted in Al, indicating that Nb and W are β-stabilizing elements. The XRD pattern of the as-cast alloy [Figure 2: see original paper] further verified the presence of β phase.

2.2 As-Rolled Microstructure

The BSE micrograph of the high Nb-TiAl alloy sheet after hot-pack rolling is presented in [Figure 3: see original paper]. The as-rolled microstructure consisted mainly of recrystallized γ grains, remnant lamellar colonies, and β phase distributed in bands along the rolling direction [Figure 3a: see original paper]. The remnant lamellar colonies were primarily composed of coarsened γ lamellae [Figure 3b: see original paper], indicating that γ coarsening occurred within lamellar colonies during hot rolling. High-magnification BSE imaging [Figure 3c: see original paper] showed that equiaxed recrystallized grains were elongated along the rolling direction, with white-contrast β phase present at triple junctions. Boride observation revealed that needle-like borides were fragmented into several segments during hot rolling [Figure 3d: see original paper], which is beneficial for subsequent microstructural control and mechanical property improvement.

2.3 Effect of Heat Treatment on As-Rolled Microstructure

According to the high Nb-TiAl alloy phase diagram, uniform fine duplex microstructures should be obtained by holding in the (α+γ) region where the two phases have equal volume fractions. Two different heat treatment schedules were designed .

After HT1 heat treatment, the microstructures are shown in [Figure 4: see original paper]. Following 8 hours at 1250°C [Figure 4a: see original paper], the white-contrast β phase bands along the rolling direction partially dissolved and transformed to α phase, while remnant lamellae began to decompose. Extending the holding time to 16 hours [Figure 4b: see original paper] eliminated the β phase completely, though small amounts of lamellar colonies remained. After 24 hours [Figure 4c: see original paper], all remnant lamellar colonies decomposed, resulting in a uniform fine duplex microstructure with an average grain size of approximately 30 m. Notably, the grains transformed from coarsened lamellae were similar in size to surrounding grains. Due to the equal volume fractions of γ and α phases, strong mutual pinning prevented grain growth even during prolonged holding.

Heat treatment HT2 produced a non-uniform duplex microstructure [Figure 5a: see original paper]. Phase diagram analysis indicates that in the $\alpha+\gamma$ region, increasing temperature increases the α phase fraction while decreasing the γ phase fraction. Holding at 1300°C resulted in a γ volume fraction significantly lower than α, weakening the pinning effect of γ on α and allowing some α grains to grow into coarse α phase. During furnace cooling, γ phase precipitated along the (0001) planes of α, forming coarse lamellar colonies [Figure 5b: see original paper] up to 100 m in size.

Heat treatments HT3 and HT4 eliminated remnant lamellar colonies and β phase in relatively short times, yielding near fully lamellar and fully lamellar microstructures, respectively. After HT3, a relatively uniform near fully lamellar structure was obtained due to minor γ grains at colony boundaries [Figure 6a: see original paper]. In contrast, HT4 resulted in significant coarsening and non-uniform lamellar colony sizes because α grains grew abnormally during holding in the α single-phase region [Figure 6b: see original paper].

2.4 Mechanical Properties and Fracture Morphology

High Nb-TiAl sheets with duplex microstructures were obtained through heat treatment HT1-3, which eliminated remnant lamellae and β phase. Mechanical properties were tested at room and elevated temperatures.

As shown in [Figure 7: see original paper], compared with the as-cast structure, both room-temperature ductility and strength were improved. The room-temperature elongation increased from 0.1% in the as-cast condition to 0.5% after heat treatment, with yield strength and ultimate tensile strength reaching 646 MPa and 691 MPa, respectively. These properties approach those of twice-forged alloys. High-temperature tensile properties of the duplex microstructure sheet are shown in [Figure 8: see original paper]. With increasing temperature, yield and tensile strengths gradually decreased while elongation increased. Below 850°C, elongation remained below 1%, but increased dramatically to 64% at 900°C, indicating a brittle-ductile transition between 850°C and 900°C.

Fracture morphologies of tensile specimens at room temperature are presented in [Figure 9: see original paper]. The as-cast specimen exhibited primarily trans-lamellar and inter-lamellar fracture [Figure 9a: see original paper], while the rolled sheet showed numerous cleavage facets with predominantly transgranular cleavage fracture [Figure 9b: see original paper], both indicating brittle fracture modes.

[Figure 10: see original paper] shows high-temperature fracture morphologies of the rolled sheet at different temperatures. At 850°C, fracture occurred mainly through mixed transgranular and intergranular modes. At 900°C, the fracture surface consisted of numerous micro-voids and larger dimples, indicating ductile fracture dominated by void nucleation and coalescence. Thus, the fracture mode of the duplex microstructure transformed from brittle transgranular/intergranular fracture to ductile void nucleation and coalescence with increasing temperature.

2.5 Discussion

In deformed TiAl-based alloys, the plastic anisotropy of lamellar colonies leads to remnant lamellar structures that adversely affect mechanical properties. For high Nb-TiAl alloys, Nb addition enhances the thermal stability of lamellar colonies, making them difficult to eliminate through heat treatment. However, after direct hot-pack rolling of as-cast high Nb-TiAl alloy, remnant lamellar colonies underwent significant coarsening, particularly of γ lamellae, substantially reducing their thermal stability and enabling elimination through heat treatment. Holding in the α+γ region allowed remnant lamellae to decompose into equiaxed grains, with required holding time decreasing at higher temperatures. Semiatin' s research indicates that to reduce surface energy, fine lamellae first coarsen, then transform to equiaxed grains through static spheroidization. In this study, lamellae within remnant colonies were already coarsened during hot rolling, requiring only static spheroidization to form equiaxed grains during heat treatment.

During thermomechanical processing, the disordered β phase, with numerous independent slip systems and lower strength than α and γ phases, acts as a lubricant between lamellar colonies, relieving stress concentration and preventing cracking. Therefore, β phase presence enhances the hot workability of TiAl-based alloys. However, at room temperature, β phase typically has an ordered B2 structure with low strength along (100) cleavage planes, promoting crack initiation and severely degrading mechanical properties. Thus, β phase should be retained during hot working and subsequently eliminated through post-deformation heat treatment. BSE micrographs of the as-rolled sheet [Figure 3: see original paper] show that β phase morphology changed but remained distributed in bands along the rolling direction. Both Nb and W are strong β-stabilizing elements that segregate severely during solidification, and W has low diffusivity. The short high-temperature exposure during rolling resulted in substantial retained β phase. Quantitative analysis showed the β phase volume fraction remained approximately 6% before and after rolling, indicating no significant decomposition. Therefore, successful sheet fabrication was partially attributed to the ductile β phase. Post-rolling heat treatment studies demonstrated that β phase gradually dissolved into α phase as Nb and W homogenized, with higher temperatures accelerating diffusion and shortening the time required for β phase elimination. Consequently, subsequent heat treatment can eliminate β phase and its detrimental effects on room-temperature mechanical properties.

Duplex microstructures in TiAl-based alloys offer high strength and good ductility. By controlling heat treatment parameters, β phase and remnant lamellae can be eliminated to obtain uniform fine duplex microstructures. Compared with as-cast structures, significant improvements in room-temperature ductility and strength were achieved, benefiting from microstructural refinement through hot-pack rolling and homogenization through heat treatment.

Conclusions

1. Large-scale high Nb-TiAl alloy sheets can be fabricated through hot-pack rolling of as-cast ingots with a deformation of 70%. The as-rolled microstructure consists of remnant lamellar colonies, recrystallized γ grains, and banded β phase distributed along the rolling direction.
2. By controlling heat treatment parameters, β phase and remnant lamellar colonies can be eliminated to obtain fine uniform duplex, near fully lamellar, and fully lamellar microstructures.
3. Sheets with duplex microstructure exhibit good room-temperature mechanical properties, with 0.5% elongation, and yield and ultimate tensile strengths of 646 MPa and 691 MPa, respectively, comparable to properties of twice-forged alloys.

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