Effect of Si on Microstructure and Room Temperature Tensile Properties of High Nb-TiAl Alloys (Postprint)

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Abstract

The effects of silicide (Nb5Si3 phase) precipitation on the microstructure and room-temperature tensile properties of high Nb-TiAl alloys were investigated. Experimental results show that the precipitation temperature of silicides is between 1000~1200 ℃, with precipitates located at lamellar colony grain boundaries, b(B2) phase segregation regions, and between lamellae. Upon Si addition, the room-temperature tensile properties of the alloy increased, because the formation of Nb5Si3 phase reduces the content of Nb, which is a b(B2) phase stabilizing element, leading to a decrease in the volume of the brittle b(B2) phase. However, for Si-containing high Nb-TiAl alloys after heat treatment, the room-temperature tensile properties gradually decrease with increasing heat treatment temperature. This is attributed to the fact that silicides precipitated along lamellae can cause crack initiation and proliferation along the lamellae, and stress can lead to further precipitation of silicides, accelerating crack propagation. Moreover, the addition of Si leads to an expansion of the γ phase region, forming a γ single-phase region between 1280~1300 ℃. Precipitation of silicides at lamellar boundaries leads to a massive γ+b(B2) phase structure, embrittling grain boundaries; whereas precipitation of silicides within lamellae results in the formation of secondary γ laths, fragmenting the initial lamellar structure.

Full Text

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Abstract

High Nb-TiAl alloys, regarded as a new generation of TiAl alloys, have attracted increasing attention due to their higher operating temperature and better oxidation resistance compared with conventional TiAl alloys. Previous work by our research group found that the precipitated particles in high Nb-TiAl alloys are Nb_5Si_3 rather than the Ti_5Si_3 phase that precipitates in conventional TiAl alloys. This study investigates the effects of Nb_5Si_3 phase precipitation on the microstructure and room-temperature tensile properties of high Nb-TiAl alloys. Experimental results show that silicide precipitation occurs between 1000-1200 °C, with precipitates located at colony boundaries, β(B2) phase segregation zones, and between lamellae. The addition of Si improves the room-temperature tensile properties of as-cast alloys because the formation of Nb_5Si_3 precipitates reduces the Nb content, which is a β(B2) phase stabilizing element, thereby decreasing the volume fraction of the brittle β(B2) phase. However, after heat treatment, the room-temperature tensile properties of Si-containing high Nb-TiAl alloys gradually deteriorate with increasing heat treatment temperature. This degradation occurs because silicides precipitated along lamellae facilitate crack initiation and propagation along the lamellar interfaces. Moreover, tensile stress induces further silicide precipitation, accelerating crack growth. Additionally, Si addition expands the γ-phase region, forming a γ single-phase region between 1280–1300 °C. Silicide precipitation at lamellar boundaries leads to massive $\gamma+\beta$(B2) phases that embrittle grain boundaries, while silicide precipitation within lamellae promotes the formation of secondary γ laths that fracture the initial lamellar structure.

Keywords: high Nb-TiAl alloy, Si alloying, microstructure evolution, room temperature tensile properties

Introduction

TiAl-based alloys are promising lightweight high-temperature structural materials for aerospace and automotive applications due to their low density, high creep resistance, and excellent high-temperature mechanical properties and oxidation resistance. However, cast TiAl alloys undergo a peritectic transformation ($L+\beta\rightarrow\alpha$) that results in coarse microstructures, pronounced casting texture, and anisotropic properties. The addition of high Nb content (5–10 at%) not only increases the service temperature of TiAl alloys by approximately 100 °C but also enhances high-temperature strength and oxidation resistance. Furthermore, Nb is a strong β(B2) phase stabilizing element, and high Nb addition changes the solidification path from the peritectic transformation (L+$\beta\rightarrow\alpha$) to $\beta\rightarrow\alpha$ solidification. This β solidification refines the microstructure and eliminates anisotropy. Due to these advantages, high Nb-TiAl alloys have become a major research direction for developing high-temperature, high-performance TiAl alloys and have attracted widespread attention. Nevertheless, the expansion of the β(B2) phase region increases β(B2) phase segregation in the alloy. The ordered β(B2) phase with a CsCl structure formed after solidification is brittle at room temperature, promoting crack initiation and propagation that is detrimental to room-temperature mechanical properties. Prolonged heat treatment can hardly eliminate the β(B2) phase completely, and it also causes coarsening and growth of lamellar colonies. Therefore, eliminating β(B2) phase segregation represents a key research focus for β-solidified TiAl alloys.

In conventional TiAl alloys, Si addition leads to the formation of Ti_5Si_3 phase with a hexagonal $D8_8$ structure after creep deformation or high-temperature heat treatment. This phase improves room-temperature ductility and enhances high-temperature oxidation resistance and creep performance. Ti_5Si_3 phase exhibits specific orientation relationships with γ and α_2 phases. Our previous work on Si alloying in high Nb-TiAl alloys revealed that the precipitated particles are not Ti_5Si_3 as in conventional TiAl alloys, but rather Nb_5Si_3 (designated as ε), which shows the following orientation relationship with the lamellar matrix: $(11\bar{0}0)_\varepsilon//(11\bar{2})_\gamma$ and $(11\bar{0}0)_\varepsilon//(11\bar{0}0)_\alpha$, $(112\bar{3})_\varepsilon//(112\bar{0})_\alpha$, $(112\bar{3})_\varepsilon//(110)_\gamma$. This work primarily investigates the precipitation conditions of Nb_5Si_3, its influence on β(B2) phase segregation, and its effects on the microstructure and tensile properties of high Nb-TiAl alloys.

Experimental Methods

The nominal compositions of the alloys used in this study were Ti-45Al-8Nb-2Mn and Ti-45Al-8Nb-2Mn-0.5Si (at%), designated as UM and US alloys, respectively. Alloy ingots weighing 2 kg were melted in a vacuum induction levitation furnace and remelted twice to ensure homogeneity before being cast into graphite molds with a diameter of 50 mm and length of 150 mm. Heat treatment of the US alloy was performed in a box furnace using the processes listed in .

Microstructural observation was conducted using a SUPRA 55 field-emission scanning electron microscope (SEM) in backscattered electron (BSE) mode. Phase composition and distribution were analyzed by electron backscatter diffraction (EBSD). SEM and EBSD samples were ground with SiC abrasive papers from coarse to fine grades up to 1500 grit and then electropolished. The electropolishing solution consisted of CH_3OH, $CH_3(CH_2)_3OH$, and $HClO_4$ with a volume ratio of 13:6:1. Phase transformation temperatures were determined using a STA 449 C Jupiter differential scanning calorimeter (DSC). Tensile test specimens were plate-shaped with a gauge length of 10 mm and cross-section of 1.5 mm × 4 mm. Tensile tests were performed on an Instron 5969 universal testing machine at a strain rate of 5×10^{-4} s^{-1}. Microstructural morphology and structural analysis were examined using a Tecnai G2 F20 transmission electron microscope (TEM). TEM samples were thinned into foils using a twin-jet electropolishing unit at 27 V and -30 °C.

Results

Microstructure of As-Cast Alloys

[Figure 1: see original paper] shows SEM-BSE and EBSD images of the as-cast microstructures of both alloys. The UM alloy exhibits a near-lamellar microstructure with numerous whisker-like β(B2) phase segregations distributed within the lamellae. After adding 0.5% Si, the US alloy shows reduced whisker-like segregation. A mixed $\gamma+\beta$(B2) layer exists at colony boundaries in the US alloy, with silicide precipitation in the middle. The silicides precipitated at colony boundaries in the as-cast condition are defined as ε_1, representing the first type of silicide precipitation formed by supersaturated Si during solidification. EBSD phase analysis results for both alloys are presented in , revealing that the US alloy contains ε (Nb_5Si_3) phase and shows significantly reduced β(B2) phase content, decreasing from 4.54% to 0.56%.

Microstructure After Heat Treatment

[Figure 2: see original paper] illustrates the microstructures of US alloy after various heat treatments. The HT1 sample shows no obvious difference from the as-cast condition, consisting of lamellar colonies with ε_1 precipitates at grain boundaries. In the HT2 sample, β(B2) segregations within lamellae begin to decompose, transforming into linearly distributed β(B2)+silicide. These precipitates are defined as ε_2, representing the second type of silicide precipitation. The HT3 sample shows silicides dispersed along lamellae, designated as ε_3. After annealing the HT3 sample for 24 h (HT4 sample), silicides within the lamellar colonies exhibit a network distribution, and lamellae grow interpenetratingly.

TEM Analysis of Precipitates

[Figure 3: see original paper] shows TEM images and selected-area electron diffraction (SAED) patterns of precipitates in the HT3 sample. The ε_2 phase is distributed linearly within lamellae, corresponding to the location of the network β(B2) segregations present in the as-cast alloy, suggesting that the β(B2) segregations decompose during heat treatment to form ε_2 at Si-rich locations. The ε_3 phase is distributed along γ/γ and γ/α_2 lamellar interfaces, being the most numerous and having smaller particle sizes compared to ε_1 and ε_2. The ε_3/γ interface is semi-coherent with periodically arranged interfacial dislocations.

Room-Temperature Tensile Properties

[Figure 4: see original paper] presents the room-temperature tensile properties of as-cast UM and US alloys and various heat-treated US alloy samples. The US alloy with 0.5% Si addition shows significantly improved tensile properties (both strength and elongation) compared to the UM alloy. However, after heat treatment, the room-temperature tensile properties of the US alloy gradually decrease, with the annealed HT4 sample exhibiting the lowest properties.

Discussion

Effect of Si on β(B2) Phase Segregation

[Figure 5: see original paper] shows the quasi-phase diagram of 8Nb-TiAl-based alloys. Niobium is a β(B2) phase stabilizing element, and the addition of 8Nb expands the β(B2) phase region toward higher Al content, increasing room-temperature β(B2) phase retention. Conversely, reducing Nb content in the β(B2) phase shrinks the β(B2) phase region and shifts it toward lower Al content. In high Nb-TiAl alloys with Si addition, ε_1 (Nb_5Si_3) precipitates form in the as-cast microstructure, reducing Nb content in the β(B2) phase and shifting the β(B2) phase region leftward and downward in the phase diagram. This reduction in the β(B2) phase region naturally decreases the room-temperature β(B2) phase content. As shown in , adding 0.5% Si significantly reduces β(B2) phase content. The β(B2) segregations are Nb-rich, providing the material basis for ε_2 precipitation. Therefore, in heat-treated samples HT2 and HT3, the linear β(B2) segregations decompose into linearly arranged ε_2 phase. Since β(B2) is brittle at room temperature and detrimental to tensile properties, its reduction improves alloy performance. Si alloying effectively alleviates β(B2) segregation without causing the grain coarsening and property degradation associated with heat treatment elimination of segregation, thereby significantly improving room-temperature tensile properties.

Effect of Silicide Precipitation on Phase Regions

DSC analysis was performed to investigate the effect of ε phase precipitation on the phase diagram, with results shown in [Figure 6: see original paper]. The DSC curves for UM and US alloys reveal several small exothermic peaks between 1000–1200 °C in the US alloy, representing ε phase precipitation. This explains why no precipitated silicides were observed in the HT1 sample, while HT2 and HT3 samples show significantly increased silicide precipitation. Another notable difference is the appearance of a γ single-phase region between 1280–1298 °C in the US alloy. This occurs because Si is a γ-phase stabilizing element that expands the γ-phase region toward lower Al content, which also explains the massive γ phase formation at colony boundaries in the HT3 sample.

Effect of Silicide Precipitation on Crack Propagation

Examination of the tensile fracture surface of the HT2 sample is shown in [Figure 7: see original paper]. Secondary electron imaging reveals a crack propagating along lamellae near the fracture surface. When the crack enters a second colony with different lamellar orientation, it penetrates through the lamellae and continues propagating along the lamellar interfaces. The dispersed silicides within the colony are distributed along γ/γ and γ/α_2 interfaces and have a semi-coherent relationship with the γ phase. The periodically arranged interfacial dislocations at these interfaces facilitate crack initiation along lamellae. The EBSD image of the fracture cross-section shows that cracks originate at β(B2) phase aggregation sites, confirming the detrimental effect of β(B2) on tensile properties. The ε_3 phase is extensively distributed at the fracture and lamellar γ/γ interfaces because tensile stress near the fracture induces substantial ε_3 precipitation, similar to stress-induced Ti_5Si_3 precipitation during creep deformation in TiAl-based alloys. Since the ε_3/γ interface contains interfacial dislocations, its precipitation accelerates crack initiation and propagation. Consequently, the substantial ε_3 precipitation in heat-treated US alloy samples degrades room-temperature tensile properties.

Effect of Annealing Treatment on Alloy Microstructure

The HT4 sample annealed at 900 °C for 24 h exhibits the lowest room-temperature tensile properties. Its microstructure, shown in [Figure 8: see original paper], reveals massive γ phase formation at colony boundaries with β(B2) and ε_1 phase distributed within the massive γ. This occurs because ε_1 precipitates at lamellar colony boundaries in the as-cast structure stabilize the γ phase during annealing, promoting massive γ formation. This microstructure coarsens grain boundaries and contains brittle β(B2) phase segregation, leading to grain boundary embrittlement. Within the lamellae, ε_2 and ε_3 precipitation generates secondary lamellae that grow at an angle to the original lamellae. TEM observation ([Figure 9: see original paper]) shows these secondary lamellae are coarse γ plates that penetrate the original $\gamma+\alpha_2$ colony, fracturing the initial lamellar structure. Since Si is a γ-phase stabilizing element, the ε_2 and ε_3 precipitates within lamellae promote formation of these secondary γ plates, which increase internal defects and are ineffective at hindering crack propagation.

In summary, annealing treatment causes ε_1 precipitation at grain boundaries, leading to massive $\gamma+\beta$(B2)$+\varepsilon_1$ phases that embrittle grain boundaries, while ε_2 and ε_3 precipitation within lamellae produces secondary γ plates that fracture the initial lamellar structure and increase internal defects. Consequently, the annealed HT4 sample exhibits the lowest room-temperature tensile properties.

Conclusions

(1) Based on formation mechanism and precipitation location, the silicide ε (Nb_5Si_3) phase can be classified into three types: ε_1 distributed at colony boundaries, formed by supersaturated Si precipitation during solidification; ε_2 linearly distributed at whisker-like β(B2) segregation sites, formed by β(B2) phase decomposition after heat treatment; and ε_3 distributed at

lamellar interfaces, formed by silicide precipitation within lamellae during heat treatment.

(2) Si addition effectively reduces β(B2) phase segregation in as-cast microstructures because ε phase formation decreases Nb content, which is a β(B2) phase stabilizing element, thereby shrinking the β(B2) phase region. Reduced β(B2) segregation improves the room-temperature tensile properties of as-cast alloys.

(3) Heat treatment degrades the room-temperature tensile properties of US alloy because precipitated ε_2 and ε_3 phases promote crack initiation and propagation along lamellae. Moreover, stress induces further silicide precipitation, accelerating crack growth.

(4) Si, as a γ-phase stabilizing element, expands the γ-phase region, forming a γ single-phase region between 1280–1300 °C. After annealing, γ phase forms near ε phase. The ε_1 phase leads to massive γ+β(B2)+ε_1 structures at colony boundaries that embrittle grain boundaries, while ε_2 and ε_3 phases promote formation of coarse secondary γ laths that fracture the initial lamellar structure.

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