



Influence of nitrogen on the microstructure and solidification behavior of high Nb containing TiAl alloys



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ABSTRACT

The influence of nitrogen interstitial atoms on the microstructure and solidification behavior of high Nb containing TiAl alloys with the composition of Ti-46Al-8Nb-xN ($x = 0.1, 0.7, 1.4, 2.5$ at.%) is discussed in this work. The α_2 volume fraction increases while the γ volume fraction decreases with the increase of nitrogen content, giving evidences that interstitial nitrogen atoms stabilize α phase and expand the region of α phase. With increasing nitrogen content from $x = 0$ to $x = 2.5$, the number of colonies in per square mm increases from ~6 to ~90 and the corresponding average colony size decreases from ~0.17 to ~0.01 mm². The results give the evidences that the nitrogen suppresses the diffusion of solute Al ahead of the solid-liquid interface and leads to the change of primary phase from β to α during solidification. The influence mechanism of nitrogen on the refinement of lamellar structure and colony size of high Nb-containing TiAl alloys is proposed. Meanwhile, the effect of nitrogen on the temperature of state phase transformation of high Nb-containing TiAl alloys is also addressed in this work.

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1. Introduction

Due to the low density, high specific strength, elastic modulus and oxidation resistance at high temperatures, γ -TiAl-based alloys have attracted considerable attention as strong candidates for high-temperature structural applications in aerospace and automotive industries [1,2]. Considering their excellent performance in the range of 600–850 °C, γ -TiAl alloys can be selected for the hot sections of the engine in the future supersonic aircrafts to overcome the high density with low specific strength of superalloys [3,4]. However, low ductility, poor fracture toughness and other intrinsic properties of intermetallic compounds are still obstacles to prevent γ -TiAl alloys from practical applications [5]. It has been found that alloying technique is an efficient way to improve the performance of γ -TiAl alloys. During past decades, numerous fruitful work has been devoted on the composition design and microstructure control to improve mechanical properties of γ -TiAl alloys [6,7]. The TNM alloy with the composition of Ti-(42–45)Al-(5–10)Nb-(0–0.5)B [8,9] exhibits improved strength properties and oxidation resistance when compare with conventional TiAl alloys. And it is reported that the Ti-48Al-2Cr-2Nb alloy has been successfully used in the GENx-1B engine [6]. Furthermore, it is necessary to note that the improved oxidation resistance, creep strength and tensile strength at the elevated temperature have been obtained in high Nb containing TiAl alloys [10,11]. The excellent properties make high Nb containing TiAl

alloys be one of the promising development directions of future γ -TiAl alloys.

Based on the composition design datum, it has been well established that alloying elements like Nb, Mo, and W show a positive effect on mechanical properties of TiAl based alloys [12]. It has been reported that, in addition to the grain refinement effect, boron adding is an efficient way to improve the room temperature tensile ductility, thermal stability of lamellar structure and accordingly prolong the creep rupture life of TiAl alloys [13,14]. Carbon atoms in solid solution increase the micro-hardness, yield strength and decrease the creep rate of γ -TiAl. And carbide precipitates can impede the colony growth of γ -TiAl [15,16]. The hardness of oxygen-doped γ -TiAl alloy is higher than the undoped one [17]. In addition, the increase of the oxygen content changes the solidification path of the binary TiAl alloys [18]. Investigations of oxygen on the high Nb containing TiAl-based alloys give evidences that the increasing oxygen content remarkably increases the amount of α_2 phase and expands the range of peritectic reaction $\alpha + L \rightarrow \gamma$ [19].

Interstitial atoms B, C, O have been proven to notably affect microstructure and correspondingly improve mechanical properties of γ -TiAl alloys. Meanwhile, it is worth to note that interstitial elements are always impossible to be completely avoided during the melting, casting and forging process. It is necessary to illustrate the role of the interstitial elements on the solidification behavior and mechanical properties of TiAl alloys. As a main component of the atmosphere, the effect of nitrogen cannot be ignored. It has been reported that nitrogen preserves a significant effect on microstructure refinement and increases the micro-hardness. And precipitation of Ti₂AlN increases the yield stress and contributes to the decrease of primary creep strain of γ -TiAl alloys [20]. The

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nitride layer formed by implantation has been proven to improve the oxidation resistance of γ -TiAl alloys [21]. The work of Nam et al. shows that the nitrogen causes a reduction in the mean lamellar thickness of γ -TiAl alloy [22]. The influence of nitrogen on mechanical properties of TiAl alloys has been well proposed in previous research. The work about the effect of nitrogen on solidification behavior and solid state phase transition of γ -TiAl alloys is still limited in literature. This work focuses on the influence of nitrogen on the solidification behavior and phase transformation of a high Nb containing TiAl alloy considering their improved strength properties and oxidation resistance at the elevated temperature. Meanwhile, the effect of nitrogen on the temperature of phase transformation is also discussed to illustrate the corresponding mechanism of nitrogen on the microstructure formation of TiAl alloys. The obtains of this work are believed to shed light on understanding the role of nitrogen atoms on the solidification behavior of high Nb containing TiAl alloys. It can also be expected to provide fundamental theoretical foundation and technical guidance for the composition design and microstructure control of TiAl alloys with refractory elements for structural applications at high temperature.

2. Experimental procedure

The nominal chemical composition of experimental alloys are Ti-46Al-8Nb- x N ($x = 0, 0.1, 0.7, 1.4, 2.5$). Unless otherwise noted, alloy contents are all shown in at.%. Each ingot was prepared in a vacuum arc-melting furnace under the protection of argon atmosphere using a non-consumable tungsten electrode. The raw materials used in this study were commercial purity titanium (99.97 wt.%), aluminum (99.97 wt.%) and Ti-Nb binary alloy (with Nb content of 52.47 wt.%). Nitrogen was introduced in the form of TiN (99.7 wt%) powder. Each ingot was re-melted 5 times to ensure the homogeneity and vertically cut along the symmetry axis using an electric discharge machine. Weight of each ingot is ~ 30 g. The alloy composition is considered to be the designed one as the weight loss of each ingot is less than 0.1 wt.%.

The phase composition of the samples were determined by X-ray diffraction (XRD) with Cu K α radiation ($\lambda = 1.54056 \text{ \AA}$). And phase fractions were determined by Rietveld analysis of XRD patterns using the easy quantitative calculation, which is based on the RIR ratio method [23]. The R value of each sample is below 5%. After multiple measurements, the RSD (relative standard deviation) was used to define the measuring error of each phase. The RSD results show that the each measuring error is less than 1%. Scanning electron microscopy (SEM) (VEGA-3 TESCAN) were used to examine the microstructure. Meanwhile, microstructure and nitride precipitation have been characterized using a transmission electron microscope (TEM) (FEI Tecnai G2 F30). Samples SEM observation were prepared by standard metallographic polishing procedures, and a modified Kroll's etchant consisting of 80 ml H₂O, 10 ml HNO₃ and 10 ml HF was used for etching. TEM foils were prepared by the ion milling method. DSC (Netzsch STA 449C) was used to investigate the solid-state phase transformation temperature of experimental alloys. And the experiments were conducted in a corundum crucible with a dynamic argon atmosphere. Dynamical DSC experiments were carried out at heating rates of 5 K/min from 900 to 1450 °C.

3. Results and discussion

3.1. Phase composition

Fig. 1(a) shows the X-ray diffraction patterns of Ti-46Al-8Nb- x N ($x = 0, 0.1, 0.7, 1.4, 2.5$). With $x = 0.1$ and 0.7, only two main phases α_2 and γ are observed in the Ti-46Al-8Nb- x N alloys. With the report of the solid solution position of carbons [24,25], it can be inferred that the solute state nitrogen atoms mainly occupy in different types of octahedral sites of α_2 and γ phases. With the further increase of nitrogen content to $x = 1.4$, Bragg peaks of a precipitated phase Ti₂AlN in the XRD pattern are observed. As nitrogen content reaches as high as $x = 2.5$,

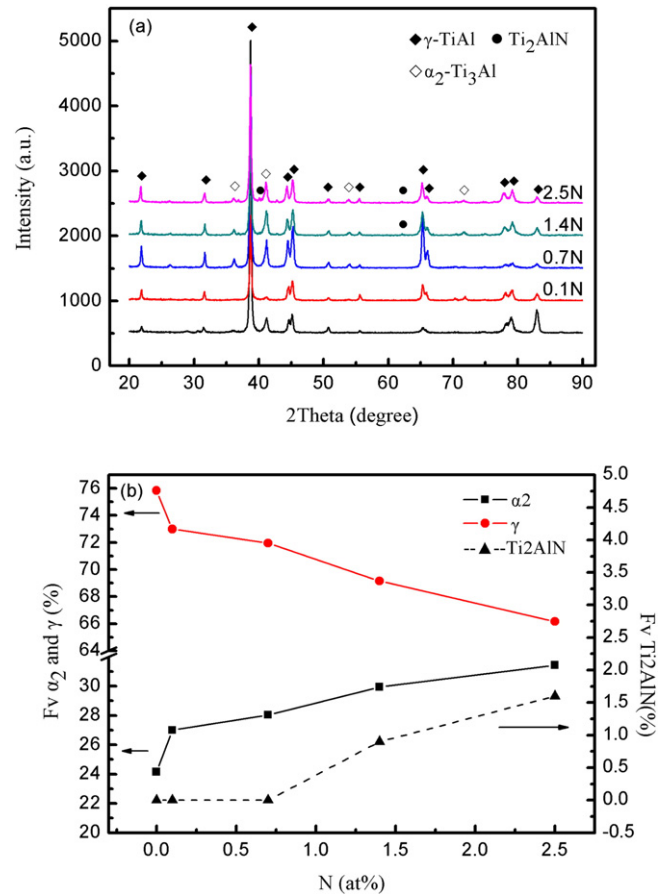


Fig. 1. (a) X-ray diffraction patterns of Ti-46Al-8Nb- x N ($x = 0, 0.1, 0.7, 1.4, 2.5$ at.%); (b) variation of α_2 , γ and Ti₂AlN volume fraction as a function of the nitrogen content, respectively.

Bragg diffraction peak intensities of Ti₂AlN are higher comparing with those of the alloy with $x = 1.4$, which indicates the increasing content of the corresponding phase. Fig. 1(b) shows the variation of α_2 , γ and Ti₂AlN volume fractions as a function of nitrogen content. As the nitrogen content increases from 0 to 0.7, the α_2 volume fraction increases from ~ 24.2 to $\sim 28.1\%$. Since α_2 is derived from α through solid state phase transition, according to the TiAl phase diagram [27], the increase of α_2 gives evidences that the expansion of α phase region. With the further increase of nitrogen content, precipitated phase Ti₂AlN with volume fraction of ~ 0.9 wt % can be observed in the alloy with $x = 1.4$. It indicates that the nitrogen content at 1.4 exceeds its solid solubility in the experimental TiAl alloy.

The evolutions of lattice parameters and cell volume of γ and α_2 phases as a function of the nitrogen content are shown in Fig. 2. With the increase of nitrogen content, the lattice parameters a and c of γ phase as shown in Fig. 2(a) preserve no significant variation except for the alloy with $x = 0.1$. As shown in Fig. 2(a), the solid solution of nitrogen in γ phase is below 0.1% due to the fact that lattice parameters and cell volume remain almost constant for the alloys with nitrogen content above 0.1%. For the alloy with 0.1% nitrogen, the nitrogen atoms are believed to be completely in solid solution in γ phase. Considering the fact that the solid solution of nitrogen in γ is much lower than that in α_2 phases, it can be regarded that the solid solution of nitrogen in γ phase has reached the saturation and resulted the significant increase of lattice parameters of γ phase from $x = 0$ to $x = 0.1$ as shown in Fig. 2(a). In this work, the variation of the lattice parameters of γ phase indicates that the solubility limit of nitrogen in γ phase is below 0.1%. The increased solubility limit of nitrogen in γ phase comparing with those of oxygen [25] and carbon [15] can be ascribed to the

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