Influence of heat treatment on the precipitation of $\gamma_1$ phase in high Nb containing TiAl-based intermetallic alloys

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Abstract

A new intermetallic compound $\gamma_1$ (Ti$_4$Nb$_3$Al$_9$) phase can be observed with the increment of Nb content in the Ti-Al-Nb ternary system. This work deals with the influence of heat treatment on the precipitation of $\gamma_1$ phase in high Nb containing TiAl-based intermetallic alloys. The experiment results showed that 1400 $^\circ$C was acceptable as the homogenization temperature for the super-high Nb containing TiAl-based alloys. The new $\gamma_1$ phase precipitated from the $\gamma$ phase matrix during the following furnace cooling process. TEM and XRD tests were applied to identify $\gamma_1$ phase. The experimental results revealed that $\gamma_1$ phase could precipitate from the Ti-48Al-10Nb alloy after exposure at 1200$^\circ$C for 100-240h.

Keywords: intermetallics; precipitation; heat treatment
1. Introduction

TiAl alloys are a serious candidate material for applications demanding low density, good corrosion resistance and high strength at elevated temperatures. The γ-TiAl-based alloys are being developed for aero-engine applications and for automotive applications. The continuous demand for weight reduction and higher engine efficiencies pushes the materials applied today towards their limits. Therefore, these industries have a strong need for developing novel light-weight materials. [1-5]

Chen and Hao et al. [6–8] then published isothermal sections at 1400, 1150 and 1000℃ of the Ti–Nb–Al phase diagram, and proved a new intermetallic compound named γ₁ with a tetragonal structure. The γ₁-Ti₄Nb₃Al₉ phase is tetragonal with lattice parameter a in the range 0.558-0.584 nm and c in the range 0.815-0.845 nm. Its space group is P4/mmm. Continuous ordering occurs in the TiAl+Nb system with increasing Nb contents. Nb atom in TiAl is proved to substitute for Ti atom and preferentially occupies the Ti sublattice using ALCHEMI technique.[9] With increasing Nb content the distribution of Nb atoms on the sublattice become ordered. The development of the continuous ordering finally leads to the formation of a new ternary compound. Although high Nb content is helpful to form γ₁ phase, but Nb is very hard to distribute in the alloy. The homogenization heat treatment for extra high Nb content has not been investigated up to now. Thus, the present study was taken to study the influence of heat treatment on the precipitation of γ₁ Phase.

2. Experiment Procedures

The cast alloys Ti-56.25Al-18.75Nb and Ti-48Al-10Nb were chosen for the present research. The specimen were produced with sponge titanium Ti, pure Al and Nb-Al interalloy (containing Nb 46 at%) by non-consumable arc melting in a purified argon atmosphere. The ingot was 30g weight and 20mm in diameter. The as-cast samples were cut by the Wire Cut Electrical Discharge Machine and enveloped in high vacuum quartz sleeve.

The alloy Ti-56.25Al-18.75Nb was heat treated at 1300 and 1400℃ for about 4-15h with different cooling rate following. The alloy Ti-48Al-10Nb was treated at 1200℃ for 100-240h. Both of the heat treatment processes were taken in the single γ phase to provide a favorable condition for the precipitation of γ₁ phase from γ matrix. X-ray diffraction (XRD) technique was used to identify the γ₁ phase. Transmission Electron Microscope (TEM), Electron Probe Micro-analyzer (EPMA) and Scanning Electron Microscope (SEM) were taken to observe the appearance of the alloys.

3. Results and Discussion

3.1. Heat treatment of Ti-56.25Al-18.75Nb
Ti-56.25Al-18.75Nb is the nominal composition of γ₁ phase (Ti₄Al₃Nb₉). Its as-cast image is showed in Fig. 1(a). Obviously the as-cast microstructure of nominal composition of γ₁ phase is dendrite structure. In the solidification process, constituent Al, as the solute, its distribution coefficient is smaller than 1. When the β phase forms from the liquid Al is discharged to the liquid. While Ti and Nb, which can stabilize the β phase, concentrate in the dentrite. The interdendritic liquid containing more Al transform to γ phase. As the Al segregation and Nb segregation exist in the as-cast alloys. So the homogenization heat treatment is necessary.

Fig. 1(b) shows the microstructure of alloy 56.25-18.75 after annealing at 1300 ℃ for 4h. the result indicates that 1300 ℃ could not eliminate the cast segregation at all. The result of 1400 ℃ for 15h is exhibited in Fig. 1(c). The alloy is homogenized efficiently. So annealing at 1400 ℃ for 15h is suggested for the homogenization heat treatment.

Based on the homogenization, water quench and furnace cooling were applied. The results are shown in Fig. 1(c) and (d). Although the composition is homogenized completely, there was not any new phase emerging. On the other hand, a new phase with needle-like morphology precipitates from the γ-matrix after the furnace cooling. The result shows that for the nominal composition of γ₁ phase Ti-56.25Al-18.75Nb the homogenization temperature of 1400 ℃ is appropriate. The new phase emerged during the slow cooling process which indicates that its precipitation is a second-order phase transition process.

3.2. The identification of needle-like γ₁ phase
The TEM test was applied to the alloy which could be found needle-like precipitation to identify the new phase. TEM micrograph shows that the microstructure of the new emerging needle-like phase is very fine. Its thickness is less than 100 nm. As the addition of high Nb containing, which increase the lattice distortion, induce the brittleness, the samples break easily and cannot be grinded to less than 100μm. As a result the thin district could be observed is very small. The district with needle-like phase is comparatively thick which make it difficult to obtain the electron diffraction patterns. The figure at the top left corner show the electron diffraction patterns of the matrix. It is identified as γ phase.

In order to identify the needle-like phase XRD examination was taken in detail. The chemical formula of γ1 phase is determined to be Ti₄Nb₃Al₉ as the result of ordered occupation of Nb atom in γ lattice. The basic diffraction lines that exhibit strong intensity for both γ and γ₁ phase are identical. The critical diffraction lines for identifying γ₁ phase, which are absent from the corresponding γ phase, are caused by superlattice diffraction.[10,11] Although many of them are relatively weak, but some relatively intense lines comparing to the relative intensity of the background, including d(4.6409), d(3.5752), d(2.2636), can be observed.

These critical lines are exhibited in Fig. 3(b) and (c). Meanwhile the XRD result taken from the specimen heat treated at 1400℃ following water quench could not be found these critical diffraction lines. So it can be concluded that the needle-like phase is γ₁.

![TEM BF micrograph of the needle-like phase and EDPs of the matrix](image)

**Fig. 2.** TEM BF micrograph of the needle-like phase and EDPs of the matrix

![XRD pattern of the specimen with needle-like phase (a) XRD pattern (b) d=4.5980 (c) d=3.5765](image)

**Fig. 3.** XRD pattern of the specimen with needle-like phase in. (a) XRD pattern (b) d=4.5980 (c) d=3.5765
3.3. Heat treatment of Ti-48Al-10Nb

Although high Nb and Al contents are favorable to precipitate the needle-like $\gamma_1$ phase, but the specimen with the composition in this area are brittle which make it difficult to continue the following experiment. On this basis Ti-48Al-10Nb alloy was investigated. In order to obtain $\gamma_1$ phase the specimen should be treated in $\gamma$ region. According to the Ti-Al binary phase diagram 1200°C was chosen as the heat treatment parameter.

It can be seen that after annealing at 1200°C for 100h some needle-like phase precipitates from the matrix. The area of this phase is less than 5% which cannot be identified in XRD patterns. Except the matrix and a few $\gamma_1$ phase

There is still another grey-white phase as shown in Fig. 4(a). The XRD result indicates that the grey-white phase is $\alpha_2$. The situation changed when the holding time was extended to 240h. The grey-white phase decreases clearly while the needle-like phase increases. As shown in Fig. 5, compare the XRD patterns for 100h with that for 240h. It can be found that for 100h the characteristic peaks of $\alpha_2$ emerge and the critical lines which could identify $\gamma_1$ phase can not be observed obviously. For 100h these lines are relatively strong and characteristic peaks of $\alpha_2$ disappear. Corresponding to the Fig. 4(a) and (b), it can be concluded that for Ti-48Al-10Nb alloy after long time heat treated in $\gamma$ region the needle-like $\gamma_1$ phase could precipitate from the $\gamma$ matrix.

![Fig. 4. EPMA images of Ti-48Al-10Nb heat treated at 1200°C. (a) 100h (b) 240h](image)

![Fig. 5. X-ray diffraction pattern of the alloy Ti-48Al-10Nb heat treated at 1200°C for 100h and 240h](image)
4. Conclusion

1. For the nominal composition of $\gamma_1$ phase Ti-56.25Al-18.75Nb alloy homogenization heat treatment is suggested being treated at 1400°C for 15h with furnace cooling following.
2. Precipitation process of $\gamma_1$ phase is a second-order phase transition process.
3. Heat treated at 1200°C for more than 200h is recommended for Ti-48Al-10Nb alloy to obtain $\gamma_1$ phase from the $\gamma$ matrix.

References


