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Solidification sequence and phase equilibria in Ti-47Al-22Nb and Ti-47Al-20Nb ternary alloys

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Abstract

The phase transformation sequences and phase equilibria in Ti-47Al-20Nb and Ti-47Al-22Nb (at.%) alloys were investigated through experiments and computational (CALculation of PHAse Diagrams) techniques. Experiments to study nonequilibria phase transformations involved quenching the alloys from high temperature followed by composition and microstructure characterization. The computed results show that with increase in temperature, the \( \alpha + \beta + \gamma \) tie-triangle phase region shifts towards low Nb content. On the contrary, experimental results show that with increase in temperature the tie-triangle of \( \alpha + \beta + \gamma \) moves towards high Nb content. These changes are attributed to the expansion of the \( \alpha \) phase region and corresponding changes of the primary \( \alpha \), \( \beta \) and \( \gamma \) phase regions with increase in temperature. In the experimentally studied alloys, a part of the \( \alpha \) phase transformed to \( \gamma \) phase by massive transformation and most of the \( \beta \) phase transformed to \( \gamma \) phase in high Nb content alloys.

1. Introduction

The Ti-Al-Nb system has promising properties for high-temperature applications, such as high strength, excellent oxidation resistance and high temperature creep resistance [1–4]. There are three classes of alloys in the Ti-Al-Nb system, i.e. \( \gamma \)-TiAl alloys [3, 5], O-Ti2AlNb based alloys [2, 6] and \( \sigma + \gamma \) two-phase alloys [4], designed for service temperatures of 600 °C–900 °C, 500 °C–800 °C and higher than 1000 °C, respectively. The \( \gamma \)-TiAl alloys usually contains \( \alpha_2 \)-Ti3Al, \( \beta \)-Ti and \( \gamma \)-TiAl phases, while the \( \sigma + \gamma \) two-phase alloys contains \( \sigma \)-Nb2Al and \( \gamma \)-TiAl phases. The properties of Ti-Al-Nb alloys strongly depends on alloy composition and microstructure [3]. Since most hot-working and heat treatments are carried out at temperatures above 1300 °C [3], understanding of the phase equilibria of \( \alpha \), \( \gamma \), \( \beta \), and \( \sigma \) phases is essential for the design of alloy compositions and heat treatment processes, especially for temperatures above 1300 °C.

Raghavan constructed the Ti-Al-Nb phase diagram in 2005 [7] and it captured most of the prior experimental results data. These prior experimental studies were mainly focusing on the phase diagram at temperatures below 1300 °C [7–11], and those at temperatures above 1300 °C are still scarce. Among the researches considering temperatures above 1300 °C, Ding et al systematically studied the Ti-Al-Nb phase equilibria at 1400 °C [12–14]. However, their study is questionable because of the short annealing times used and non-recognition of nonequilibrium phase transformations. In 2009, Witusiewicz et al [15] and Cupid et al [16] calculated the Ti-Al-Nb phase diagram using the CALPHAD (CALculation of PHAse Diagrams) method. As we discussed in our prior works [17, 18], the calculated results from Witusiewicz et al and Cupid et al are inconsistent at high temperatures due to an unreliable thermodynamic input parameter adopted from some insufficient experimental data, as shown in figure 1. The calculated results show that the regions and positions of the \( \alpha - \beta - \gamma \) three-phase regions are significantly different. The \( \alpha - \beta - \gamma \) three-phase region from the results of Witusiewicz et al is smaller than that from Cupid et al and it is located at higher Nb content for the former than the latter. Thus, from the results of Witusiewicz et al the \( \alpha \) single-phase region moves towards Nb vertex and shrinks with increasing temperature and the opposite trend occurs in the results of Cupid et al. It should be
Two button ingots weighing 25 g each with nominal compositions of Ti-47Al-22Nb (alloy #1) and Ti-47Al-20Nb (alloy #2) (in at.%) were prepared by non-consumable arc melting (wungsten electrode). The raw materials are high purity Ti (99.99 wt.%) and Nb (99.99 wt.%) metals. To ensure compositional homogeneity, buttons were re-melted ten times with turning in ultra-high purity argon atmosphere. The actual compositions were measured through different techniques (Al: titrimetric method, Ti: ICP-AES, N and O: inert gas fusion infrared and thermal-conductivity method, Nb: the remaining weight) and are shown in table 1. Sample specimens with dimensions of $8 \times 9 \times 8$ mm were cut from the two ingots by electro-discharge machining (EDM). Then the specimens were packed into different evacuated quartz capsules followed by electrolytic polishing and the other half was ground to powder for x-ray diffraction (XRD) experiments. The microstructures of the specimens were then examined using ZEISS SUPRA 55 scanning electron microscope (SEM) with backscattered electron (BSE) mode.

Figure 1. The superposition of isothermal sections [17, 18] calculated by Witusiewicz et al [15] and Cupid et al [16], respectively: (a) 1300 °C; (b) 1400 °C. Part (a) is reprinted from [18], Copyright (2017), with permission from Elsevier. Part (b) is reprinted from [17]. Copyright (2018), with permission from Elsevier.
The working distance and accelerating voltage were 10 mm and 15 kV, respectively. A JXA-8230 electron probe microanalysis (EPMA) was used to study the compositions of the phases in the specimens with a voltage of 20 kV and a beam size of 1 μm. X-ray diffraction (XRD, Bruker D8 Advance, Cu target with a voltage of 40 kV and a current of 40 mA) and electron backscatter diffraction (EBSD) were used to study the phase constituents. Oxford Instruments HKL Channel 5 software was utilized to acquire and index the EBSD patterns.

3. Results and discussion

3.1. Phase transformation sequences based on calculations

We calculated the solidification and solid phase transformation sequences of alloys #1 and #2 using Pandat software and Witusiewicz’s database [15], and the results are shown in figure 2. According to the calculations, the determined phase transformation sequences of alloys #1 and #2 are: L → L + β → L + β + γ → L + γ → γ and L → L + β → L + β + γ → β + γ → γ, respectively. The primary phase in both these alloys is β phase, and then γ phase precipitates from the remaining mixture of liquid and β phase. In order to check the accuracy of the calculation and explore the phase transformation sequences of these alloys in detail, we investigated the as-cast microstructures and heated the alloys to different temperatures and the obtained results are reported in next section.

3.2. Solidification sequence

Figure 3 shows the as-cast microstructures of alloy #1 cut from the bottom of the ingot (figures 3(a), (b)) and the corresponding XRD pattern (figure 3(c)). The XRD result shows that there are two phases in this alloy, i.e. β and γ. Due to the enrichment of Ti and Nb elements in the β phase, the bright and grey phases in the SEM-BSE maps are the β and γ phases, respectively. The as-cast microstructures are similar to those previously obtained for the

| Table 1. The actual compositions of the as-cast ingots. |
|---------------------------------|--------|--------|--------|--------|
| Alloy  | Ti(at.%) | Al(at.%) | Nb(at.%) | O(wt.%) | N(wt.%) |
| #1     | 30.4    | 47.7    | Bal.    | 0.036  | 0.0050  |
| #2     | 33.9    | 46.5    | Bal.    | 0.028  | 0.0038  |

| Table 2. Heat treatments (HT) for the alloy specimens. |
|---------------------------------|--------|--------|
| Alloy   | Heat treatment | Abbreviation |
| #1, #2 | 1400 °C/15 h WQ | HT1 |
| #1, #2 | 1350 °C/61 h WQ | HT2 |
| #1     | 1400 °C/5 h FC to 1300 °C/51 h WQ | HT3 |

* WQ: water quenching; FC: furnace cooling.
Ti-44.5Al-18.5Nb alloy [24]. The bright β-segregation regions indicate that the β phase is the primary phase [25]. The grain boundaries of the primary β phase are highlighted by black dashed lines as shown in figure 3(b). The equiaxed β phase grains indicate that all the liquid solidifies into the β phase. Therefore, the phase transformation of alloy #1 show that all the liquid transforms into β phase and then the β phase transforms into γ phase with a Widmanstatten morphology.

Figure 4 shows the SEM and XRD results of alloy #2. The microstructure shown in figure 4(a) is obtained from the top of the as-cast ingot and includes dendritic arrays. The 90° angle between the secondary arms and the primary dendrite spines indicates that the primary phase has a bcc crystal structure [25] and from the XRD result (figure 4(c)) this shows that it is β phase. The grey γ phase is located in the interdendritic region. By contrast, figure 4(b), cut from the bottom of the as-cast ingot, features a homogeneous microstructure with two equiaxed phases, and is similar to the as-cast microstructure of alloy #1. The equiaxed grains in figure 4(b) show that no other phase directly forms from the liquid. Therefore, alloy #2 should have the same phase transformation sequence as alloy #1, i.e. all the liquid transforms into β phase and then the β phase transforms into γ phase with a Widmanstatten morphology.

Shown in figure 5 is the Ti-Al-Nb liquid projection proposed by Raghavan et al [7], Witusiewicz et al [15] and Cupid et al [16], including the locations of the alloys considered in the present study. The results obtained from this study are in good agreement with the computed results of Cupid et al and Witusiewicz et al but there is no relationship with those from Raghavan et al. However, the results of Witusiewicz et al (see figure 2) also show a L + β + γ three-phase region during the solidification sequence, and the existence of such a region was not confirmed in our present study. It is also important to note that results from Raghavan et al show that the two alloys should be located at the γ primary phase region. This is in opposite with the present experimental results where it is revealed that the primary β phase region should extend to the higher Al content zone.

3.3. Phase equilibria at 1400 °C

Figure 6(a) illustrates the SEM-BSE image of alloy #1 after HT1 heat treatment (see table 2). Figures 6(b) and (c) are the EBSD phase constituent and orientation maps for the same area in figure 6(a). The image can be divided into four regions, delineated and marked by white lines based on microstructures, phase constituents and orientations, as shown in figures 6(a)–(c). Small amounts of β phase exists in the regions A and B as shown in figure 6(b). From the Ti-Al-Nb phase diagrams [15–18], the β phase is stable at higher temperatures when compared to the α and γ phases. Therefore, this shows that the regions A and B should be β phase at 1400 °C and will decompose into α and γ phases during quenching. From the EBSD phase constituent map (figure 6(b)).
most β phase decomposes into γ phase (marked as A), some β phase decomposes into α phase (marked as B), and only a small amount of β phase remains. The morphology (marked as C in figure 6(a)) and random orientation (marked as C in figure 6(c)) of the γ phase show that the γ phase comes from the α phase via massive transformation during quenching [25]. There is a relatively large α grain in region D (see figure 6(c)) and this phase is retained during quenching from high temperature. Figure 6(d) is the magnified image of the area surrounded by the red dashed lines in figure 6(a). This show that some β phase decomposed into lathy α phase by martensitic transformation, and this in agreement with findings from Hu and Jiang for the Ti-44Al-4Nb-4Hf-0.1Si alloy [23]. Figure 6(e) shows the pole figures for the β and α phases in the area marked B in figure 6(a). As expected, the orientation relationship between the lathy α phase and the parent β phase corroborate well with the classical Burgers orientation relationship, i.e., \( \{ [110]_B / \{0001\}_\alpha \), \( \{111\}_B / \{11-20\}_\alpha \). Figure 6(f) is the magnified image of the area surrounded by the blue dashed lines of figure 6(a). We can clearly observe some lathy
\( \gamma \) phase with Widmanstatten and massive morphologies in the region marked A in figures 6(a) and (f). It is difficult to identify the orientation relationship between the \( \gamma \) phase (\( \beta \rightarrow \gamma \)) and the \( \beta \) phase since only a small fraction of the \( \beta \) phase is now remaining. Thus, the present work cannot confirm the detailed \( \beta \rightarrow \gamma \) phase
transformation sequence due to limitations of the available characterization techniques. However, there are two possible transformation sequences: (1) $\beta$ can directly transform into $\gamma$ by martensitic, Widmanstatten and massive transformations. (2) the $\beta$ phase can first transform into $\alpha$ phase by martensitic and Widmanstatten transformations, and then the $\alpha$ phase will subsequently transform into $\gamma$ phase by massive transformation. In summary, the larger $\alpha$ grain and the decomposed $\beta$ region observed here indicates that only $\alpha$ and $\beta$ phases co-exist in alloy #1 at 1400 °C.

The morphology of alloy #2 (see figure 7) after HT1 heat-treatment (see table 2) is similar to that of alloy #1. There are three contrasting regions in quenched alloy #2. The bright is the $\beta$ phase and it decomposes into $\gamma$ phase and a small amount of $\alpha$ phase during water quenching (WQ). The grey phase is retained $\alpha$ phase from high temperature quenching. The dark phase is the $\gamma$ phase formed from high-temperature $\alpha$ phase through massive transformation. Therefore, only $\alpha$ and $\beta$ phases exists in alloy #2 at a temperature of 1400 °C.

Figure 8 shows the positions of the EPMA analysis in alloy #1 after HT1 heat-treatment. Here, it is important to note that points 2–8 are found in the range of the $\beta$ phase which decomposes into $\gamma$ and $\alpha$ phases, while points 10–17 are in the high temperature retained $\alpha$ phase range, and points 17–25 are in the massive $\gamma$ phase region transformed from high-temperature $\alpha$ phase. Then, the remaining points are located at the boundaries of different phases. No significant difference in composition is demonstrated between the retained $\alpha$ and the massively transformed $\gamma$ phases and this shows that $\alpha \rightarrow \gamma$ massive transformation occurs during quenching. The compositions of $\alpha$ and $\beta$ phases can be obtained by averaging compositions of the points of the same phase in both alloys #1 and #2. Table 3 shows the compositions of $\alpha$ and $\beta$ phases in the quenched alloys.

3.4. Phase equilibria at 1350 °C and 1300 °C
There are two contrasting regions in the quenched alloy #1 after HT2 heat-treatment as shown in figure 9(a). The XRD result shows that $\beta$, $\gamma$ and $\alpha$ (a little) phases exist, as shown in figure 9(b). The $\alpha$ phase comes from the decomposition of the $\beta$ phase (see the $\beta$ grain boundary in the inset of figure 9(a)) during quenching. Therefore,
there are two phases ($\beta$ and $\gamma$) in alloy #1 at a temperature of 1350 °C and during quenching a small fraction of $\beta$ phase transforms to $\alpha$ phase. There are four contrasting regions in alloy #2 after HT2 heat-treatment as shown in figure 9(c). Similar with observations made in section 3.3 for alloys quenched from 1400 °C, the $\beta$ phase in this case also decomposes during quenching. The XRD result in figure 9(d) shows that three phases (including $\alpha$, $\beta$ and $\gamma$) exist in alloy #2. The diffraction peaks of (200) and (002) lattice planes for $\gamma$ phase overlap, indicating that a part of $\gamma$ phase is transformed from $\alpha$ phase in the process of quenching via massive transformation [25]. The clearly distinguishable light grey and dark grey phases are, respectively, $\alpha$ and $\gamma$ phases retained from high temperature. Therefore, there are three phases ($\alpha$, $\beta$ and $\gamma$) at 1350 °C and some $\alpha$ and $\beta$ phases decomposes during quenching. Included in table 3 also are the equilibrium compositions of the phases for the quenched alloys based on EPMA results.

Figure 10(a) shows the SEM-BSE images of alloy #1 after HT3 heat-treatment. From the XRD result (see figure 10(b)), three phases co-exist in this alloy and are the grey $\beta$, bright $\sigma$ and dark $\gamma$ phases. Table 3 also

**Table 3. Phase compositions of the specimens quenched from different temperatures.**

| Temperature(°C) | Alloy | Phase | Composition (at.%) |
|----------------|-------|-------|-------------------|
|                |       |       | Al   | Ti   | Nb   |
| 1400           | #1    | $\beta$ | 47.19 | 30.16 | 22.65 |
|                |       | $\alpha$ | 49.56 | 29.90 | 20.54 |
|                | #2    | $\beta$ | 44.16 | 34.06 | 21.78 |
|                |       | $\alpha$ | 47.03 | 33.82 | 19.15 |
| 1350           | #1    | $\beta$ | 44.95 | 31.75 | 23.92 |
|                |       | $\gamma$ | 50.46 | 29.47 | 20.07 |
|                | #2    | $\beta$ | 43.72 | 33.99 | 22.29 |
|                |       | $\alpha$ | 46.48 | 34.24 | 19.28 |
| 1300           | #1    | $\beta$ | 42.24 | 33.85 | 23.91 |
|                |       | $\gamma$ | 49.24 | 30.93 | 19.83 |
|                |       | $\sigma$ | 39.38 | 24.21 | 36.41 |

**Figure 9.** SEM images (a) and XRD result (b) of the alloy #1 after HT2, together with SEM images (c) and XRD result (d) of alloy #2 after HT2 heat-treatment.
includes the equilibrium compositions of the phases in the quenched alloy #1 measured by EPMA. These compositions are in good agreement with our previous result [18].

3.5. Phase transformation between 1300 °C and 1400 °C

Figure 11(a) shows the phase diagram calculated for temperatures from 1300 °C to 1400 °C using Pandat software and the database from Witusiewicz et al [15]. The α + β tie-line, β + γ tie-line, α + β + γ tie-triangle and σ + β + γ tie-triangle drawn in figure 11(b) are based on the EPMA results listed in table 3. The α + β + γ tie-triangle at 1300 °C from our previous work [25] is also included in figure 11(b). The Nb content of the α + β + γ tie-triangle (at 1400 °C) should be higher than those of the α + β tie lines. Therefore, the α + β + γ tie-triangle should be located in zones with Nb contents higher than 22%, i.e. more than the calculated result having about 14% Nb content (see figure 11). With increase in temperature from 1300 °C to 1400 °C, the α + β + γ tie-triangle moves towards the Nb vertex (figure 11(b)) and this is in contrast with the calculated result where the α + β + γ tie-triangle moves away from the Nb vertex (figure 11(a)). As a result, the experimental study show that the α phase region expand with increase in temperature, i.e. the boundary of α phase region expand from 19% Nb at 1350 °C to over 22% Nb at 1400 °C. In contrast, calculation results show that shrinkage from 15% Nb at 1350 °C to 14% Nb occurred at 1400 °C. The changes of the α phase region and α + β + γ tie-triangle location implies there is a change of liquidus projection especially the primary α, β and γ phase regions as well as the invariant reaction.

From the experimental results the sequence of phase transformations in alloy #1 from 1400 °C to 1300 °C can be determined to be: α + β(1400 °C) → α + β + γ → β + γ(1350 °C) → σ + β + γ(1300 °C). Although no α + β + γ ternary phase equilibrium was observed in alloy #1 in the present study, based on the phase rule there should be a α + β + γ three-phase equilibrium region between the α + β and β + γ two-
phase regions. Furthermore, the sequence of phase transformations in alloy #2 from 1400 °C to 1300 °C can be determined to be: \( \alpha + \beta (1400 \, ^\circ \text{C}) \rightarrow \alpha + \beta + \gamma (1350 \, ^\circ \text{C}) \rightarrow \beta + \gamma (1300 \, ^\circ \text{C}) \). In both alloys, the fraction of \( \alpha \) phase reduces whilst the \( \gamma \) phase rapidly increases with a decrease in temperature. Therefore, we can conclude that with an increase in Nb content in these alloys the Gibbs free energy of the \( \gamma \) phase becomes lower than that of the \( \alpha \) phase at the decomposing temperature of \( \beta \) phase during quenching. From the phase evolution \((\beta \rightarrow \alpha + \beta \rightarrow \alpha + \gamma)\) in Ti-44Al-8Nb alloy reported by Cheng et al [28], it was found that for low Nb content alloys the Gibbs free energy of the \( \gamma \) phase is higher than that of the \( \alpha \) phase at the decomposing temperature of \( \beta \) phase during quenching. Thus, it can be concluded that during quenching the \( \beta \) phase transforms into \( \gamma \) phase for alloys with high Nb content and transforms into \( \alpha \) phase for alloys with low Nb content.

4. Conclusions

In this work, the as-cast and heat-treated microstructures of Ti-47Al-20Nb and Ti-47Al-22Nb alloys were comprehensively studied through experiments and calculations. The experiments included arc melting, quenching and characterization using SEM-BSE, EPMA, XRD and EBSD methods, whilst calculations were performed with the aid of CALPHAD based software. Through systematic analysis of the computed results and experimental phase equilibria at high temperatures, solidification sequences and nonequilibrium phase transformations of these alloys were determined. The main conclusions drawn from this work can be summarized as follows.

1. For alloys with high Nb content considered most of \( \beta \) phase transforms to \( \gamma \) phase during quenching and this is different from the martensitic transformation \((\beta \rightarrow \alpha)\) observed in alloys with low Nb contents.

2. Experiments show that as the temperature changes from liquid to 1300 °C, the Ti-47Al-22Nb alloy undergoes the following stages: liquid \( \rightarrow \) liquid + \( \beta \) \( \rightarrow \) \( \beta \rightarrow \alpha + \beta \) \((1400 \, ^\circ \text{C})\) \( \rightarrow \alpha + \beta + \gamma \) \( \rightarrow \beta + \gamma \) \((1350 \, ^\circ \text{C})\) \( \rightarrow \alpha + \beta + \gamma \) \((1300 \, ^\circ \text{C})\) \( \rightarrow \alpha + \beta + \gamma \) \((1300 \, ^\circ \text{C})\) \( \rightarrow \beta + \gamma \) transforms. In contrast computation results show the following sequence stages: Ti-47Al-20Nb: L \( \rightarrow \) L + \( \beta \) \( \rightarrow \) L + \( \beta \) \( \rightarrow \) L + \( \beta \) \( \rightarrow \beta + \gamma \) \( \rightarrow \gamma \), and Ti-47Al-22Nb: L \( \rightarrow \) L + \( \beta \) \( \rightarrow \beta + \gamma \) \( \rightarrow \gamma \). In both alloys, the fraction of \( \alpha \) phase reduces and the liquidus surfaces corresponding to the primary \( \alpha \), \( \beta \) and \( \gamma \) phase regions are altered.

3. With increase in temperature, the tie-triangle of \( \alpha + \beta + \gamma \) move towards high Nb content and as a result the \( \alpha \) phase region expand and the liquidus surfaces corresponding to the primary \( \alpha \), \( \beta \) and \( \gamma \) phase regions are altered.

4. The apparent differences between the experimental and calculated phase diagrams at high temperatures is mainly attributed to the unreliable database adopted from literature (due to inadequate experimental results) and the present work will bridge the gap through availing some of the missing database.

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