Microstructure evolution and its effect on mechanical properties of cast Ti48Al6NbxSi alloys

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Abstract: In order to improve mechanical properties of TiAlNb alloys, different contents of silicon were added into Ti48Al6Nb alloy. The Ti48Al6NbxSi ($x=0, 0.1, 0.2, 0.3, 0.4$ and 0.5 , at.%) alloys were prepared by vacuum arc melting. The phase constitution, microstructure evolution and mechanical properties of the alloys were studied. Results show that the Ti48Al6NbxSi alloys consist of γ -TiAl phase, α_2 -Ti₃Al phase and B2 phase, and Ti₅Si₃ silicide phase is formed when the addition of silicon is higher than 0.3at.%. The addition of silicon leads to the decrease in γ phase and increase in α_2 phase. The formation of silicide decreases the amount of Nb dissolved in the TiAl matrix, and therefore decreases B2 phase. Compressive tests show that the ultimate strength of the alloys increases from 2,063 MPa to 2,281 MPa with an increase in silicon from 0 to 0.5at.%, while the fracture strain decreases from 34.7% to 30.8%. The increase of compressive strength and decrease of fracture strain can be attributed to the decrease of B2 phase and the formation of Ti₅Si₃ phase by the addition of silicon. The strengthening mechanism is changed from solid solution strengthening when the addition of silicon is less than 0.3at.% to combination of solid solution strengthening and secondary phase strengthening when the addition of silicon is higher than 0.3at.%.

Key words: TiAl-based alloy; microstructure; mechanical property; strengthening

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The γ -TiAl based alloys have been widely used in aeronautic and aerospace engineering for its low density, high melting temperature and high specific strength^[1-3]. They are considered as candidate materials of turbine blades to replace the Ni-based high temperature alloys due to their good creep and oxidation resistance at elevated temperature^[4-5]. However, the poor ductility and low fracture toughness of the γ -TiAl based alloys at room temperature limit their further application^[6-8].

Many research works show that addition of Nb can improve the room temperature plasticity, liquidus and high temperature oxidation behavior of TiAl alloys^[8-9]. The high-Nb TiAl alloys can maintain a metastable B2 phase because Nb is a β -stabilizing element^[10-13]. B2 phase with a body centered cubic lattice structure is softer than γ (TiAl) and α_2 (Ti₃Al) phases in the TiAl alloys, can serve as the grain-refinement and plastic-enhancement component at elevated temperature^[3, 14-15]. Nb can increase the high temperature strength, ductility, and toughness of TiAl alloys. The service temperature of high-Nb TiAl alloys can exceed 800 °C^[16-17].

Silicon is one of the most important alloying elements of TiAl alloys^[16, 18-20]. Ti₅Si₃ phases are formed during the solidification process of TiAl alloys when the addition of silicon reaches a certain level. The Ti₅Si₃ phases, which are considered as thermodynamically stable reinforce particles, show very strong interfacial bonding with the TiAl matrix and promote the oxidation resistance of TiAl alloys^[16, 21]. The Ti₅Si₃ phase has a high hardness (about 11.3 GPa)^[22], and can act as the strengthening phase in the TiAl alloys, preventing the high temperature deformation of TiAl alloys, and thus improving the plasticity and high temperature strength^[23-24].

High-Nb TiAl alloy, Ti48Al6Nb (at.%) with different addition amounts of silicon was prepared in the present study. The effects of silicon on the microstructure evolution, phase constitution, and mechanical properties of the high-Nb TiAl alloy ingots were studied.

1 Experimental

The Ti48Al6Nb x Si (at.%, $x=0, 0.1, 0.2, 0.3, 0.4$ and 0.5) TiAl-based alloy ingots with different silicon additions were melted in a vacuum arc melting furnace using tungsten electrode. The raw materials were sponge titanium, particle silicon, ingot aluminum and Al-70wt.%Nb intermediate alloy. The actual weight of aluminum is 1.04 times of the calculated one for it was easily burned and volatilized. The ingots were melted five times in the argon atmosphere to ensure the composition homogeneity.

Each solidified ingot was cut into two halves from the center by electrical-discharge wire cutting machine (DK7732). Three columnar specimens with dimensions of $\Phi 4$ mm $\times$ 6 mm were cut from one half of the ingot. A specimen with thickness of 8 mm was cut from near the center of the other half for microstructure observation. The specimens for microstructure observation were ground and mechanically polished, and then etched with standard Kroll's reagent of 3 ml HF, 10 ml HNO₃ and 100 ml H₂O for about 10 s. The phase constitution was identified by an X-ray diffractometer (XRD, Panalytical, Empyrean Cu K α , 20 Kv, 8°·min⁻¹), and the 2θ scattering range of 20° to 90°. Scanning electron microscopy (SEM, JEOL, JXA-8230) was used to study the microstructure employing backscatter electron (BSE) mode. Energy dispersion spectrum (EDS) was applied to measure the elemental composition of different phases. The volume fraction of different phases was measured using an image analyzer. Compression tests of columnar specimens were performed using a computer controlled Instron 5569 mechanical testing machine. At least 3 specimens were measured for each alloy with an compression rate of 0.5 mm·min⁻¹.

2 Results and discussion

2.1 Phase analysis

Figure 1 shows the XRD patterns of Ti48Al6Nb alloy with different additions of silicon. It can be seen that the alloys are composed of γ -TiAl phase, α_2 -Ti₃Al phase and B2 phase, and no obvious Ti₅Si₃ diffraction peaks are observed after adding silicon. The intensity of diffraction peaks for γ phase is the highest [Fig. 2(a)] for the Ti48Al6Nb alloy with no Si addition, showing that the addition of silicon will reduce the amount of γ phase. The intensity of diffraction peaks for α_2 phase is the lowest [Fig. 2(b)] for the alloy with no Si, indicating that the amount of α_2 phase is increased by addition of silicon.

Silicon is typical α phase stable element which can reduce the zone of β phase in the TiAl binary phase diagram^[20]. The aluminum equivalent of silicon in TiAl alloys is -2.8. Therefore, TiAl alloys with addition of silicon will move to

Fig. 1: XRD patterns of Ti48Al6Nb x Si alloys: (a) $x=0$; (b) $x=0.1$; (c) $x=0.2$; (d) $x=0.3$; (e) $x=0.4$; (f) $x=0.5$

the lower aluminum content parts of the TiAl binary phase diagram. Thus, the amount of γ phase decreases and α_2 phase increases by adding silicon to the Ti48Al6Nb alloy according to the XRD results. In addition, the solubility of silicon in the TiAl alloys is low, for it is an interstitial element in the alloys. Furthermore, the mixing enthalpy of silicon-titanium is very low (-66 kJ·mol⁻¹), thus, it is easy for silicon to form intermetallics in the TiAl alloys with higher content of silicon. However, the silicide intermetallics are not observed in the present alloys, which is possibly attributed to their tiny amount.

2.2 Microstructure

Figure 3 shows the microstructures of Ti48Al6Nb x Si alloys with different additions of silicon. It can be seen that the morphology of Ti48Al6Nb x Si alloys changes from coarse columnar grains to fine equiaxed grains when the addition of silicon increases from 0 to 0.5at.%. The primary and secondary dendrite lengths of Ti48Al6Nb x Si alloys are summarized in Table 1. The Ti48Al6Nb alloy without addition of silicon shows coarse columnar grains, and the bright B2 phase forms in the center of the dendrite structures, as shown in Fig. 3(a). The primary and secondary dendrite lengths of Ti48Al6Nb are 246 μ m and 152 μ m, respectively. The grains evolve to refined columnar grains when the addition of silicon is 0.1at.%, as shown in Fig. 3(b), and the primary and secondary dendrite lengths of Ti48Al6Nb0.1Si are decreased to 178 μ m and 108 μ m, respectively. When the addition of silicon is increased to 0.2at.%, the primary and secondary dendrite lengths of Ti48Al6Nb0.2Si are further

Fig. 2: Diffraction peaks of γ -TiAl phase and α_2 -Ti₃Al phase: (a) γ -TiAl phase; (b) α_2 -Ti₃Al phase

Fig. 3: Microstructure of Ti48Al6Nb_xSi alloys: (a) $x=0$; (b) $x=0.1$; (c) $x=0.2$; (d) $x=0.3$; (e) $x=0.4$; (f) $x=0.5$

Table 1: Dendrite length and volume fraction of B2 and Ti₅Si₃ for Ti48Al6Nb_xSi alloys

Si content (at.%)	Primary dendrite length (μm)	Secondary dendrite length (μm)	B2 (vol.%)	Ti ₅ Si ₃ (vol.%)
0	246	152	2.7	0
0.1	178	108	2.5	0
0.2	149	62	2.6	0
0.3	132	50	2.3	1.2
0.4	100	40	2.2	1.6
0.5	60	32	1.9	2.1

refined to 149 μm and 62 μm, as shown in Fig. 3(c). When the addition of silicon increases to 0.3at.%, Ti₅Si₃ silicide phase particles form at the grain boundaries [Fig. 3(d)], and the grains of Ti48Al6Nb0.3Si are further refined. When the addition of silicon increases to 0.4at.% and 0.5at.%, the dendrite structure of the alloys changes to fine equiaxed grains

[Figs. 3(d) and (f)]. The primary and secondary dendrite lengths of Ti48Al6Nb0.5Si are decreased to 60 μm and 32 μm.

The volume fractions of B2 phase and Ti₅Si₃ silicide phase of Ti48Al6Nb_xSi alloys are calculated by counting the pixel area of Photoshop software, and the results are listed in Table 1. It shows that the volume fraction of B2 phase for the

Ti48Al6Nb x Si alloys has an obvious decreasing trend when the addition of silicon increases from 0 to 0.5at.%. The Ti₅Si₃ silicide phase forms when the content of silicon is no less than 0.3at.%. The volume fraction of Ti₅Si₃ silicide phase for Ti48Al6Nb0.3Si alloy is about 1.2%, and the volume fraction of Ti₅Si₃ silicide phase for Ti48Al6Nb0.4Si alloy is increased to 1.6%. When the content of silicon is increased to 0.5at.%, the volume fraction of Ti₅Si₃ silicide phase is increased to 2.1%. Meanwhile the volume fraction of B2 phase is slightly decreased to about 1.9%.

Figure 4 shows the microstructures of Ti48Al6Nb x Si alloys with a higher magnification. Different phases of the alloys are labeled with different marks. The elemental compositions of the bright B2 phase (marked with A), the grey γ phase area (marked with B), the matrix α_2/γ colony (marked with C) and the silicide phase (marked with D) are summarized in Table 2. It shows that the bright B2 phase is rich in Nb and poor in Al, while the grey area

is rich in Al and poor in Nb for all the Ti48Al6Nb x Si alloys. There is no obvious change in the content of Nb dissolved in the α_2/γ colonies of Ti48Al6Nb x Si alloys. The Nb in B2 phase for Ti48Al6Nb x Si alloys is decreased when increasing the addition of silicon from 0 to 0.5at.%. By adding silicon with 0.1at.% and 0.2at.%, the content of Nb dissolved in B2 becomes less than that with no silicon. At the same time, the content of Nb is increased in the grey area. When the content of silicon is increased to 0.3at.%, the content of Nb dissolved in B2 phase is decreased to 6.97%, and that dissolved in the grey phase is obviously decreased to 4.64%, for a certain amount of Nb is dissolved in the Ti₅Si₃ silicide phase. When the addition of silicon increases to 0.4at.% and 0.5at.%, there is no obvious change in the content of Nb dissolved in B2 phase and grey phase. However, the content of Nb dissolved in Ti₅Si₃ silicide phase is apparently increased from 4.54% to 6.27% when the addition of silicon increases from 0.3at.% to 0.5at.%.

Fig. 4: Microstructure of Ti48Al6Nb x Si alloys: (a) $x=0$; (b) $x=0.1$; (c) $x=0.2$; (d) $x=0.3$; (e) $x=0.4$; (f) $x=0.5$

B2 phase in the TiAl alloys is a metastable phase transformed from the high temperature stable β phase and can improve the high temperature deformation behaviors of alloys. B2 phase forms when the β phase stable element is segregated between lamellae or in the lamellar colonies (β -segregation). However, silicon can partially substitute Nb in B2 phase, thus the addition of Si can decrease the content of Nb in the B2 phase and increase the content of Nb in the matrix alloy before forming Ti₅Si₃. Therefore, the amount of B2 phase decreases when silicon is added into the Ti48Al6Nb alloy ($x=0.1$ at.%, 0.2at.%). When the addition of silicon is increased to 0.3%, Ti₅Si₃ phase begins to form in the alloy. The formation of Ti₅Si₃ phase can dissolve a certain content of Nb (shown in Table 2), and therefore lead to a decrease in the amount of Nb dissolved in the TiAl matrix alloy. Thus, the amount of B2 phase decreases. When the addition of silicon is further

increased to 0.4at.% and 0.5at.%, the increment of silicon leads to an obvious increase in the amount of Ti₅Si₃ phase. Thus, the total amount of element Nb dissolved in Ti₅Si₃ phase increases, and therefore leads to a decrease of B2 phase in the TiAl alloy.

2.3 Mechanical properties

The uniaxial compressive test results of Ti48Al6Nb x Si alloys with different additions of silicon are shown in Fig. 5 and Table 3. The compressive strength of the TiAl alloys increases with increasing the content of silicon, while the fracture strain of the alloys first slightly increases and then has a decreasing trend. The compressive strength of Ti48Al6Nb alloy is 2,063 MPa and the fracture strain is 34.7%. When adding 0.1at.% silicon, the compressive strength of Ti48Al6Nb0.1Si alloy increases to 2,091 MPa and the fracture strain is slightly increased to 35.2%. The compressive strength of

Table 2: EDS results for different positions of Ti48Al6Nb_xSi alloys

Si content (at.%)	Position	Ti (at.%)	Al (at.%)	Nb (at.%)	Si (at.%)
0	A	47.71	43.86	8.43	
	B	44.78	51.06	4.06	
	C	44.82	48.96	6.22	
0.1	A	44.87	47.62	7.30	0.21
	B	44.06	50.95	4.82	0.17
	C	45.50	48.42	5.99	0.09
0.2	A	45.10	47.45	7.12	0.33
	B	46.03	46.61	7.10	0.26
	C	44.51	49.46	5.95	0.08
0.3	A	45.91	46.28	6.97	0.85
	B	42.75	52.08	4.64	0.52
	C	46.27	47.20	6.19	0.34
0.4	D	47.53	31.77	4.54	16.16
	A	46.60	46.39	6.64	0.37
	B	42.69	52.19	3.91	1.20
0.5	C	44.53	48.77	6.24	0.46
	D	49.86	23.70	6.12	20.33
	A	45.60	46.76	6.99	0.65
	B	43.14	51.55	4.79	0.53
	C	44.65	48.94	6.06	0.35
	D	49.10	21.08	6.27	23.55

Fig. 5: Compressive properties of Ti48Al6Nb_xSi alloys: (a) $x=0$; (b) $x=0.1$; (c) $x=0.2$; (d) $x=0.3$; (e) $x=0.4$; (f) $x=0.5$

Ti48Al6Nb0.2Si alloy increases to 2,120 MPa when the content of silicon is 0.2at.%, and the fracture strain is decreased to 33.5%. When the content of silicon is 0.5at.%, the compressive strength of the TiAl alloy reaches its maximum of 2,281

Table 3: Compressive properties of Ti48Al6Nb_xSi alloys

Si content (at.%)	Strength (MPa)	Strain (%)
0	2,063	34.73
0.1	2,091	35.23
0.2	2,120	33.48
0.3	2,138	33.39
0.4	2,160	31.84
0.5	2,281	30.76

MPa. At the same time, the fracture strain of the alloy reaches its minimum of 30.8%. When adding 0.5at.% silicon, the compressive strength of the Ti48Al6Nb alloy improves about 11% from 2,063 MPa to 2,281 MPa. In contrast, the addition of Si leads to a decrease in the compression strain of the alloys from 34.7% to 30.8% when the content of Si increases from 0.1at.% to 0.5at.%.

2.4 Discussion

The aforementioned phenomenon of the compressive strength and fracture strain of the Ti48Al6Nb_xSi alloys can be attributed to the decrease of B2 phase and the formation of Ti₅Si₃ phase by the addition of silicon.

As has been stated above, the addition of silicon will increase solubility of Nb in the Ti48Al6Nb_xSi alloys when the content of silicon is less than 0.3at.%, and Ti₅Si₃ phase particles will form on the lamellar colony interface of the Ti48Al6Nb_xSi alloys when the content of silicon is ≥ 0.3 at.%. This is because the solubility of silicon in the α phase of TiAl alloys is very low. During the solidification process, silicon will be squeezed from α phase and segregated in the liquid melt between the dendrites. When the content of silicon is higher than 4% in the segregation area, Ti₅Si₃ phase will form at the interface of the colonies^[25-27]. Thus, when the content of silicon is < 0.3 at.%, the solidification process of the Ti48Al6Nb_xSi alloys is as follows, $L \rightarrow L+\beta \rightarrow L+\alpha+\beta_r \rightarrow \alpha+\beta_r \rightarrow \alpha+\gamma+\beta_r \rightarrow (\alpha_2+\gamma)+\gamma+B2$. When the content of silicon is ≥ 0.3 %, the solidification process of the Ti48Al6Nb_xSi alloys is as follows, $L \rightarrow L+\beta \rightarrow L+\alpha+\beta_r \rightarrow \alpha+\beta_r+Ti_5Si_3 \rightarrow \alpha+\gamma+\beta_r+Ti_5Si_3 \rightarrow (\alpha_2+\gamma)+\gamma+B2+Ti_5Si_3$.

Figure 6 shows the schematic diagram of microstructure evolution during the solidification process of Ti48Al6Nb alloys with different contents of silicon. As shown in Fig. 6(a1)-(a4), when the content of silicon is $x<0.3$ at.%, β phase with higher solubility of Nb firstly precipitates from the melt. At the same time, Nb migrates from the remaining melt into β phase, and Si will migrate from β phase into the remaining melt [Fig. 6(a1)]. Then α phase forms by the peritectic reaction, $L+\beta \rightarrow \alpha$. Meanwhile, Nb atoms segregate in α phase and form β -segregation [Fig. 6(a2)]. As the solidification process proceeds, many α colonies with β -segregation form after the melt is consumed [Fig. 6(a3)]. After that, the α_2/γ lamellae form by the solid-state phase transformation from α phase, and the residual β phases (β -segregation areas) transfer into B2-

Fig. 6: Schematic diagram showing microstructure evolution of Ti48Al6NbxCi alloys:

(a1) – (a4): $x < 0.3\text{at.}\%$; (a1) formation of β phase; (a2) formation of α phase with β -segregation;

(a3) α colonies with β -segregation; (a4) formation of B2 and α_2/γ lamellae

(b1) – (b4): $x \geq 0.3\text{at.}\%$; (b1) formation of β phase; (b2) formation of α phase and segregation of Si;

(b3) formation of Ti_5Si_3 at the interface of α colonies; (b4) formation of B2 and α_2/γ lamellae

structure [Fig. 6(a4)]. Consequently, the microstructure of the Ti48Al6NbxCi alloys with lower content of silicon consists of α_2/γ lamella and B2 phase.

When the content of silicon is $\geq 0.3\text{at.}\%$ as shown in Fig. 6(b1–b4), the initial two stages of the solidification process of the TiAl alloy are the same as that when the content of silicon is $< 0.3\text{at.}\%$. During the solidification process, β phase with higher solubility of Nb also primarily precipitates from the alloy melt [Fig. 6(b1)], and then α phase with β -segregation forms by the peritectic reaction, as shown in Fig. 6(b2). After that, Nb atoms segregate in α phase and Si atoms are squeezed from α phase and segregated in the liquid melt between the dendrites. Ti_5Si_3 phase then forms at the interface of the α colonies [Fig. 6(b3)]. After the melt is consumed, α colonies with β -segregation are transformed into α_2/γ lamellae by the solid-state phase transformation, and β -segregations transfer into B2 phase [Fig. 6(b4)]. Finally, the microstructure of the Ti48Al6NbxCi alloys with higher content of silicon consists of α_2/γ lamella, B2 phase and Ti_5Si_3 phase.

Figure 7 shows the relationship between the compressive strength of the alloys and volume fraction of B2 phase and Ti_5Si_3 phase. It shows that the decrease of B2 phase and the formation of Ti_5Si_3 phase lead to the increase of compressive strength and the decrease of fracture strain of the Ti48Al6NbxCi alloys. When the addition of silicon is less than $0.3\text{at.}\%$, silicon acts as an interstitial solid solution element in the Ti48Al6NbxCi alloys. Though the addition of silicon decreases the amount of B2 phase, the ultimate strength of the alloys at room temperature is increased for the solid solution strengthening. Meanwhile, the interstitial silicon atoms lead to lattice distortion of alloys, which will increase the resistance of the dislocation motion, and make the slipping deformation of alloys more difficult, therefore decreasing the fracture strain of the alloys. When the content of silicon is $0.3\text{at.}\%$, the formation of Ti_5Si_3 phase can dissolve a certain amount of Nb

Fig. 7: Relationship between compressive strength of alloys and volume fraction of B2 and Ti_5Si_3 phase

atoms, and leads to a decrease in the amount of B2 phase. At this time, the formed Ti_5Si_3 phase acts as the secondary phase, and exhibits the secondary phase strengthening mechanism in the alloy. When continuing to increase the content of silicon to $0.4\text{at.}\%$ and $0.5\text{at.}\%$, the amount of B2 phase decreases while that of Ti_5Si_3 phase further increases. Thus, the secondary phase strengthening mechanism caused by Ti_5Si_3 phase is still the main strengthening mechanism. Therefore, the ultimate strength of the TiAl alloy still increases with increasing of the silicon. At the same time, the deformation of silicide phase cannot coordinate with that of the matrix alloy during the compression test, and the deformation of the alloys is much harder due to the formation of silicide phase. Consequently, the fracture strain of alloys is decreasing with addition of silicon.

Therefore, the strengthening mechanism of Ti48Al6NbxCi alloys with different contents of silicon is solid solution strengthening when the addition of silicon is less than $0.3\text{at.}\%$, and changed to the combination of solid solution strengthening and secondary phase strengthening when the addition of silicon is no less than $0.3\text{at.}\%$.

3 Conclusions

Ti48Al6Nb x Si (at.%, $x=0, 0.1, 0.2, 0.3, 0.4$ and 0.5) alloys are prepared by vacuum arc melting, and the microstructure evolution, phase constitution, and mechanical properties of the alloys were systematically studied. Several conclusions can be drawn as follows:

(1) The addition of silicon, a α phase stabilizing element, leads to a decrease in the amount of γ phase and an increase in the amount of α_2 phase.

(2) The alloys consist of γ -TiAl phase, α_2 -Ti₃Al phase and B2 phase when Si addition is less than 0.3at.%, and Ti₅Si₃ silicide phase is formed when the addition of silicon is no less than 0.3at.%, which can decrease the volume fraction of B2 phase. Furthermore, the formation of silicide decreases the amount of Nb dissolved in the TiAl matrix.

(3) Compressive tests show that the compressive strength increases from 2,063 MPa to 2,281 MPa when Si addition increases from 0 to 0.5at.%, while the fracture strain has a decreasing tendency from 34.7% to 30.8%. The increase of strength and decrease of fracture strain are attributed to the decrease of B2 phase and the formation of Ti₅Si₃ phase.

(4) The strengthening mechanism is changed from solid solution strengthening when the addition of silicon is less than 0.3at.% to the combination of solid solution strengthening and secondary phase strengthening when the addition of silicon is no less than 0.3at.%.

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