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Experimental study on phase relationships in the Si-rich portion of the Nb–Si–W ternary system

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ABSTRACT

This paper describes the experimental study on liquidus projection and the isothermal section at 1873 K of the Nb–Si–W ternary system in the Si-rich portion. The microstructures and solidification paths of the as-cast alloys were analysed. The constituent phases and their equilibrium compositions of the as-cast + heat-treated alloys were determined. The microstructure observation, the phase identification and the composition measurement were performed using scanning electron microscopy (SEM), X-ray diffraction (XRD) and electron probe microanalysis (EPMA), respectively. No ternary compound was found. For the liquidus projection in the investigated Si-rich portion, there exist four primary solidification regions, Diamond–Si, $\beta(\text{Nb,W})_5\text{Si}_3$, Nb(W)Si_2 and W(Nb)Si_2 , and two eutectic invariant reactions, $\text{L} \rightarrow \beta(\text{Nb,W})_5\text{Si}_3 + \text{W(Nb)Si}_2 + \text{Nb(W)Si}_2$ and $\text{L} \rightarrow \text{W(Nb)Si}_2 + \text{Nb(W)Si}_2 + \text{Diamond-Si}$. The type of the latter invariant reaction was determined with the help of the thermodynamic assessment of the system. Together with literature reported investigations for the Nb–W-rich portion, the whole liquidus projection of the Nb–Si–W ternary system was constructed. For the isothermal section at 1873 K in the investigated Si-rich portion, there are three three-phase regions, liquid + $\text{Nb(W)Si}_2 + \text{W(Nb)Si}_2$, $\text{Nb(W)Si}_2 + \alpha\text{Nb(W)}_5\text{Si}_3 + \beta(\text{Nb,W})_5\text{Si}_3$ and $\text{Nb(W)Si}_2 + \text{W(Nb)Si}_2 + \beta(\text{Nb,W})_5\text{Si}_3$, and seven two-phase regions, liquid + Nb(W)Si_2 , $\text{Nb(W)Si}_2 + \alpha\text{Nb(W)}_5\text{Si}_3$, $\alpha\text{Nb(W)}_5\text{Si}_3 + \beta(\text{Nb,W})_5\text{Si}_3$, $\text{Nb(W)Si}_2 + \beta(\text{Nb,W})_5\text{Si}_3$, liquid + W(Nb)Si_2 , $\text{Nb(W)Si}_2 + \text{W(Nb)Si}_2$ and $\text{W(Nb)Si}_2 + \beta(\text{Nb,W})_5\text{Si}_3$.

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1. Introduction

Nbss/ Nb_5Si_3 *in situ* composites exhibit a good balance in mechanical properties between the room temperature toughening provided by the Nb-rich solid solution phase Nbss and the high temperature strength up to 1500 °C, provided by the intermetallic phase Nb_5Si_3 [1]. Nbss/ Nb_5Si_3 *in situ* composites, generally based on the phase equilibrium relationship of the Nb-rich region of the Nb–Si phase diagram, have been studied by many researchers [2–5]. Mendiratta et al. [2] investigated the as-cast + hot-extruded + heat-treated Nbss + Nb_5Si_3 two-phase alloys with compositions of 10 and 16 at% Si, showing the ductile-phase toughening at low temperatures and the reasonable strength retention at high temperatures (1400–1600 °C). The fracture toughness was about 21 $\text{MPa}\sqrt{\text{m}}$ for the Nb–10Si alloy with Nbss + Nb_5Si_3 two-phases, while that of Nb_5Si_3 intermetallic phase has been reported to be about 3 $\text{MPa}\sqrt{\text{m}}$ at room temperature [6]. The fracture toughnesses for the as-cast + hot-extruded + heat-treated Nb–16.5Si alloy were also found to be

12.8 $\text{MPa}\sqrt{\text{m}}$ at room temperature and 17 $\text{MPa}\sqrt{\text{m}}$ at 1400 °C by Mendiratta et al. [3]. Bewlay et al. [4] found the fracture toughness of the directionally solidified Nb–Si *in situ* composites (10–25 at% Si) was about 50% more than that of the as-cast alloys and 20% less than that of the extruded alloys of the equivalent compositions. Swkido et al. [5] investigated the directionally solidified and arc-melted Nb–17.5Si alloys with or without 10 at% Ti. The directionally solidified alloys had the higher fracture toughness compared with the arc-melted ones, in accordance with Ref. [4]; the fracture toughness should be improved by low-rate solidification, Ti alloying or heat-treatment; for the Nb–17.5Si alloy, the fracture toughness of 14.5 $\text{MPa}\sqrt{\text{m}}$ at room temperature and the compressive strength of 580 MPa at 1673 K were achieved by directional solidification at the rate of 10 mm/h.

However, one major impediment of the Nbss/ Nb_5Si_3 alloys for high-temperature applications is their catastrophic oxidation behaviour. To obtain material systems with improved environmental resistance while retaining their structural properties, studies with addition of alloying elements, such as Ti, Al, Hf, Cr, Mo and W, were initiated on the Nbss/ Nb_5Si_3 *in situ* composites. Sha et al. [7,8] studied the effect of W addition on the yield strength of Nb–10Mo–10Ti–18Si-based *in situ* composites, and

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found that the yield strength ($\sigma_{0.2}$) and the specific yield strength ($\sigma_{0.2s}$) ($\sigma_{0.2}$ divided by the density) at 1670 K increased markedly with increasing W content; the directionally solidified samples showed higher $\sigma_{0.2}$ and $\sigma_{0.2s}$ than the as-cast samples; even at 1770 K, the directionally solidified sample with 15 at% W showed a high $\sigma_{0.2}$ of about 650 MPa. Sha et al. [9] also found that the Nb–10W alloy showed the high fracture toughness of about 15.3 MPa \sqrt{m} and the low $\sigma_{0.2}$ of about 90 MPa at 1670 K while the Nb–10Si–10W showed the high $\sigma_{0.2}$ of about 330 MPa at 1670 K and the low fracture toughness of about 8.2 MPa \sqrt{m} , and concluded that the toughness was supplied by the metallic Nbss phase and the high-temperature strength was mainly provided by the brittle silicide phase. Ma et al. [10] found that both the low-temperature deformability and the high-temperature strength of Nbss/Nb–silicide *in situ* composites were improved by the addition of appropriate amounts of Mo and/or W. By the addition of W, Xiong et al. [11] also improved significantly the oxidation resistance of Nbss/Nb₅Si₃ *in situ* composite (Nb–20Si–10W alloy). Consequently, by alloying with W, the Nbss/Nb₅Si₃ *in situ* composites may get a good balance among high-temperature strength, room temperature toughness and high-temperature oxidation resistance. The Nb–Si–W ternary system is becoming an important system to be investigated for the development of the Nb–Si-based high temperature materials.

The phase relationship is an important guidance for materials design and also an essential basis for the CALPHAD (CALculation of PHase Diagrams) approach. In order to get a comprehensive and consistent thermodynamic description for one of the phases of a ternary system, it is necessary to understand the phase equilibrium behaviour of the other related phases in the system. For the Nb–Si–W ternary system, literature reports are mainly concentrated on the Nb–W-rich portion with the application importance. From the view point of the thermodynamic assessment of the system, the purpose of the present paper is to study the constituent phases and the solidification processes of the as-cast alloys in the Si-rich portion of the Nb–Si–W ternary system, so that the whole liquidus projection of the Nb–Si–W ternary system can be constructed together with the literature reported investigations for the Nb–W-rich portion. Meanwhile, some selected as-cast alloys were heat-treated at 1873 K for 72 h and by studying the phase equilibria of these alloys, the isothermal section for the Si-rich portion was obtained.

2. Literature study on the phase diagrams of the binary and the ternary systems

2.1. The Nb–Si binary phase diagram

The Nb–Si binary phase diagram is based on the assessment by Geng et al. [12]. The stable phases in this system are the terminal phases Bcc–Nbss and Diamond–Si, the intermetallic compounds Nb₃Si, α Nb₅Si₃, β Nb₅Si₃ and NbSi₂ and the liquid phase referred to

Table 1
Invariant reactions involving the liquid phase in the Nb–Si system [12].

Reaction type	Reaction	Composition of L (at% Si)	T (°C)
Congruent	L → β Nb ₅ Si ₃	39.0	2526
Congruent	L → NbSi ₂	66.7	1962
Eutectic	L → Bcc + Nb ₃ Si	17.3	1916
Peritectic	L + β Nb ₅ Si ₃ → Nb ₃ Si	20.3	1977
Eutectic	L → β Nb ₅ Si ₃ + NbSi ₂	57.7	1873
Eutectic	L → NbSi ₂ + Diamond–Si	98.3	1407

Table 2
Invariant reactions involving the liquid phase in the W–Si system [13].

Reaction type	Reaction	Composition of L (at% Si)	T (°C)
Congruent	L → W ₅ Si ₃	0.375	2108
Congruent	L → WSi ₂	0.667	2002
Eutectic	L → Bcc + W ₅ Si ₃	0.329	2089
Eutectic	L → W ₅ Si ₃ + WSi ₂	0.525	1940
Eutectic	L → WSi ₂ + Diamond–Si	0.960	1388

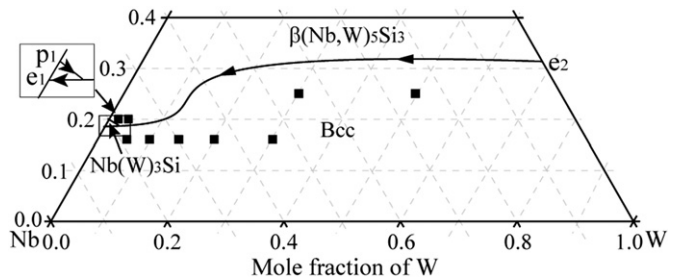


Fig. 1. Liquidus projection for the Nb–W-rich portion of the Nb–Si–W ternary system [14].

as “L” hereafter. The invariant reactions involving the liquid phase are listed in Table 1.

2.2. The W–Si binary phase diagram

The W–Si binary phase diagram is based on the assessment by Vahlas et al. [13]. The stable phases in this system are the terminal phases Bcc–Wss and Diamond–Si, the intermetallic compounds W₅Si₃ and WSi₂ and the liquid phase. The invariant reactions involving the liquid phase are listed in Table 2.

2.3. The Nb–W binary phase diagram

The Nb–W binary system was relatively simple, involving an infinite soluble solid phase and a liquid phase. No invariant reaction exists.

2.4. Previous experiments on the liquidus projection of the Nb–Si–W ternary system

Ma et al. [14] constructed a partial liquidus projection of the ternary system in the Nb–W rich region up to 37.5 at% Si, as shown in Fig. 1. There exist three primary phase regions, Bcc, β (Nb,W)₅Si₃ and Nb(W)₃Si, and one invariant point for the transitional invariant reaction L + β (Nb,W)₅Si₃ → Nb(W)₃Si + Bcc. The liquidus projection for the Si-rich portion has not been reported.

2.5. Previous experiments on the isothermal section of the Nb–Si–W ternary system

There is not much information about the phase equilibria of the Nb–Si–W ternary system. Ma et al. [14] speculated a partial isothermal section for the Nb–W rich portion of Nb–Si–W ternary system at 1973 K. The samples were arc-melted followed by annealing at 1973 K for 48 h and furnace cooled.

3. Experimental procedures

Each sample with a gross weight about 5 g was arc-melted by a non-consumable tungsten electrode in a water-cooled copper

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