

Microstructure optimization of directionally solidified hypereutectic Nb–Si alloy

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Abstract: Nb–16Si–24Ti–10Cr–2Al–2Hf alloy was directionally solidified with withdrawal rates of 1.2, 6, 18, 36 and 50 mm/min and then heat treated at 1400, 1450 and 1500 °C with withdrawal rate of 50 mm/min for 10 h. The effects of withdrawal rate and heat treatment temperature on the microstructure were studied. The microstructure of directionally solidified alloy was composed of the primary Nb₅Si₃, Nbss/Nb₅Si₃ eutectic cells and Cr₂Nb, which distribute paralleled to the growth direction. The microstructure becomes more refined with the increasing withdrawal rate, accompany with the evolution of eutectic cells morphology. After heat treatment, Nbss phase connects and forms a continuous matrix, and the Cr₂Nb phase becomes smaller and distributes more dispersedly. After heat treatment at 1450 °C for 10 h, the alloy achieves balance between the optimization of microstructure and alleviation of solute segregation.

Key words: Nb–Si alloy; directional solidification; eutectic; withdrawal rate; microstructure; heat treatment

1 Introduction

With high melting points, low densities and attractive high temperature strength and excellent creep resistance, the Nb–Si based ultrahigh temperature alloys are expected to be promising candidate materials for employment at 1200–1400 °C [1–4]. Current studies focus on the Nb–Ti–Si–Cr–Al–Hf in situ composites which consist of the ductile niobium solid solution Nbss phase, creep resistant but brittle phase Nb₅Si₃, and with or without Cr₂Nb phase (C14 Laves phase) which improves the oxidation resistance [5–8]. A balanced effect of ductile and brittle phases can be achieved by appropriate volume fractions of composition phases [3,9]. Moreover, morphological characteristics of component phases play an important role in the mechanical properties of structural materials [10,11]. SEKIDO and KIMURA [12] reported that directional solidification could produce well-aligned regular structures and improve fracture toughness and high temperature strength. However, high withdrawal rate during directional solidification may result in the decrease of composition homogenizing [13,14]. Appropriate heat treatment is an effective way to alleviate solute

segregation and also eliminate meta-stable phases in the alloy [15,16]. Until now, the effect of withdrawal rate and heat treatment temperature on the microstructure evolution and composition homogenizing of directionally solidified (DS) Nb–Si based alloys is still unclear. Therefore, it is instructive to study the microstructure optimizing of Nb–Si based alloys.

In this study, Nb–16Si–24Ti–10Cr–2Al–2Hf alloy was directionally solidified by liquid-metal-cooled method (LMC) and then heat treated (HT). The purpose of the present work was to investigate the effects of withdrawal rate and heat treatment temperature on the solidification behavior and optimize the microstructure of the alloy.

2 Experimental

A master alloy button, with the nominal composition of Nb–16Si–24Ti–10Cr–2Al–2Hf (mole fraction, %), was prepared by vacuum non-consumable arc-melting (VCAM). Master alloy rods with 13 mm in diameter and 90 mm in length were prepared by the electro-discharge machining (EDM). The directional solidification was performed using a laboratory-scale Bridgman LMC furnace, equipped with a self-contributed

Y_2O_3 crucible (18 mm in outer diameter, 15 mm in inner diameter and 200 mm in length), as described in our previous work [17,18]. After holding for 20 min at 1850 °C in the atmosphere of argon, the assembled crucibles were withdrawn at 1.2 mm/min (DS1.2), 6 mm/min (DS6), 18 mm/min (DS18), 36 mm/min (DS36) and 50 mm/min (DS50), respectively. Then the DS50 samples were heat treated at 1400 °C (HT1400), 1450 °C (HT1450) and 1500 °C (HT1500) for 10 h, respectively, in a high vacuum heat treatment furnace. Microstructure analysis was performed by a scanning electron microscope (SEM, JXA-8100) equipped with an energy dispersive X-ray spectroscopy (EDS, INCA PentaFETx3). The phases were identified by micro-area X-ray diffraction (XRD, D/max2550HB+/PC Cu K_α).

3 Results and discussion

3.1 Microstructure and composition of directionally solidified alloy

Figure 1 shows the XRD patterns of DS alloy with withdrawal rates (R_w) of 1.2, 6, 18, 36 and 50 mm/min, respectively. The DS alloy with withdrawal rates of 1.2, 6, 18 and 36 mm/min was composed of the same phases: Nbss, $\alpha\text{-Nb}_5\text{Si}_3$, $\gamma\text{-Nb}_5\text{Si}_3$ and Cr_2Nb . However, it is interesting that the $\gamma\text{-Nb}_5\text{Si}_3$ disappears in the alloy with 50 mm/min. $\gamma\text{-Nb}_5\text{Si}_3$ (hexagonal structure) is considered a meta-stable phase, while $\beta\text{-Nb}_5\text{Si}_3$ (tetragonal structure) is the stable phase at a lower temperature and $\alpha\text{-Nb}_5\text{Si}_3$ (tetragonal structure) is the stable phase at high temperatures. This indicates that the meta-stable phase can be eliminated by the increase of withdrawal rate.

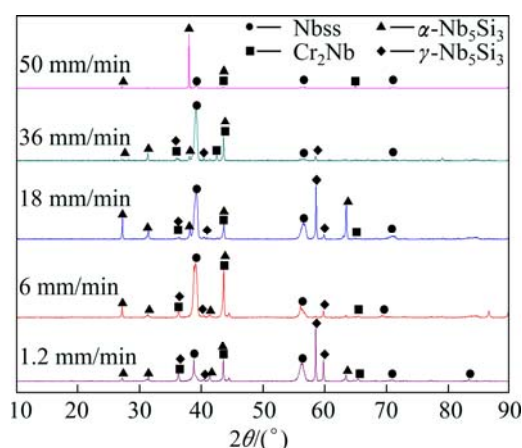


Fig. 1 XRD patterns of directionally solidified Nb-16Si-24Ti-10Cr-2Al-2Hf alloy with different withdrawal rates

Figures 2 and 3 show the longitudinal and transversal microstructures of the steady-state zones of the alloy with withdrawal rates of 1.2, 6, 18, 36 and 50 mm/min, respectively. The microstructures of all DS specimens were composed of gray primary Nb_5Si_3 laths,

Nbss/ Nb_5Si_3 eutectic clusters or cells, and black Cr_2Nb phase. Besides, dark grey Ti-rich Nbss phase was found surrounding Nbss/ Nb_5Si_3 eutectic clusters or cells.

From the longitudinal sections of the DS specimens (Fig. 2), it can be seen that the growth direction of the component phases was parallel to the withdrawal direction. Primary Nb_5Si_3 laths, with straight boundary, were 1 mm or longer in length and possessed quadrilateral morphology in transversal sections (Fig. 3). The Nbss presented the morphology of dendrites and the secondary dendrite arms were fully developed. The Nbss/ Nb_5Si_3 eutectic clusters or cells were found to present three types of morphologies. The first one, marked as eutectic I, was an irregular lamellar eutectic morphology aligning along the growth direction on longitudinal section (see Figs. 2(a) and (b)) and presented irregular quadrilateral petaloid morphology on transversal-section (see Figs. 3(a) and (b)). In the center of the eutectic cells, fine Nbss and Nb_5Si_3 were arranged alternately and in the exterior margin of most eutectic cells, Nbss became coarser and some dendrite Nbss formed. The second one, marked as eutectic II, was a complex and regular eutectic structure. It was composed of eutectic cells which coupled arranged along the growth direction, and presented as fishbone, fibrous and cluster shaped morphology (see Figs. 2(c)–(f)). On transversal section, the eutectic cells consisted of sub-spherical Nbss and Nb_5Si_3 which distributed alternatively with a petaloid morphology. And granular Nbss did not become coarser in the exterior margin (see Fig. 3(c)–(f)). The third one, marked as eutectic III, was a quasi-regular structure of eutectic morphology, whose microstructure was the rod-like Nb_5Si_3 inside and the lamellar-like Nbss/ Nb_5Si_3 eutectic cells in the periphery (see Figs. 2(g) and (h)). The small faceted phase Nb_5Si_3 connected and formed a continuous matrix, and the rod-like structure was perpendicular to the lamellar-like Nbss/ Nb_5Si_3 eutectic cells. On the transversal section, it was found that the fine Nbss/ Nb_5Si_3 eutectic cells were arranged surrounding the quadrilateral Nb_5Si_3 , with divorced Nbss dendrites in the exterior margin (see Figs. 3(g) and (h)). With the increase of withdrawal rate, the microstructure of the DS alloy became more oriented and homogeneous. The morphology of the eutectic cells varied from eutectic I to eutectic III.

Table 1 shows the quantitative metallographic analysis of directional solidification microstructures under different withdrawing rates. It can be found that with the increase of the withdrawing rates, the volume fraction of primary Nb_5Si_3 phase, Ti-rich Nbss, Nbss/ Nb_5Si_3 eutectic and Nb_5Si_3 in the eutectic cell decreased, the width of Nb_5Si_3 laths and the size of eutectic cells reduced, and the Nbss dendrites were refined. Based on the above analysis, it could be

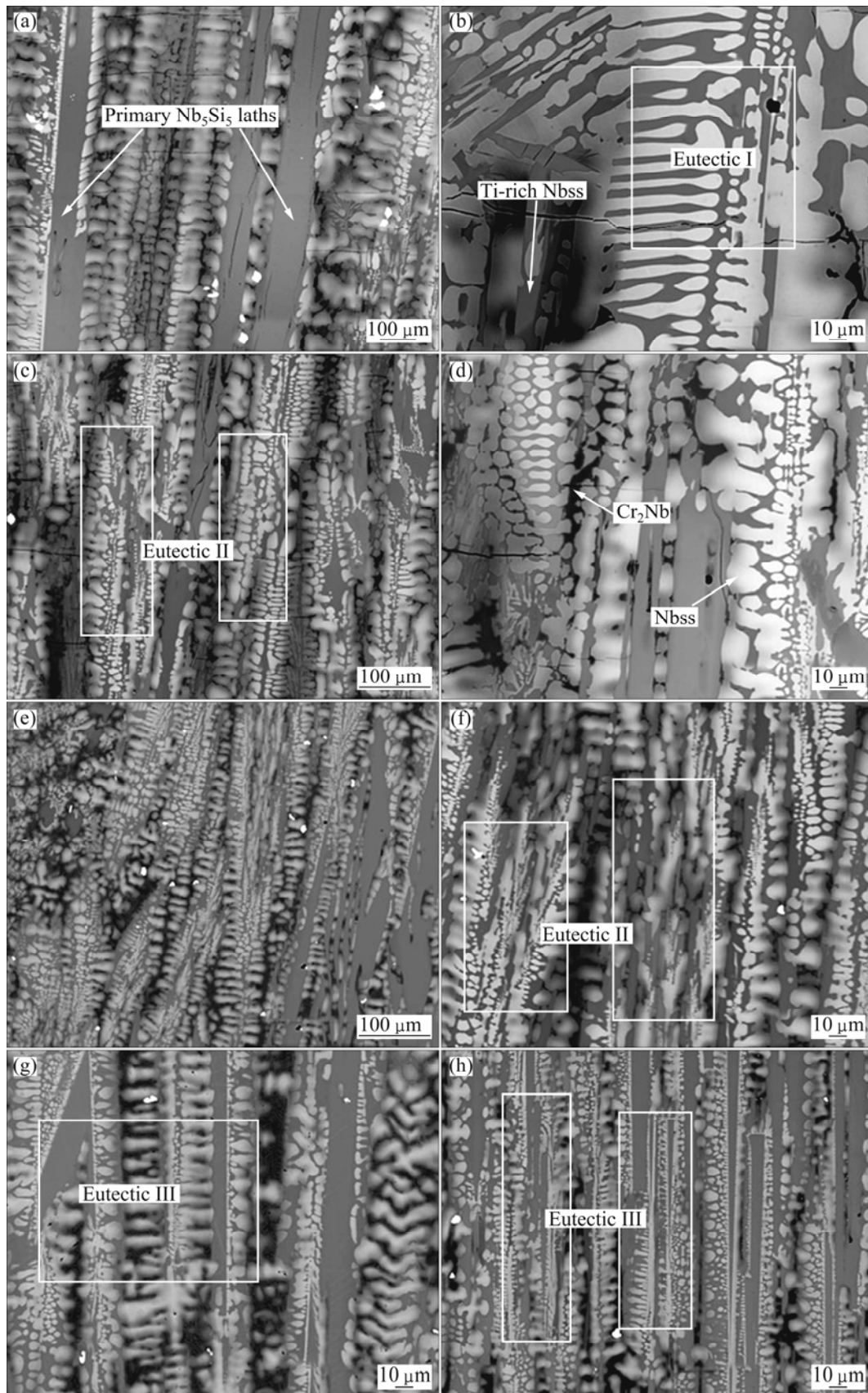


Fig. 2 Back scattered electron (BSE) images of microstructure on longitudinal-section of steady-state zones of directionally solidified Nb-16Si-24Ti-10Cr-2Al-2Hf alloy with withdrawal rates of 1.2 mm/min (a, b), 6 mm/min (c, d), 18 mm/min (e, f), 36 mm/min (g) and 50 mm/min (h)

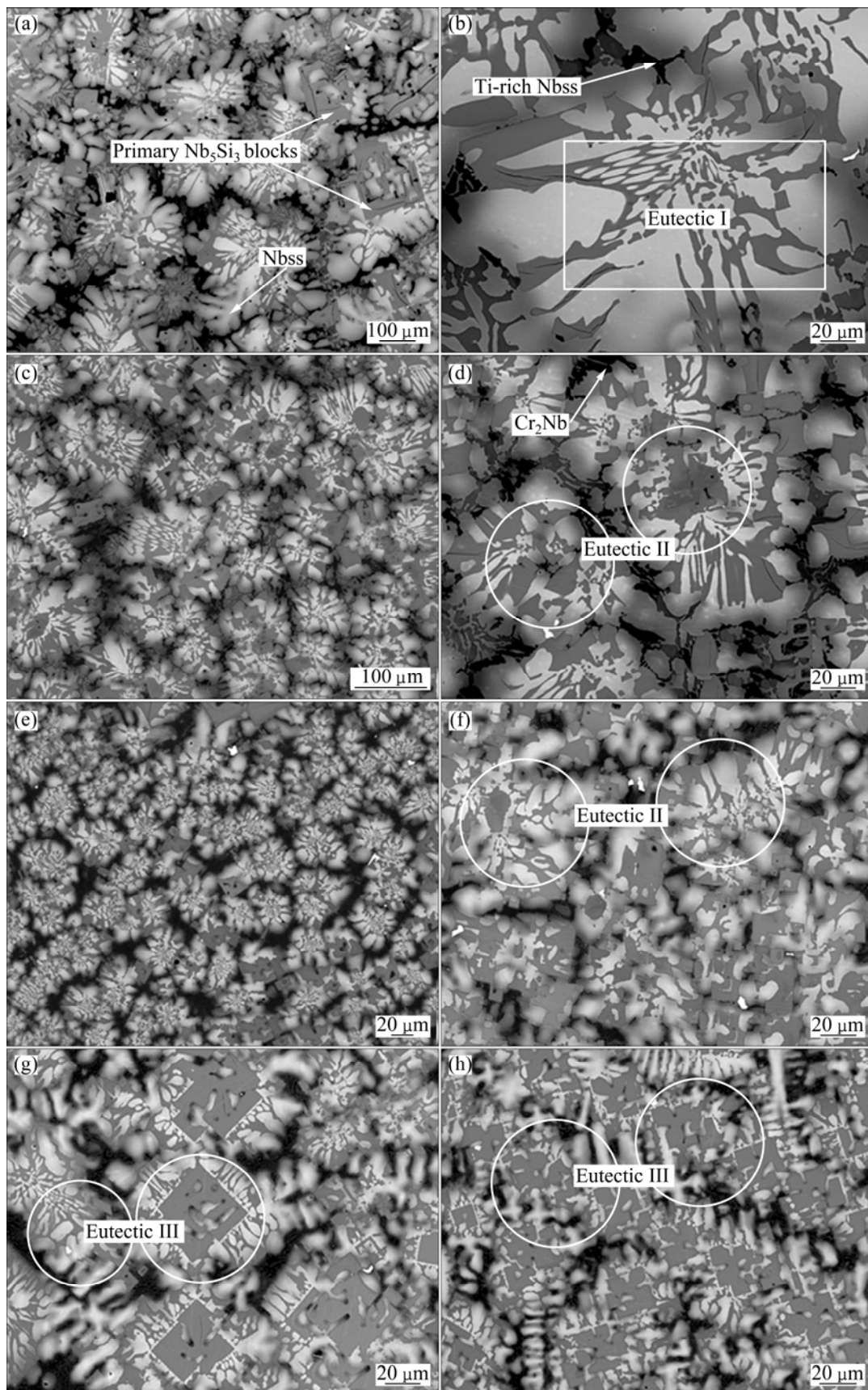


Fig. 3 Back scattered electron (BSE) images of microstructure on transversal-sections of steady-state zones of directionally solidified Nb-16Si-24Ti-10Cr-2Al-2Hf alloy with withdrawal rates of 1.2 mm/min (a, b), 6 mm/min (c, d), 18 mm/min (e, f), 36 mm/min (g) and 50mm/min (h)

Table 1 Quantitative metallographic analysis of directional solidification microstructures under different withdrawing rates

$R_w/$ (mm·min ⁻¹)	$d_1/\mu\text{m}$	$d_2/\mu\text{m}$	$(v_2/v_1)/$ %	$v_3/$ %	$v_4/$ %
1.2	100.16–235.79 (139.44)	250.63–300.72 (278.67)	19.3	25.7	3.6
6	70.16–100.38 (84.79)	111.49–137.69 (105.80)	28.6	21.5	3.3
18	46.39–63.15 (55.65)	67.89–88.23 (76.15)	32.5	17.6	3.2
36	30.72–37.33 (34.27)	42.21–60.38 (50.69)	40.8	13.8	2.9
50	17.13–28.15 (21.15)	30.05–40.30 (34.71)	41.6	10.9	2.8

d_1 : Size of primary Nb₅Si₃; d_2 : Average diameter of eutectic cells; v_1 : Volume fraction of eutectic clusters or cells; v_2 : Volume fraction of Nb₅Si₃ in eutectic cluster or cells; v_3 : Volume fraction of primary Nb₅Si₃; v_4 : Volume fraction of Ti-rich Nbss

concluded that the alloy directionally solidified with the 50 mm/min possessed optimal microstructure.

Table 2 illustrates the compositions of constituent phases of DS alloys with different withdrawing rates (1.2, 18, 50 mm/min). From Table 2, it was clear that the content of refractory element Nb in primary Nb₅Si₃ phase was 2%–4% (mole fraction) higher than that in the Nb₅Si₃ phase in eutectics and the content of low melting point elements (Ti and Cr) was 2%–4% (mole fraction) lower than that in the Nb₅Si₃ phase in eutectics. This was due to the fact that primary Nb₅Si₃ phase formed first, which contained more refractory elements, and at the same time it excluded low melting point elements to the surrounding liquid phase, leading to the later formed Nb₅Si₃ phase in eutectic contained more Ti and Cr. For the above reason, the content of low melting point elements in Nbss in eutectics was also lower than that in Ti-rich Nbss, which was observed at boundaries of eutectic cells and formed after the eutectics. Thus, necessary measures need to be taken to alleviate severer micro-segregation.

3.2 Microstructure and composition after heat treatment

In order to homogenize the composition of DS50 alloy, heat treatments were preceded at 1400, 1450 and 1500 °C for 10 h. Figure 4 illustrates the microstructures after heat treatment and the phases present were confirmed to be Nbss, α -Nb₅Si₃ and Cr₂Nb, the same phases as directionally solidified alloy. From Fig. 4, it can be found that after heat treatment at 1400 °C for 10 h, Nbss connected and formed a continuous matrix while the boundaries of primary Nb₅Si₃ were still smooth. Eutectic cells changed into fine Nb₅Si₃ fibers distributed uniformly in Nbss matrix. However, the morphology,

Table 2 Compositions of constituent phases of directionally solidified Nb–16Si–24Ti–10Cr–2Al–2Hf alloy with different withdrawing rates

$R_w/$ (mm·min ⁻¹)	Phase	$x/\%$					
		Nb	Ti	Si	Cr	Al	Hf
1.2	Nb ₅ Si ₃ blocks	48.36	13.90	35.67	0.32		1.69
	Nbss	61.30	24.37		11.50	1.96	0.87
	Nb ₅ Si ₃ in EU	45.92	16.15	35.72	0.39		1.84
	Ti-rich Nbss	45.55	40.82		7.17	0.98	6.40
	Cr ₂ Nb	26.80	16.17		54.04		2.98
18	Nb ₅ Si ₃ blocks	48.49	13.60	35.81	0.43		1.67
	Nbss	64.91	23.33		10.15	0.91	0.67
	Nb ₅ Si ₃ in EU	44.04	17.29	35.38	0.87		1.92
	Ti-rich Nbss	40.20	34.52		22.35	0.44	2.48
	Cr ₂ Nb	22.74	18.99		54.14		3.15
50	Nb ₅ Si ₃ blocks	48.18	14.04	35.63	0.52		1.63
	Nbss	64.10	23.69		10.23	1.27	0.72
	Nb ₅ Si ₃ in EU	46.21	16.73	34.35	1.25		1.48
	Ti-rich Nbss	28.93	47.47		18.76		1.99
	Cr ₂ Nb	23.98	17.54		55.81		2.67

EU represents the abbreviation for eutectic cells; Ti-rich Nbss regions were observed in boundaries of eutectic cells.

distribution and volume fraction of Cr₂Nb were not different from those of the DS alloy. After heat treatment at 1450 °C for 10 h, primary Nb₅Si₃ blocks with sharp interfaces converted into relatively small size particles with blunted and round interfaces, and the volume fraction of Cr₂Nb phase decreased and the Cr₂Nb phase became smaller and distributed more dispersedly. After heat treatment at 1500 °C for 10 h, a lot of eutectic cells in the DS specimens lost their lamellar morphologies. A semi-solid microstructure was generated with sub spherical Nbss and small faceted Nb₅Si₃ blocks arranging alternately. The formation of Nbss/Cr₂Nb eutectic cells (as marked by the rectangle in Fig. 4(e)) suggested that the incipient melting temperature of Nbss/Cr₂Nb eutectics was between 1450 °C and 1500 °C.

Table 3 shows the compositions of constituent phases of DS50 alloy after heat treatments. It could be found that the Ti-rich Nbss regions disappeared and the Ti content increased by 5%–6% (mole fraction) in Nbss than the DS alloy. The disappearance of Ti-rich Nbss regions suggested the diminishment of micro-

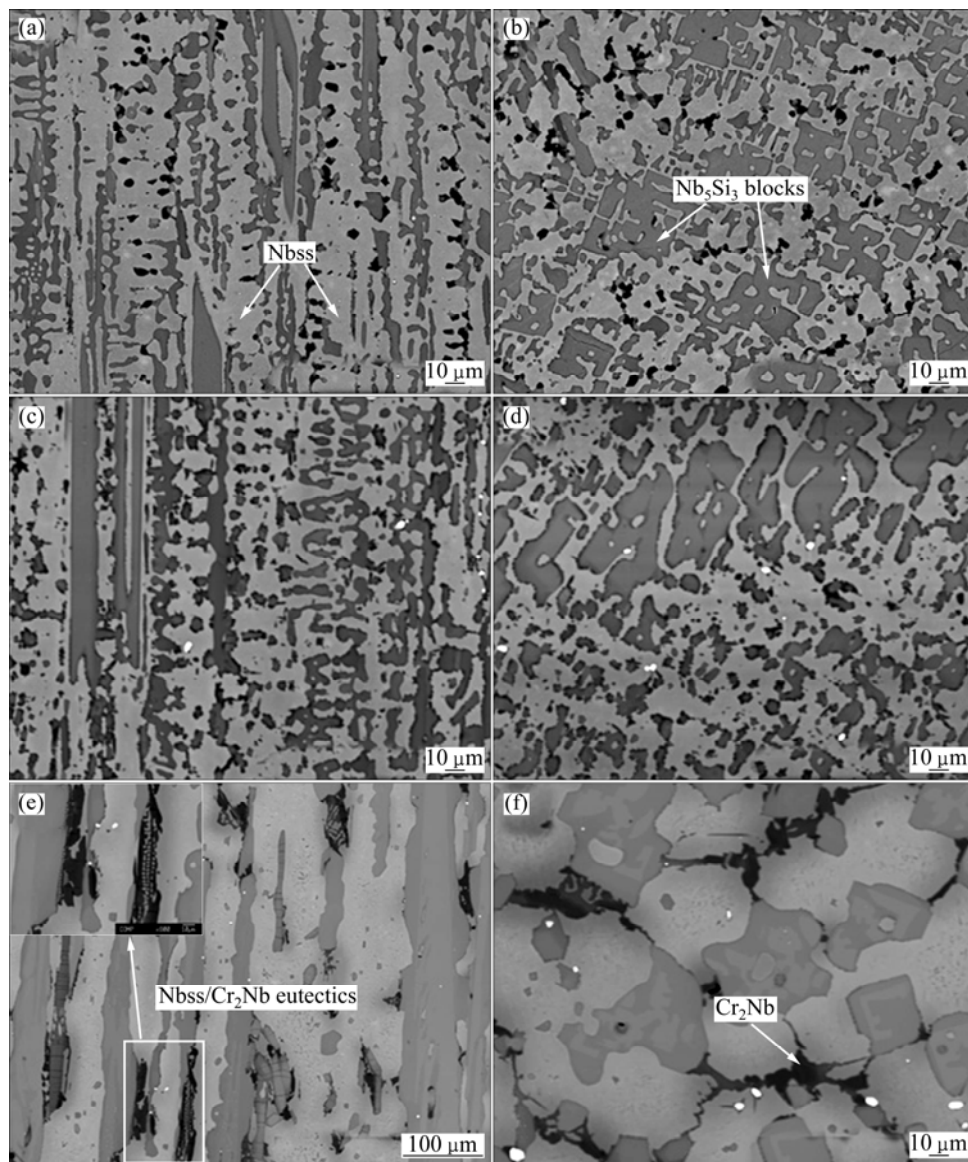


Fig. 4 BES images of longitudinal-section (a, c, e) and transversal-section (b, d, f) of directionally solidified Nb–16Si–24Ti–10Cr–2Al–2Hf alloy with withdrawal rate of 50 mm/min after high temperature heat treatments: (a, b) 1400 °C for 10 h; (c, d) 1450 °C for 10 h; (e, f) 1500 °C for 10 h

Table 3 Compositions of constituent phases of Nb–16Si–24Ti–10Cr–2Al–2Hf alloy after different heat treatment (mole fraction, %)

Temperature/°C	Phase	$x(\text{Nb})/\%$	$x(\text{Ti})/\%$	$x(\text{Si})/\%$	$x(\text{Cr})/\%$	$x(\text{Al})/\%$	$x(\text{Hf})/\%$
1400	Nb ₅ Si ₃ blocks	48.25	14.05	35.71	0.38		1.61
	Nbss	53.93	30.47		12.98	1.84	0.68
	Nb ₅ Si ₃ in EU	44.10	17.61	35.89	0.68		1.72
	Cr ₂ Nb	27.78	13.87		54.97		3.38
1450	Nb ₅ Si ₃ blocks	47.80	14.25	35.72	0.68		1.55
	Nbss	52.96	30.29	0.49	13.36	1.99	0.91
	Nb ₅ Si ₃ in EU	44.46	17.12	36.11	0.59		1.72
	Cr ₂ Nb	25.61	15.76		55.68		2.95
1500	Nb ₅ Si ₃	45.05	16.27	35.97	0.75		1.96
	Nbss	53.36	31.68		12.92	1.56	0.48
	Cr ₂ Nb	25.96	16.31		54.78		2.95

segregation after heat treatment. The composition of Nb_5Si_3 in HT1500 was obviously different for the reason of the disappearance of the Nbss/ Nb_5Si_3 eutectics. Moreover, the HT1450 alloy possessed the highest average Cr content in Nbss due to the solution of part Cr_2Nb , which resulted in the minimum volume fraction of Cr_2Nb in the alloy.

From above results, it could be concluded that the HT1450 alloy possessed optimal microstructure and homogeneous compositions of component phases.

4 Discussion

4.1 Mechanism of effects of withdrawal rate on morphology of eutectic cells

With the increase of withdrawal rate, the microstructure of the DS alloy becomes more oriented and homogeneous. The morphology of the eutectic cells varies from eutectic I to eutectic III. As mentioned in Ref. [19], it can be concluded that the growth trend of small faceted phase could be weakened or transformed to non-small faceted trend with increasing growth rate, and the volume fraction of small faceted phase in the eutectics (ϕ_f) exerts a great influence on the eutectic morphology. Since Nb_5Si_3 is a small faceted phase and the growth of Nbss/ Nb_5Si_3 eutectics is irregular, the increase of volume fraction of Nb_5Si_3 in eutectic cluster or cells (v_2/v_1 , shown in Table 1) which increased proportionally with the withdrawal rate, results in the transformation of eutectic morphology.

4.2 Mechanism of effects of heat treatment on compositions of constituent phases

With the increase of heat treatment temperature, the content of Ti in Nb_5Si_3 increases while the content of Nb decreases, which may be caused by the diffusion of Ti and Nb driven by the concentration difference between Nbss and Nb_5Si_3 . The Ti atom is more active and diffuses more easily than Nb due to its smaller radius. However, the sum of the mole fraction of Ti and Nb is almost consistent. During high temperature heat treatment, the distribution ratio of alloying elements between Nbss and Nb_5Si_3 changes for the alloying elements diffusing in different phases. Since the distribution ratio of Cr (D_{Cr}) changes more obviously, it could be used to analyze the effect of heat treatment temperature on the variance of compositions of constituent phases. As shown in Fig. 5, D_{Cr} decreases with the increasing heat temperature. Although the solid solution of Cr in Nb_5Si_3 is higher with the increase of heat treatment temperature [20], the content of Cr in Nbss is still far higher than that in Nb_5Si_3 . This high concentration gradient of Cr promotes the diffusion from Nbss to Nb_5Si_3 , and is more accelerated with the increasing heat treatment

temperature, thus leads to the decrease of D_{Cr} . The micro-segregation is also alleviated.

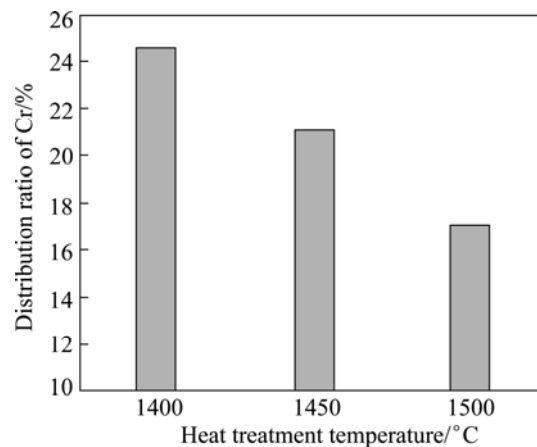


Fig. 5 Distribution ratio of Cr between Nbss and Nb_5Si_3 at different heat treatment temperatures

5 Conclusions

1) In the withdrawal rate range between 1.2 and 36 mm/min, the DS alloys are composed of the same phases: Nbss, $\alpha\text{-Nb}_5\text{Si}_3$, $\gamma\text{-Nb}_5\text{Si}_3$ and Cr_2Nb . However, the $\gamma\text{-Nb}_5\text{Si}_3$ disappears in the DS50 alloy. The microstructures of all DS specimens are composed of primary Nb_5Si_3 laths, Nbss/ Nb_5Si_3 eutectic clusters or cells, Cr_2Nb and Ti-rich Nbss phase around Nbss/ Nb_5Si_3 eutectics.

2) The Nbss/ Nb_5Si_3 eutectics present three types of morphologies which change from eutectic I to eutectic III with the increase of withdrawal rate. In the DS50 alloy, the microstructure is refined; however, the higher withdrawing rate results in the severer micro-segregation.

3) As the heat treatment temperature increases, the compositions of component phases are more homogeneous and the microstructure of the alloy becomes further smooth and rounded, but lost lamellar morphologies totally at 1500 °C. Heat treatment at 1450 °C for 10 h possesses optimal microstructure for the alloy.

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定向凝固过共晶 Nb–Si 基合金的组织优化

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摘 要: 采用液态金属冷却定向凝固炉制备 Nb–16Si–24Ti–10Cr–2Al–2Hf 合金, 凝固速率分别为 1.2、6、18、36、50 mm/min, 随后对定向凝固速率为 50 mm/min 的合金进行(1400 °C, 10 h), (1450 °C, 10 h)和(1500 °C, 10 h)的热处理。研究了定向凝固速率和热处理温度对合金微观组织的影响。结果表明: 合金的定向凝固组织主要由沿着试棒轴向生长的初生 Nb₅Si₃ 相和耦合生长的 Nbss/Nb₅Si₃ 共晶胞组成, 在共晶胞边缘, 有少量的 Cr₂Nb 存在。横截面上共晶胞边界明显, 随着凝固速率的增加, 定向凝固组织明显细化, Nbss/Nb₅Si₃ 共晶胞形貌也发生变化。合金经过热处理, Nbss 连成基体, 部分 Cr₂Nb 相溶解, 微观成分偏析减小。经过(1450 °C, 10 h)热处理, 实现了对过共晶 Nb–Si 基合金的组织优化。

关键词: Nb–Si 合金; 定向凝固; 共晶; 抽拉速率; 组织; 热处理

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