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Effect of high temperature treatments on microstructure of Nb-Ti-Cr-Si based ultrahigh temperature alloy

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Abstract: To investigate the effects of homogenizing and aging treatments on the microstructure of an Nb-Ti-Cr-Si based ultrahigh temperature alloy, coupons were homogenized at 1 200−1 500 °C for 24 h, and then aged at 1 000 °C for 24 h. The results show that the heat-treated alloy is composed of Nb solid solution (Nbss), $(Nb,X)_5Si_3$ and Cr_2Nb phases. With the increase of heat-treatment temperature, previous Nbss dendrites transformed into equiaxed grains, and petal-like Nbss/$(Nb,X)_5Si_3$ eutectic colonies gradually changed into small $(Nb,X)_5Si_3$ particles distributed in Nbss matrix. A drastic change occurred in the morphology of the Laves phase after homogenizing treatment. Previously coarse Cr_2Nb blocks dissolved during homogenizing at temperature above 1 300 °C, and then much finer and crowded Cr_2Nb flakes precipitated in the Nbss matrix in cooling. Aging treatment at 1 000 °C for 24 h led to further precipitation of fine particles of Laves phase in Nbss matrix and made the difference in concentrations of Ti, Hf and Al in Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb phases reduced.

Key words: homogenizing treatment; aging treatment; Nb-Ti-Cr-Si based ultrahigh temperature alloy; microstructural evolution

1 Introduction

Nb-silicide based ultrahigh temperature alloys have been studied as alternative materials to Ni-based superalloys because of their higher melting temperature, lower density and higher temperature strength [1−2]. Although the comprehensive performance of the alloys has been improved constantly by multi-alloying [3−4], preparation improvement [5−7] and optimization of microstructure [8], there are gaps between their actual performance and the ideal goal. Recently, Nb-Ti-Cr-Si based alloys have attracted ever-increasing attentions, and their phase constituents are Nb solid solution (Nbss), $(Nb,X)_5Si_3$ and Cr_2Nb [9−10]. Among these three phases, Nbss is introduced to improve the ambient temperature fracture resistance, while the silicide and Laves phases are introduced to improve high temperature creep strength and oxidation resistance. In order to achieve a balance among properties of good creep strength, excellent oxidation resistance and acceptable fracture resistance, the volume fraction and the morphology of each constituent phase must be controlled reasonably. However, the arc-melted Nb-Ti-Cr-Si based alloy always possesses an inhomogeneous microstructure with some meta-stable phases [11−12]. Therefore, proper heat-treatment is necessary to eliminate or alleviate both the meta-stable phases and solute segregation in the alloy. Unfortunately, few reports about the effects of the high temperature heat treatment on the microstructure of Nb-Ti-Cr-Si based alloys can be found in open literatures, and it is still unclear how the microstructure changes in the multi-component and multi-phase alloy during heat treatments. The aim of the present work is to reveal the effects of homogenizing and aging treatments on the microstructure of Nb-Ti-Cr-Si based alloy.

2 Experimental

A multi-component Nb-Ti-Cr-Si based ultrahigh temperature alloy with nominal composition of Nb-29Ti-8Si-11Cr-5Hf-3Al-1.5B-0.06Y (molar fraction, %) was prepared in a high vacuum consumable arc-melting furnace with a water-cooled copper crucible under argon atmosphere. The ingot was remelted four times in order to homogenize its chemical composition.

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The samples were cut into cubes with dimensions of 8 mm×8 mm×8 mm by an electro-discharge machine from the ingot. Two-step heat treatments were employed and the coupons were firstly homogenized at 1 200, 1 300, 1 400 and 1 500 °C for 24 h, respectively, and subsequently aged at 1 000 °C for 24 h. Heat treatments were carried out in a high vacuum heat treatment furnace. The furnace chamber was heated up when the vacuum level in it was higher than 1.0×10^{-3} Pa. High-purity (99.99%) argon was let into the furnace chamber as a protective atmosphere when the temperature was higher than 1 000 °C. All specimens were furnace cooled. The phases were determined using a Panalytical X'Pert Pro X-ray diffraction (XRD) analyzer and the microstructure and the compositions of constituent phases were analyzed using a JSM−6460 scanning electron microscope with an energy dispersive spectroscope (Inca X-sight). The composition of some selected specimens was also analyzed in a JXA−8100 electron probe microanalyzer.

3 Results and discussion

3.1 Microstructure of arc-melted alloy

The XRD pattern of the arc-melted alloy is shown in Fig. 1. According to the XRD result, the phases of the arc-melted alloy are Nbss, $(Nb,X)_5Si_3$ (here X represents Ti, Hf and Cr) and Cr_2Nb Laves phases. The $(Nb,X)_5Si_3$ phase is found to have hexagonal hp16 Mn_5Si_3 type structure (named γ-$(Nb,X)_5Si_3$) and the Laves phase had C14 crystal structural. The meta-stable phase Nb_3Si is not found in the current study, which is in agreement with the research result of IEWARI et al in Nb-30Ti-8Si-10Cr-10Al-5Hf alloy [13−14]. Any substitution of Ti in Nb_3Si is expected to lower its crystallizing temperature; however, the crystallizing temperature of Nb_5Si_3 is affected appreciably by the addition of Ti because the temperatures for the crystallization of Nb_5Si_3 and Ti_5Si_3 phase are relatively similar [13]. Furthermore, Nb_5Si_3 phase has the higher liquidus temperature compared with other phases [14]. Once M_5Si_3 silicides form, M_3Si silicides would be suppressed. Therefore, high Ti content may affect the solidification path of the alloy. In addition, some research suggests that the formation of Nb_3Si depends on the Hf concentration in the alloy [13]. Higher solubility of group IV elements in the silicide phase could be another factor affecting the preferential formation of the M_5Si_3 type silicides over that of Nb_3Si type. Thus, the absence of the M_3Si type silicides in the present alloy can be explained on the basis of the higher formation temperature of M_5Si_3 silicide and the influence of Hf on stabilizing M_5Si_3 type silicide. It is also found that the silicide is only γ-$(Nb,X)_5Si_3$ without any other kind; however, the microstructure is different from that in Ref. [11], where it was found that β-$(Nb,X)_5Si_3$ and γ-$(Nb,X)_5Si_3$ are always present in the Nb-20Ti-18Si-5Cr-3Al-4Hf-1.5B-0.06Y(molar fraction, %) alloy. By comparing the composition of $(Nb,X)_5Si_3$ in the current alloy with that in Ref. [11], it is found that (Ti+Hf) concentration in $(Nb,X)_5Si_3$ of the present alloy is 1.5 times higher than that in Ref. [8], which infers that the synergy of Ti and Hf impose obvious effects on the structure of the $(Nb,X)_5Si_3$, and the hp16 Ti_5Si_3 type silicide could be stabilized other than β-$(Nb,X)_5Si_3$ with a higher (Ti+Hf) concentration. The experimental results in Refs. [3, 8] also confirmed this effect of (Ti+Hf) concentration on the formation of silicide.

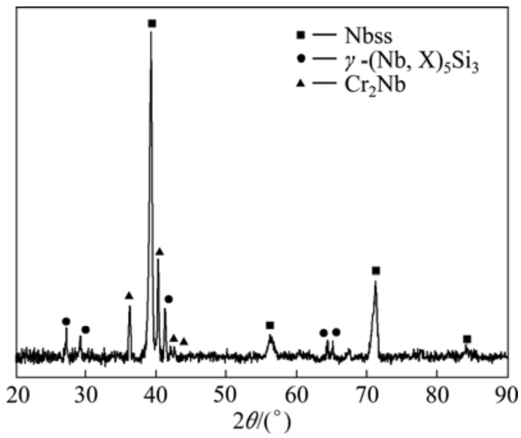

Fig. 1 XRD pattern of arc-melted Nb-Ti-Cr-Si based alloy

The BSE image of the arc-melted Nb-Ti-Cr-Si based ultrahigh temperature alloy is shown in Fig. 2(a). It can be seen that the microstructure of the arc-melted alloy is composed of non-faceted dendrites, lamellar or rod-like eutectic colonies and much finer eutectic colonies. According to the EDS data (Table 1), the primary non-faceted dendrites are Nbss, the lamellar or rod-like eutectic colonies are composed of Nbss and $(Nb,X)_5Si_3$, and the much finer eutectic colonies are composed of Nbss and Cr_2Nb (as shown in Fig. 2(b)).

From Table 1, it can be found that strong solute partitioning tendency for various elements exists among Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb phases. Hf preferentially partitioned into both silicide phase and Laves phase, while Cr and Al have tendency to solute into the Nbss matrix. Combining XRD pattern in Fig.1 with chemical composition data in Table 1, it can be concluded that the silicide phase and the Laves phase could be written as $(Nb,Ti,Cr,Hf)_5(Si,Al)_3$ and $(Cr,Si,Hf)_2(Nb,Ti)$, respectively.

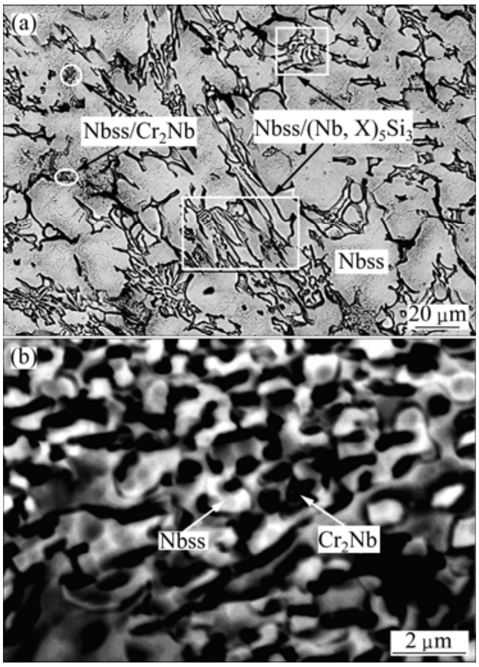

Fig. 2 Microstructure of arc-melted Nb-Ti-Cr-Si based alloy: (a) BSE image of arc-melted alloy; (b) Morphology of Nbss/Cr_2Nb eutectic colonies observed by EPMA

Table 1 Compositions of constituent phases in arc-melted Nb-Ti-Cr-Si based alloy

Phase	x(Al)/%	x(Si)/%	x(Ti)/%	x(Cr)/%	x(Nb)/%	x(Hf)/%
Nbss	4.83	4.28	30.68	9.20	47.96	3.05
$(Nb,X)_5Si_3$	3.09	34.97	22.69	1.21	3.42	7.62
Cr_2Nb	1.64	7.89	17.38	45.10	20.37	7.62

3.2 Microstructure after homogenizing treatment

The XRD patterns of homogenized specimens are shown in Fig. 3. It can be found that the constituent phases are still Nbss, γ-$(Nb,X)_5Si_3$ and Cr_2Nb. The homogenizing treatments do not influence the constituent phases. However, the oxide phase HfO_2 appears in the microstructure homogenized at 1 500 °C for 24 h. Furthermore, it can be found that γ-$(Nb,X)_5Si_3$ is very stable during homogenizing treatments and the transformation of γ-$(Nb,X)_5Si_3$→α-$(Nb,X)_5Si_3$ does not happen, which might be due to the addition of a relatively high content of Hf into the present alloy that can stabilize γ-$(Nb,X)_5Si_3$ phase [11].

The BSE images of the homogenized specimens are shown in Fig. 4. As shown in Fig. 4(a), after homogenizing treatment at 1 200 °C for 24 h, both the morphology and the amount of the eutectic colonies do not change apparently. These results indicate that this heat treatment temperature is too low to change the microstructure noticeably. Needle-like Laves phases distribute unevenly in Nbss matrix, as shown in

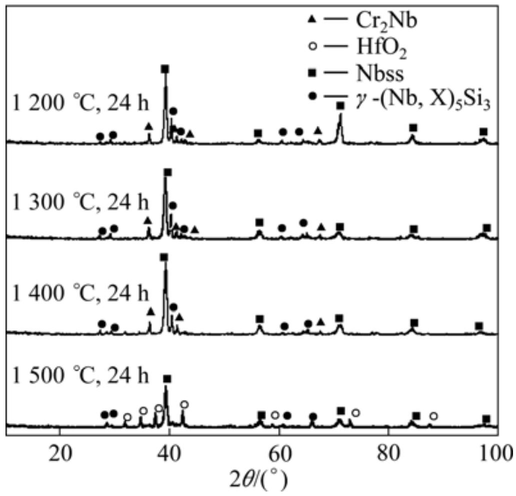

Fig. 3 XRD patterns of Nb-Ti-Cr-Si based alloy after homogenizing treatments

Fig. 4(a). After homogenizing treatment at 1 300 °C for 24 h, previously coarse silicide blocks with sharp interfaces convert into small size particles with relatively blunted and round interfaces, as shown in Fig. 4(b). The Laves phases are present along the boundaries between Nbss and $(Nb,X)_5Si_3$ phases. However, the Laves precipitates become a little coarser than those in the specimen homogenized at 1 200 °C for 24 h. As shown in Fig. 4(c), after homogenizing treatment at 1 400 °C for 24 h, examination of large areas of the specimens reveals that the previous coarse particles of Laves phase which still exists after homogenizing treatment at 1 300 °C for 24 h has dissolved and re-precipitated as much finer and crowded plates, which indicates that the coarse Laves blocks in the arc-melted alloy dissolves between 1 300 and 1 400 °C. Furthermore, some $(Nb,X)_5Si_3$ blocks coarsened after homogenizing treatment at 1 400 °C for 24 h, as shown in Fig. 4(c). However, after homogenizing treatment at 1 500 °C for 24 h, the faces of the previously cubic shape specimen were slightly distorted and the specimen experienced initial melting during the heat treatment.

The microstructure of specimen after homogenizing treatment at 1 500 °C for 24 h (Fig. 4(d)) was significantly different from that in the specimen homogenized below 1 400 °C. Coarsening occurred, and the Laves precipitates were present not only along boundaries of $(Nb,X)_5Si_3$ blocks but also within the Nbss matrix. The HfO_2 bright particles appeared along the boundaries of $(Nb,X)_5Si_3$ blocks.

Homogenization treatment is generally employed to remove solute segregation and any meta-stable phase formed during the solidification in the alloy. The homogenization of the arc-melted alloy produces two important changes in the microstructure: 1) the morphology of the $(Nb,X)_5Si_3$ blocks changed during

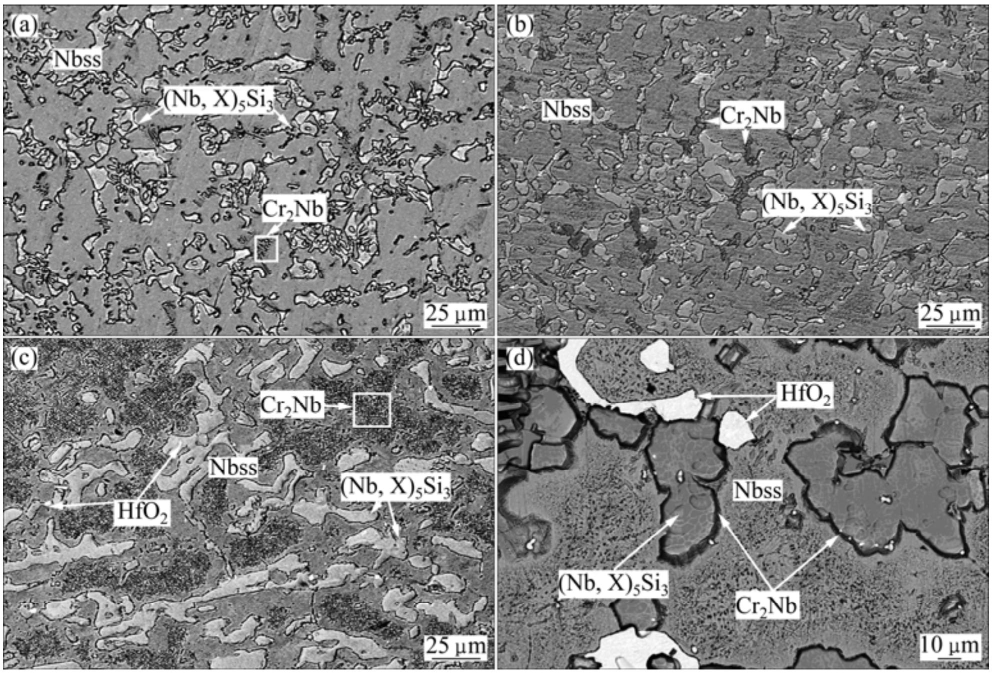

Fig. 4 BSE images of Nb-Ti-Cr-Si based alloy after homogenizing treatments: (a) 1 200 °C, 24 h; (b) 1 300 °C, 24 h; (c) 1 400 °C, 24 h; (d) 1 500 °C, 24 h

Table 2 Composition of constituent phases of Nb-Ti-Cr-Si based alloy after different homogenizing treatment

Homogenizing treatment	Phase	x(Al)/%	x(Si)/%	x(Ti)/%	x(Cr)/%	x(Nb)/%	x(Hf)/%	x(O)/%
1 200 °C, 24 h	Nbss	4.39	0.28	34.1	8.94	50.77	1.76	−
	$(Nb,X)_5Si_3$	3.41	30.65	24.89	1.52	32.51	7.01	−
	Cr_2Nb	1.81	5.82	12.77	51.4	22.22	5.98	−
1 300 °C, 24 h	Nbss	4.14	0.25	33.57	12.89	47.95	1.21	−
	$(Nb,X)_5Si_3$	3.25	30.64	24.6	0.99	33.05	7.63	−
	Cr_2Nb	1.82	5.62	13.52	51.46	23.13	4.51	−
1 400 °C, 24 h	Nbss	4.18	0.43	31.04	15.04	47.83	1.48	−
	$(Nb,X)_5Si_3$	3.12	30.31	25.1	2.17	32.69	6.64	−
	Cr_2Nb	1.84	1.17	16.49	46.45	29.87	4.18	−
1 500 °C, 24 h	HfO_2	0	2.31	0.18	0	0.52	25.97	71.02
	Nbss	4.45	1.62	24.54	12.35	55.95	1.13	−
	$(Nb,X)_5Si_3$	2.93	29.55	23.22	4.03	38.01	2.26	−

homogenizing treatments. The previous Nbss/$(Nb,X)_5Si_3$ eutectic colonies in the arc-melted alloy broke gradually into small $(Nb,X)_5Si_3$ particles in Nbss matrix with increasing homogenizing temperature. 2) The morphology of the precipitates of Laves phase Cr_2Nb changed apparently from blocks to needles and more Cr_2Nb needles precipitated in Nbss with increasing homogenizing temperature. The previously fine Nbss/Cr_2Nb eutectic colonies in arc-melted alloy broke into needle-like Cr_2Nb precipitates in Nbss matrix during homogenizing treatment at 1 200 °C for 24 h; however, the needle-like Cr_2Nb coarsened during homogenizing treatment at 1 300 °C for 24 h, resulting in coarse Laves phase particles appearing with medium gray contrast usually contact with silicides. Whereas, in the specimen homogenized at 1 400 °C for 24 h, the coarse Laves particles disappear and re-precipitate in cooling in the form of finer and barely discernible platelets. These observations clearly indicate that the previously coarse Laves phase dissolves between 1 300 and 1 400 °C and re-precipitates in cooling in the Nbss matrix.

Based on these observations, the morphology and distribution of the various phases in the Nb-Ti-Cr-Si based alloy dramatically change with the increase of homogenizing temperatures. The effects of Cr addition into the Nb-Si based alloys were demonstrated by many studies. ZHAO et al [15] studied Nb-Cr-Si phase diagram using diffusion multiples and pointed out that

Nb, Nb_5Si_3 and NbCrSi phases form three-phase equilibrium and the Laves phase was not in equilibrium with Nb and Nb_5Si_3 in the ternary between 1 000 and 1 200 °C. ZHAO et al [15] appreciated that there was a four phase equilibrium of Nb+Nb_5Si_3+NbCrSi+C14 Laves phase at temperature above 1 200 °C by suitable heat treatment. However, it was suggested that the phase transformation from C14 Laves phase to the NbCrSi phase was very sluggish. But GENG [3] suggested that Nb and Nb_5Si_3 were in equilibrium with Cr_2Nb at 1 500 °C for 100 h. The phase was composed of Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb from 1 200 to 1 500 °C. It can be inferred that phase equilibrium was influenced by the added alloying elements in the alloy.

From Table 3, it can be seen that the partitioning tendency of all the elements does not change after different homogenizing treatments, but the concentrations of Ti, Cr, Al and Hf in Nbss vary with the increase of homogenizing temperature. It can be seen that the concentration of Ti in Nbss decreases significantly whereas the concentration of Cr increases with increasing homogenizing temperature. It indicates that the solubilities of Ti and Cr are strongly temperature dependent. However, the behaviors of Ti and Cr in the Nbss in these results are different from those in Ref. [3], in which an increasing Ti concentration in Nbss was accompanied by the increase of Cr concentration and the contents of Al and Hf in the Nbss did not change obviously.

3.3 Microstructure after aging treatment

Subsequent to the homogenization treatment, samples were subjected to aging treatment at 1 000 °C for 24 h. The XRD patterns of aging treatment specimens are shown in Fig. 5. It is found that constituent phases after aging treatment at 1 000 °C for 24 h are similar to those of homogenizing treatment, only the intensity and number of diffraction peaks of Laves phase increase. It infers that aging treatment does not influence the constituent phases instead of the volume fraction of Laves phase.

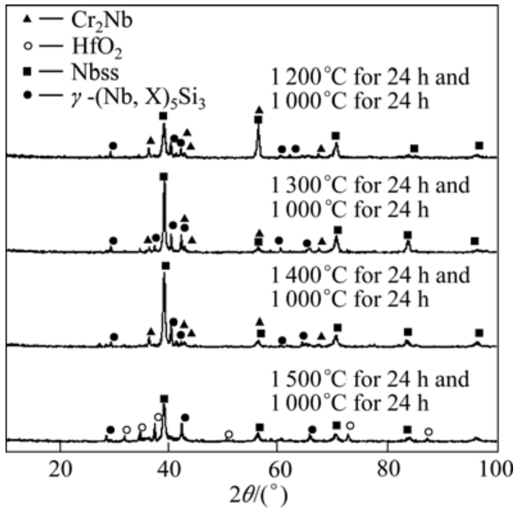

Fig. 5 XRD patterns of Nb-Ti-Cr-Si based alloy after homogenizing and aging treatments

The microstructure and compositions of phases after aging treatment at 1 000 °C for 24 h are shown in Fig.6 and Table 3, respectively. After being homogenized at 1 200, 1 300, 1 400 and 1 500 °C for 24 h and then aging treated at 1 000 °C for 24 h, respectively, these alloys produce two important changes in the microstructure: 1) fine particles of Laves phase in Nbss matrix further precipitate; 2) the difference in concentrations of Ti, Hf and Al in Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb phases reduces. With regard to point 1, these fine precipitated phases are found to be richer in Cr and Ti than those of the coarse Laves phase in the alloy after homogenizing treatment at 1 300 °C for 24 h. These results are in agreement with the report by TEWARI et al [16], and

Table 3 Compositions of constituent phases of Nb-Ti-Cr-Si based alloys after aging treatments

Aging treatment	Phases	x(Al)/%	x(Si)/%	x(Ti)/%	x(Cr)/%	x(Nb)/%	x(Hf)/%	x(O)/%
(1 200 °C, 24 h)+(1 000 °C, 24 h)	$(Nb, X)_5Si_3$	2.86	34.36	22.14	1.06	31.93	7.66	−
	Cr_2Nb	1.49	6.52	14.48	45.25	24.39	7.87	−
	Nbss	4.71	0.59	31.76	6.54	54.59	1.81	−
(1 300 °C, 24 h)+(1 000 °C, 24 h)	$(Nb, X)_5Si_3$	3.11	34.55	21.04	1.21	32.06	8.03	−
	Cr_2Nb	1.48	7.04	14.21	46.87	25.43	4.9	−
	Nbss	4.79	0.95	30.75	6.15	55.65	1.7	−
(1 400 °C, 24 h)+(1 000 °C, 24 h)	$(Nb, X)_5Si_3$	2.84	34.54	22.88	1.43	30.33	7.98	−
	Cr_2Nb	1.58	1.62	23.08	45.99	23.74	3.99	−
	HfO_2	0	1.01	0	0	1.07	32.87	65
(1 500 °C, 24 h)+(1 000 °C, 24 h)	Nbss	4.11	1.5	26.34	12.55	54.64	0.85	−
	$(Nb, X)_5Si_3$	3.41	32.31	22.83	1.22	37.3	2.93	−

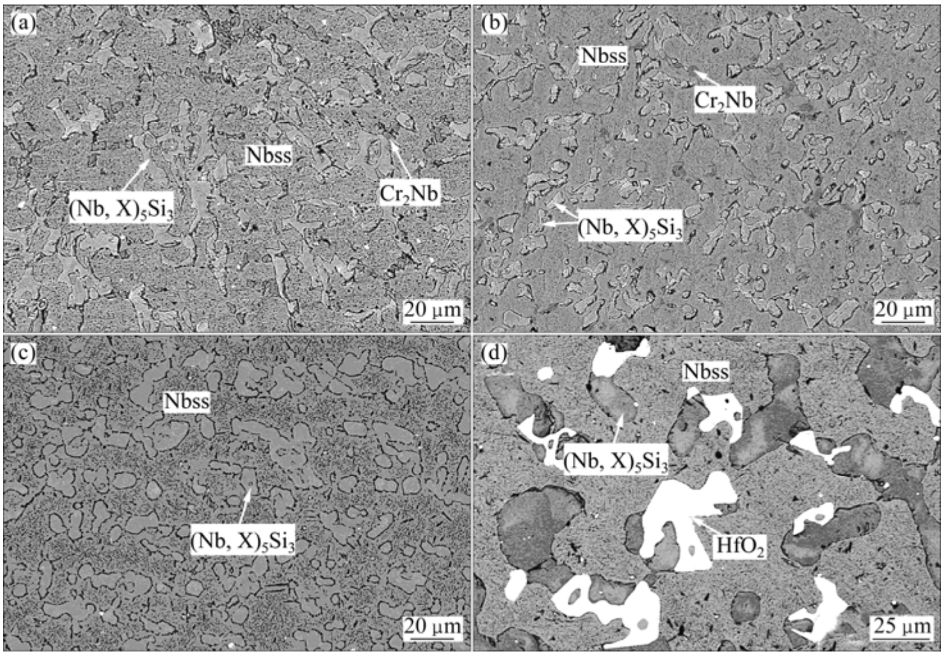

Fig. 6 BSE images of Nb-Ti-Cr-Si based alloy after aging treatments: (a) 1 200 °C for 24 h and 1 000 °C for 24 h; (b) 1 300 °C for 24 h and 1 000 °C for 24 h; (c) 1 400 °C for 24 h and 1 000 °C for 24 h; (d) 1 500 °C for 24 h and 1 000 °C for 24 h

the fine Laves phase is also influenced by some other factors. The precipitated Laves phase is always observed in the alloy with Cr concentration higher than 7%, and addition of Hf might promote the formation of fine precipitates of the Laves phase [14]. Refs. [14−15] indicate that the formation of Laves phase during aging treatment at 1 000 °C for 24 h is important because they can provide strength to the soft Nbss matrix and provide an easy path for crack nucleation and propagation and thereby reduce the ductility appreciably [14−15].

The second important issue concerned the difference in the concentrations of Ti, Hf and Al in Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb phases being reduced during aging treatment at 1 000 °C for 24 h. It can be seen from Table 3 that Cr content in Nbss is approximately 6.15%−6.86% (molar fraction), the contents of Ti, Cr, Al and Hf in Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb are almost similar to those of the specimen after heat treatment at 1 500 °C for 24 h and 1 000 °C for 24 h.

It should be noticed that specimens after heat treatment at 1 500 °C for 24 h and 1 000 °C for 24 h bring about significant change in the microstructure and composition of various phases (as shown in Fig. 6(d) and Table 3). However, the Laves phase along the boundaries of $(Nb,X)_5Si_3$ during homogenizing treatment at 1 500 °C for 24 h is not found after heat treatment at 1 500 °C for 24 h and then 1 000 °C for 24 h. It is concluded that the Laves phase is dissolved during aging treatment, indicating that the Laves phase is extraordinary instable.

The microstructure near the edges of the specimen after heat treatment at 1 500 °C for 24 h and 1 000 °C for 24 h is shown in Fig. 7, in which HfO_2 and TiN exhibit white and black contrasts, respectively. According to the WDS microanalyses of these nitrides, the contents of Ti, N and Nb are 57.51%, 40.25% and 2.24%, respectively. The formation of TiN in the alloy is attributed to the strong nitride forming tendency of Ti at elevated temperature.

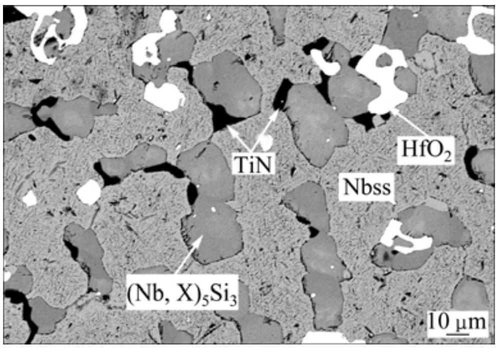

Fig. 7 BSE image of TiN and HfO_2 formed near edge of specimen after heat treatment at 1 500 °C for 24 h and then 1 000 °C for 24 h

4 Conclusions

1) The heat-treated Nb-Ti-Cr-Si based ultrahigh temperature alloy is composed of Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb after various homogenizing and aging treatments. HfO_2 is found in the alloy after heat treatment both at 1 500 °C for 24 h and 1 500 °C for 24 h then 1 000 °C

for 24 h.

2) With the increase of heat-treatment temperature, previous Nbss dendrites in the arc-melted alloy transform into equiaxed crystals. The previous Nbss/$(Nb,X)_5Si_3$ eutectic colonies break into small $(Nb,X)_5Si_3$ blocks in Nbss matrix, whereas the previous Nbss/Cr_2Nb eutectic colonies in arc-melted microstructure transform into needle-like Laves precipitates in Nbss matrix after homogenizing treatment at 1 200 °C for 24 h and become coarse Laves blocks after homogenizing at 1 300 °C for 24 h. The previous Laves Cr_2Nb particles dissolve during homogenizing treatment at 1 400 °C for 24 h, and much finer and crowded Cr_2Nb platelets form during cooling. These observations suggest that the previous coarse Laves phase particles dissolve between 1 300 and 1 400 °C.

3) Aging at 1 000 °C for 24 h after homogenizing treatments improves the precipitation of fine needle-like Cr_2Nb in Nbss matrix and reduces the difference in concentrations of Ti, Hf, and Al in Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb phases.

References

[1] MENDIRATTA M G, DIMIDUK D M. Phase relations and transformation kinetics in the high Nb region of the Nb-Si system [J]. Scripta Metallurgica et Materialia, 1991, 25(1): 237−243.

[2] YANG Y, BEWLAY B P, CHANG Y A. Liquid-solid phase equilibria in Metal-Rich Nb-Ti-Hf-Si alloys [J]. Phase Equilibria and Diffusion, 2007, 28(5): 107−114.

[3] GENG J, TSAKIROPOULOS P, SHAO G S. A study of the microstructures and oxidation of Nb-Si-Cr-Al-Mo in situ composites alloyed with Ti, Hf and Sn [J]. Intermetallics, 2007, 15(3): 382−395.

[4] GUO Hai-sheng, GUO Xi-ping, JIA Li-na, WU Xing-jun, DING Xu, GAO Li-mei, REN Jia-song. Effects of compositions and melting processes on the microstructure of multicomponent Nb-Si based super-high temperature alloys [J]. Rare Metal Materiala and Enginering, 2008, 37(7): 1299−1303. (in Chinese)

[5] YAO Cheng-fang, GUO Xi-ping, GUO Hai-sheng. Microstructural characteristics of integrally directionally solidified Nb-Ti-Si base ultrahigh temperature alloy with crucibles [J]. Acta Metallurgica Sinica, 2008, 44(5): 579−584. (in Chinese)

[6] WANG Yong, GUO Xi-ping. Effect of solidifying rate on integrally directionally solidified microstructure and solid/liquid interface morphology of an Nb-Ti-Si based alloy [J]. Acta Metallurgica Sinica, 2010, 46(4): 506−512. (in Chinese)

[7] GUO Xi-ping, GUAN Ping, DING Xu, ZHANG Jun, KUSABIRAKI K, FU Heng-zhi. Microstructure and mechanical properties of an advanced Niobium based ultrahigh temperature alloy [J]. Materials Science Forum, 2005, 539−543(4): 3690−3695.

[8] GENG J, SHAO G S, TSAKIROPOULOS P. Study of three-phase equilibrium in the Nb-rich corner of Nb-Si-Cr system [J]. Intermetallics, 2006, 14(7): 832−837.

[9] SHA Jiang-bo, YANG Chun-yan, LIU Jun. Toughening and strengthening behavior of an Nb-8Si-20Ti-6Hf alloy with addition of Cr [J]. Scripta Materialia, 2010, 62(11): 859−862.

[10] YANG Chun-yan, CHEN Ying, SHA Jiang-bo. Effect of Cr addition on microstructures and mechanical properties of an Nb-16Si-22Ti-2Al-2Hf alloy at room and high temperatures [J]. Chinese Journal of Aeronautics, 2010, 31(9): 1893−1899. (in Chinese)

[11] GUO Hai-sheng, GUO Xi-ping. Effects of homogenizing and aging treatments on the microstructure and microhardness of Nb-silicide based ultrahigh temperature alloy [J]. Int J Mater Res, 2010, 101(7): 900−906.

[12] JIA Li-na, GUO Xi-ping, YI Liang-yu. Effects of heat treatments on the microstructure of Nb-Si based in-Situ composites [J]. Rare Metal Materials and Enginering, 2008, 37(5): 878−882.

[13] TEWARI R, SONG H, DEY G K, CHATTERJEE A, VASUDEVAN V K. Microstructural evolution in Niobium-based alloys [J]. Metallurgical and Materials Transactions A, 2008, 39(7): 1506−1518.

[14] TEWARI R, SONG H, CHATTERJEE A. Microstructural characterization of multicomponent Nb-Ti-Si-Cr-Al-X alloys [J]. Metallurgical and Materials Transactions A, 2006, 37(9): 2669−2682.

[15] ZHAO J C, JACKSON M R, PELUSO L A. Determination of the Nb-Cr-Si phase diagram using diffusion multiples [J]. Acta Materialia, 2003, 51(20): 6395−6405.

[16] DAVID N, CARTIGNY Y, BELMONTE T, FIORANI J M, VILASI M. Thermodynamic description of the Cr-Nb-Si isothermal section at 1473 K [J]. Intermetallics, 2006, 14(4): 464−473.

高温热处理对 Nb-Ti-Cr-Si 基超高温合金显微组织的影响

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摘　要：为了研究高温均匀化及时效热处理对 Nb-Ti-Cr-Si 基超高温合金显微组织的影响，对样品进行均匀化处理，于 1 200−1 500 °C 保温 24 h，随后于 1 000 °C 保温 24 h 进行时效。结果表明，热处理后的组织主要由 Nbss、$(Nb,X)_5Si_3$ 和 Cr_2Nb 组成。随着均匀化处理温度的升高，电弧熔炼态的树枝状 Nbss 转变为等轴状，原先花瓣状的 Nbss/$(Nb,X)_5Si_3$ 共晶组织消失，转变为分布于 Nbss 基体上的小块状$(Nb,X)_5Si_3$ 组织。Cr_2Nb 的形貌随均匀化处理温度的升高而发生明显变化。当均匀化处理温度达到 1 300 °C 以上，原先粗大的 Cr_2Nb 发生溶解，在随后的冷却过程中在 Nbss 基体上沉淀析出细小、密集的针状 Cr_2Nb。经高温均匀化和时效复合处理后，Nbss 基体上析出更为细小、密集的沉淀相 Cr_2Nb，使得 Nbss、$(Nb,X)_5Si_3$ 和 Cr_2Nb 相中 Ti、Hf 和 Al 元素的含量差别缩小。

关键词：均匀化处理；时效处理；Nb-Ti-Cr-Si 基超高温合金；组织变化

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