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Microstructure and Microhardness upon High Temperature Heat Treatments in an Nb-Ti-Cr-Si Based Ultrahigh Temperature Alloy

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Abstract: In order to investigate the effects of high temperature heat treatments on the microstructures and microhardness of an Nb-Ti-Cr-Si based ultrahigh temperature alloy, coupons were homogenized at 1200~1500°C for 24h and then aged at 1000°C for 24h. The results show The Nb-Ti-Cr-Si based ultrahigh temperature alloy is composed of Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb phases after various homogenizing and aging treatments. HfO_2 is found in the alloy after heat treatment both at 1500°C for 24 h and 1500°C for 24 h then 1000°C for 24 h. With increase in heat-treatment temperature, previous Nbss dendrites in the arc-melted alloy transform into Nbss equaxied 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 after homogenizing treatment at 1200°C for 24 h and become coarse Laves blocks after homogenizing at 1300°C for 24 h. The previous Laves Cr_2Nb particles dissolve into Nbss during homogenizing treatment at 1400°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 1300 and 1400°C, and the Ti and Cr concentrations decrease with increase in heat-treatment temperatures. Aging at 1000°C for 24h after homogenizing treatments improves the precipitation of fine needle-like Cr_2Nb in Nbss matrix and Cr concentration in Nbss reduces. The variation of partitioning ratios of alloying elements among Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb causes the change in microhardness of Nbss and $(Nb,X)_5Si_3$. The microhardness of Nbss and $(Nb,X)_5Si_3$ reaches the maximum value after 1300°C/24h+1000°C/24h heat treatment.

Key words: Homogenizing treatment; Aging treatment; Nb-Ti-Cr-Si based ultrahigh temperature alloy; Partitioning ratios; Microhardness

INTRODUCTION

Nb-silicide based ultrahigh temperature alloys have been studied as alternative materials to Ni-based superalloys because of their high melting points, low densities and favorable mechanical properties. However, a major barrier to the practical applications is their poor high temperature 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.

It was found that addition of a higher content of Cr improved the high temperature oxidation resistance due to formation of the Laves phase, so a new kind of Nb–Ti–Cr–Si based alloys have attracted ever increasing attentions. Their phase constituents are Nb solid solution (Nbss), $(Nb,X)_5Si_3$(X represents Ti 、Hf and Cr elements) and Cr_2Nb. 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.

However, the arc-melted Nb-Ti-Cr-Si based alloys always possess an inhomogeneous microstructure with some meta-stable phases due to the high cooling speed during solidification [10]. Therefore, heat-treatment is necessary to eliminate or alleviate both meta-stable phases and solute segregation in these alloys. In addition, reasonable heat treatments can also optimize the microstructures and enhance their mechanical properties. The purpose of the present paper is to reveal the effects of homogenizing and aging treatments on the microstructure and microhardness of an Nb-Ti-Cr-Si based ultrahigh temperature alloy.

2. EXPERIMENTAL PROCEDURES

A multi-component Nb-Ti-Cr-Si based ultrahigh temperature alloy with the nominal composition of Nb-29Ti-8Si-11Cr-5Hf-3Al-1.5B-0.06Y (at. %) was prepared by a high vacuum consumable arc-melting furnace with a water cooled copper crucible under argon atmosphere. The ingot was remelted for four times in order to homogenize the chemical composition. Coupons with the size of $8\times8\times8mm^3$ used for microstructure characterization and heat treatments were cut from the master alloy ingot by electro discharge machining.

Two step heat treatments were employed and they were firstly homogenizing treatments at 1200, 1300, 1400 and 1500°C for 24h respectively and subsequently aging treatments at 1000°C for 24h. 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 wt %) argon was let into the furnace chamber as a protective atmosphere when the temperature was higher than 1000°C. All samples were furnace cooled. X-ray diffraction (XRD) was used to identify the phase present in the alloys and to calculate lattice parameters of the Nbss.

We used a Philips diffractometer with monochromatic CuK_{α}(λ=1.540562Å) radiation. The crystal structures of individual phase were identified by matching the characteristic XRD peaks against JCPPS data. The Nelson-Rilay extrapolation method was used to determine the lattice parameter. The microstructures were observed by scanning electron microscopy (SEM), and the chemical compositions of the constituent phases were analysed by a JSM-6460 scanning electron microscope equipped with an energy dispersive X-ray analyzer (EDXA). The Vickers microhardness of each phase was measured using an HXP-1000TM hardness machine with a load of 0.1g. The microhardness values were the average of at least 10 indentations.

3. RESULTS AND DISCUSSION

3.1 Arc-melted microstructure: Figure 1 shows XRD pattern of the arc-melted alloy studied in this work. XRD result indicated that the microstructure of the arc-melted alloy consisted of niobium solid solution Nbss, $(Nb,X)_5Si_3$ (here X presented Ti, Cr, Al and Hf) and Cr_2Nb Laves phase.

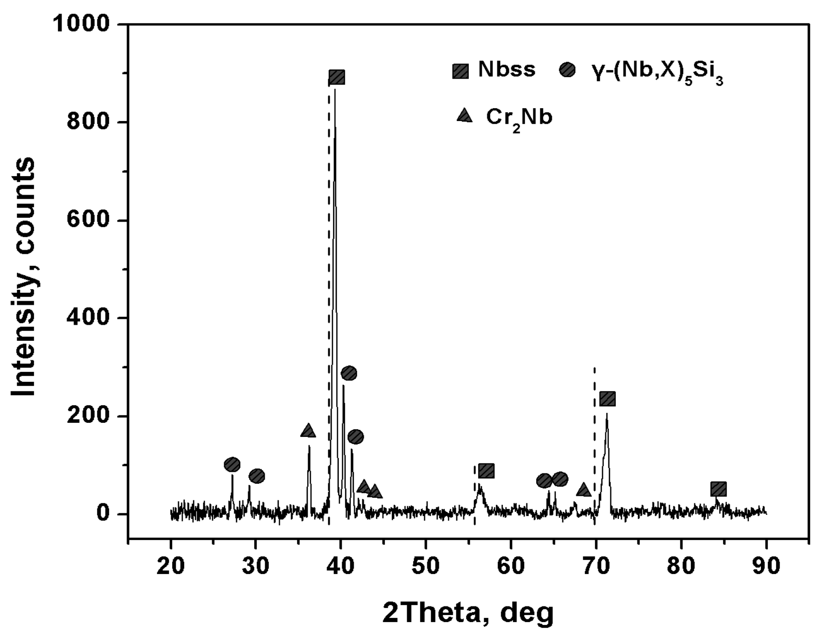

Figure 1: X- ray diffraction pattern of the arc-melted Nb-Ti-Cr-Si based ultrahigh temperature alloy

The BSE image in Figure 2 reveals that microstructures of the arc-melted alloy are composed of bright dendrites, lamellar or rod-like eutectic colonies and much finer eutectic colonies. According to EDXA data and the results of XRD, the dendrites are Nbss, the lamellar or rod-like eutectic colonies are composed of Nbss and $(Nb,X)_5Si_3$, and fine eutectic colonies are composed of Nbss and Cr_2Nb. It was noticed that the chemical composition of Nbss in Nbss/Cr_2Nb eutectic colonies is richer in Ti and Cr elements than that in primary Nbss.

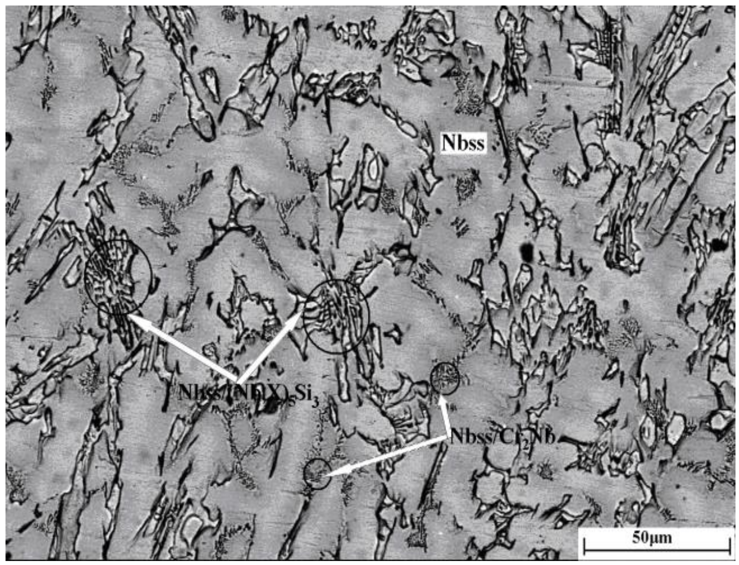

Figure 2: Microstructure of the arc-melted Nb-Ti-Cr-Si based ultrahigh temperature alloy

Effects of homogenizing and aging treatments on phase constituents: Figure 3 and Figure 4 show the XRD patterns of homogenized and aged specimens respectively. It can be seen that the constituent phases are still Nbss, γ-$(Nb,X)_5Si_3$ and Cr_2Nb. However, the phases are composed of Nbss, γ-$(Nb,X)_5Si_3$ and HfO_2 after heat treatment both at 1500°C/24h and 1500°C/24h+1000°C/24h. Furthermore, it can be found that γ-$(Nb,X)_5Si_3$ was very stable and the transformation of γ-$(Nb,X)_5Si_3$→α-$(Nb,X)_5Si_3$ didn't happen, which might be due to addition of a relatively high content of Hf in the present alloy that stabilized γ-$(Nb,X)_5Si_3$ phase.

In addition, Cr_2Nb phases were not detected at 1500°C/24h and 1500°C/24h+1000°C/24h, which suggested that Cr_2Nb phase were dissolved at temperature 1500°C. Compared the XRD results of homogenizing treatments to those of aging treatments, it can be found that the intensity and numbers of diffraction peak of Laves phase increased due to further much finer Laves phase precipitates in the Nbss matrix after specimens being aging treatment at 1000°C/24h.

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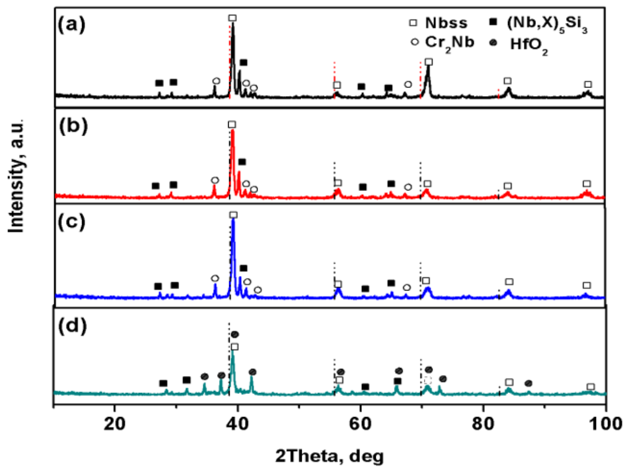
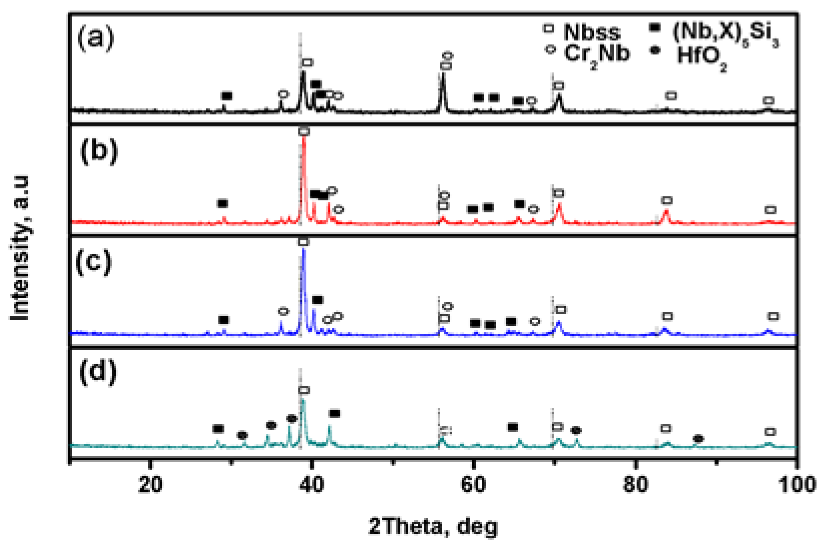


(a) 1200°C/24h (b) 1300°C/24h (c) 1400°C/24h (d) 1500°C/24h

Figure 3: X-ray diffraction of the Nb-Ti-Cr-Si based ultrahigh temperature alloy after homogenizing treatment

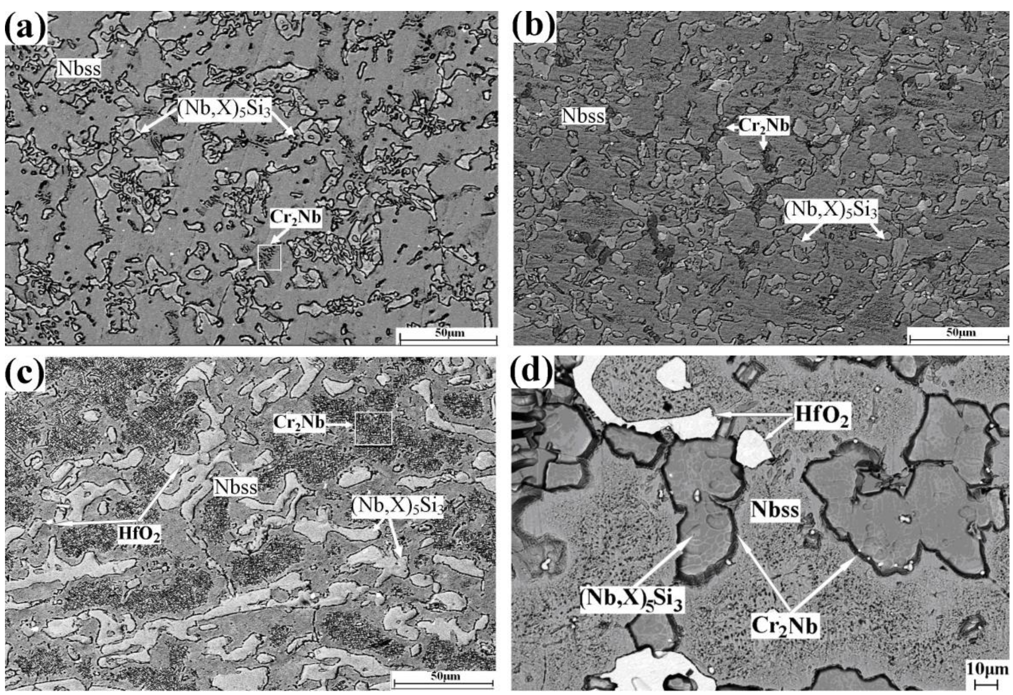


(a) 1200°C/24h (b) 1300°C/24h (c) 1400°C/24h (d) 1500°C/24h

Figure 4: X-ray diffraction of the Nb-Ti-Cr-Si based ultrahigh temperature alloy after homogenizing and then aging treatments

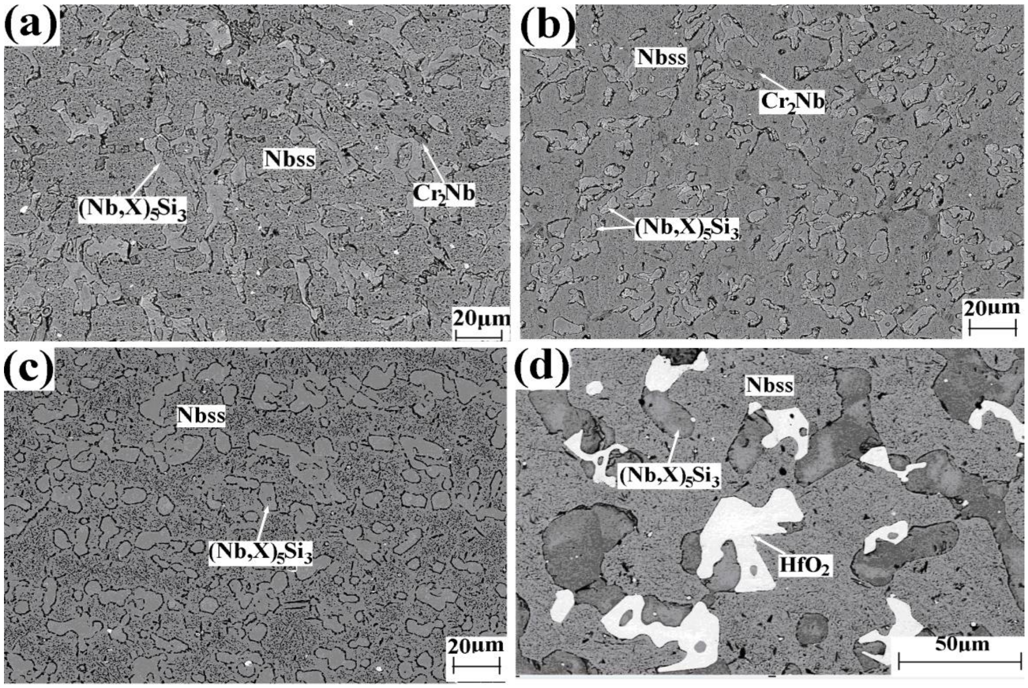
3.3 Effects of homogenizing and aging treatments on microstructures: The typical microstructures of Nb-Ti-Cr-Si based alloys after homogenizing and aging treatment are shown in **Figure 5 and Figure 6.** It can be seen from Figure 6(a), the microstructure in the specimens homogenized at 1200°C/24h is similar to that of arc-melted alloy, which indicated that this heat treatment temperature, 1200°C, was too low to change the microstructure. However, after homogenizing treatment at 1300°C/24h, the rod-like Nbss/$(Nb,X)_5Si_3$ eutectic colonies dissolved into small silicide blocks with receptively blunted and round interface in Nbss matrix.

The previously needle-like Laves phase became a litter coarser blocks in contact with the silicides, and its volume fraction increased. Comparison of the microstructures of specimen homogenized at 1400°C/24h and 1300°C/24h, it was revealed a dramatic change in the morphology and distribution of the Laves phase. In the specimen homogenized at 1400°C/24h, the coarser block Laves phase which were present in the specimen homogenized at 1300°C/24h have disappeared and reprecipitated as much finer and crowded plates, which indicated that the previously coarse Laves blocks dissolved between 1300 and 1400°C. It was also found that the smaller silicide blocks converted into long continuous plate shaped microstructures, and previously many smaller blocks silicides might be integrated together as a result of forming the continuous microstructure. With respect to specimen homogenized at 1500°C/24h, the microstructure was significantly different from that of other specimens, and the Laves precipitates were present not only along boundaries of $(Nb,X)_5Si_3$ blocks but also in the Nbss matrix. The HfO_2 bright particles appeared along the boundary of $(Nb,X)_5Si_3$ blocks, as shown in **Figure.5(d).**



(a) 1200°C/24h (b) 1300°C/24h (c) 1400°C/24h (d) 1500°C/24h

Figure 5: BSE images of the Nb-Ti-Cr-Si based ultrahigh temperature alloy after homogenizing treatments



(a) 1200℃/24h+1000℃/24h (b) 1300℃/24h℃+1000℃/24h

(c) 1400℃/24h+1000℃/24h (d) 1500℃/24h+1000℃/24h

Figure 6: BSE images of the Nb-Ti-Cr-Si based ultrahigh temperature alloy after homogenizing and then aging treatments

From **Figure 6**, it can be found that the microstructures in specimens aged at 1000℃/24h are similar to those in the correspond homogenized specimens, and the most striking change was the presence of finer precipitates in the Nbss matrix. These finer precipitates were found to be rich in Cr and Ti. Because of the very small size of these precipitates, it was difficult to accuracy ascertain their chemical composition of these precipitates by SEM-EDS. According to the results of reference [9], these precipitates were another Laves phase.

Based on these observations, the arc-melted alloy after heat treatment produced two important changes in the microstructures: 1) the silicide phase was quite stable during the heat treatments, only its morphology changed with the increase in heat treatment temperatures. The previously Nbss/$(Nb,X)_5Si_3$ eutectic colonies in the arc-melted alloy broke up gradually into small $(Nb,X)_5Si_3$ particles in Nbss matrix, and then formed a continuous long plate shaped microstructure with the increase in homogenized temperatures. 2) A dramatic change in the morphology and distribution of the Laves phase was occurred after heat treatments. There were three kinds of Laves phases, such as coarse Laves phase, finer precipitated Laves phase during homogenizing treatment at 1400℃/24h and reprecipitated Laves phase during aging treatment at 1000℃/24h. The coarse Laves phase appeared usually in contact with silicides (As shown in Figure 5(a), (b), (c) (d)), suggesting that the silicide-Nbss matrix interface was effective in reducing the surface energy barrier for nucleation of this phase. Furthermore, as the silicide phase has little or no tolerance for Cr, rejection and accumulation of Cr atoms to a level higher than the solubility limit in the Nbss led to formation of the Laves phase along the silicide-Nbss interface. However, the cross-road finer precipitated Laves phase was only present in the central region of the Nbss matrix (As shown in Figure 5(c)), which indicated that the coarser Laves dissolved between 1300°C and 1400°C, and formed on cooling by solid-state precipitation in the Nbss matrix. The reprecipitated Laves phase after aging treatment at 1000°C/24h is smaller than that of finer precipitated Laves phase, and these finer reprecipitates were present within and along the grain boundaries of Nbss matrix (As shown in Figure 6). However, these finer precipitates disappeared beyond 1200°C, which indicated that they were different from the coarse Laves phase in both arc-melted and homogenized specimens. Thus, it was concluded that these finer precipitates had a dissolution temperature above 1200°C, which is in good agreement with the results in reference [9]. The found that theses finer precipitates were absent above 1100°C, which suggested that the Cr concentration in the Nbss matrix reached a saturation at 1100°C in these alloys, which in turn lead to the precipitation of the Laves phase at lower aging temperature below 1100°C.

3.4 Effects of homogenizing and aging treatments on the lattice parameters of the Nbss: In addition, from Figure 1, Figure 3 and Figure 4, it is easily seen that the diffraction peaks of Nbss both in the arc-melted alloy and the heat treated alloys are shifted to the right (higher 2θ values) peaks expected for pure niobium, which would suggest that the Nbss phases in these alloys have reduced lattice parameter. The estimated lattice parameters of Nbss are shown in Figure 5, where the data of pure niobium (3.303A, JCPDS34-370) are also given for comparison. The calculated results (As shown Figure 8) also confirmed that the lattice parameters of Nbss were smaller than that of pure niobium.

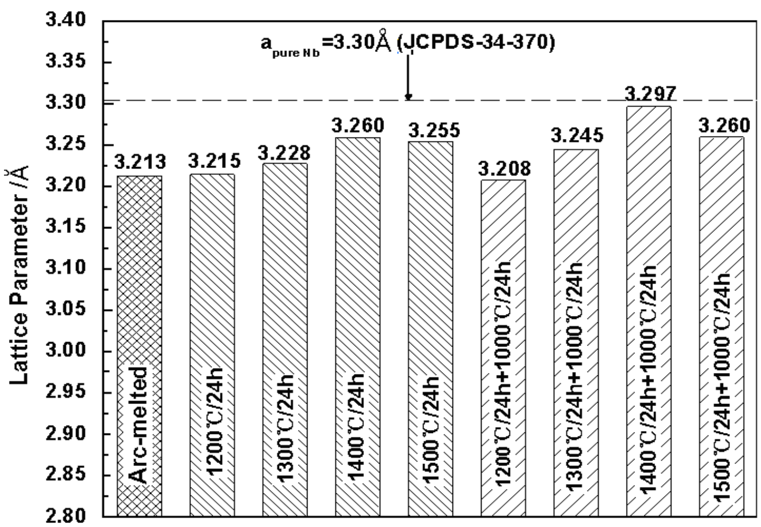


Fig. 8: Calculated lattice parameters of the Nbss in Nb-Ti-Cr-Si based ultrahigh temperature alloy from its diffraction peaks in XRD patterns in Fig.1, Fig.3 and Fig.4

The contracted Nbss lattice parameter would be significantly attributed to substitution of Al and Cr for Nb. Although the Hf dissolved into Nbss probably expands the Nbss lattice, the influence of the Cr and Al substitution would dominate over that of Hf. It can be seen from Figure 5, the lattice parameters of

Nbss have a increased tendency with the increase in temperatures during heat treatments, which suggested that the heat treatments signified the redistribution of solutes among Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb. The increase of Nbss lattice parameter can be explained on the basic of reducing the Cr and Al concentration in Nbss with the increase in heat treatment temperature. In addition, it was reported that Si concentration in Nbss could be affected the lattice parameters of Nbss, although the solubility limit of Si into Nb is less than 0.5at% at 1873K according to the Nb-Si binary phase diagram, a small amount of Si may remarkably reduce the unit cell volume of Nbss due to strong covalent bonding]. The lattice parameters of Nbss in homogenized specimens were lager than those of correspond aged specimens due to lower both the (Al+Cr) concentration and Si concentration in Nbss during aging treatments.

3.5: Effects of homogenizing and aging treatments on the lattice parameters of the Nbss: The chemical elements showed strong solute partitions tendency among the Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb, with Hf and Si showing strong preference for the silicide phase, Al and Cr having preference for the Laves phase, and Nb and Ti are partitioned to Nbss. The partitioning tendencies of various elements in heat treated specimens are similar to that of the arc-melted alloy. However, the Ti and Cr concentration in the Nbss changed dramtically after heat treatment. Ti and Cr concentrations in Nbss different condition was showed in **Figure 9**. It can be seen that Ti concentration in Nbss decreased with the increase of homogenization temperature. When the homogenization temperature is 1200~1300℃. The Ti concentration in Nbss is almost changed. When the homogenization temperature is increased 1400℃ or 1500℃, the Ti concentration is reduced significantly. The Ti concentration in Nbss decreased with the homogenization temperature increases in the subsequent homogenization+ aging heat treatment.

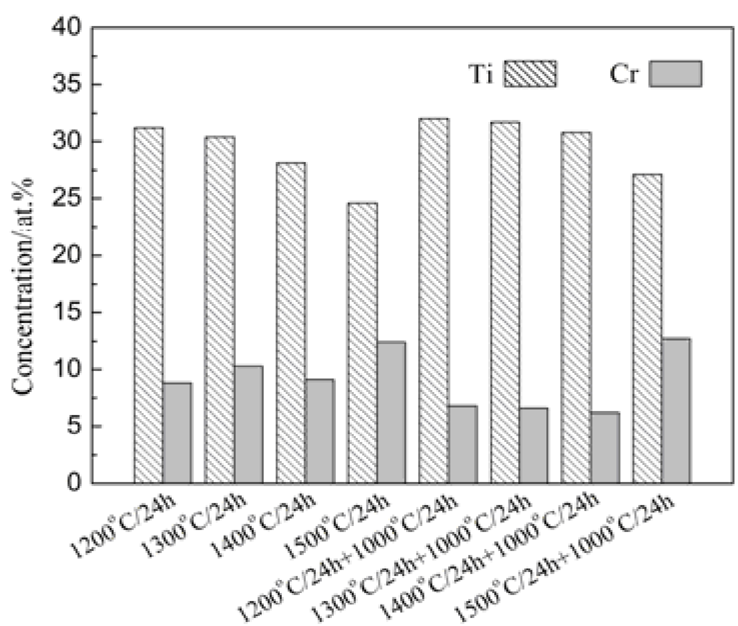


Fig. 9: Ti and Cr concentrations in Nbss with different heat treatment condition

However, the change of Cr concentration in Nbss is more complicated. The Cr concentration in Nbss is increase when the homogenization temperature is 1200~1300. However, the Cr concentration in Nbss decrease when the homogenization is 1400°C. The Cr concentration in Nbss reached max at the homogenization temperature 1500°C. The Cr concentration in Nbss is decreased significantly after homogenization+aging treatment except 1500°C/24h+1000°C/24h.

According to the above analysis, the change of Ti and Cr concentration in Nbss is closely related to the homogenization temperature, and the reason for the change is due to their different elements concentration among the adjacent phases. Ti could diffuses from Nbss to adjacent silicides and Cr_2Nb phase resulting in a decrease in Ti concentration in Nbss. Cr element is mainly distributed in Cr_2Nb phase, and Cr could diffuses from Cr_2Nb to Nbss. Cr_2Nb phase dissolved when the homogenization temperature is 1300 ~ 1400°C, and Cr atoms dissolved in the Nbss. The formation of supersaturated solid solution would precipated from Nbss matrix in the subsequent furnace cooling, which resulted Cr in Nbss decreased. The Cr concentration in Nbss increased abruptly due to the decrease in Ti concentration at the temperature 1500°C. In addition to the 1500°C/24h+1000°C/24h specimen, the Cr concentration in Nbss decreased significantly due to the fine Cr_2Nb precipited in the Nbss matrix.

3.6 Effects of homogenizing and aging treatments on the microhardness of Nbss and $(Nb,X)_5Si_3$: Figure 10 shows the variation of microhardness of both Nbss and $(Nb,X)_5Si_3$ phase in the arc-melted and heat treated conditions. The Laves phase is too small to measure its microhardness. It was found that the microhardness of $(Nb,X)_5Si_3$ changed indistinctively during homogenizing treatment compared with that of the arc-melted alloy. However, the microhardness of $(Nb,X)_5Si_3$ increased clearly during the aging treatment attributed to the reduction of Ti and Al concentration and the increase of Hf in $(Nb,X)_5Si_3$ during the aging treatment according to the partitioning ratios of Ti, Al and Hf.

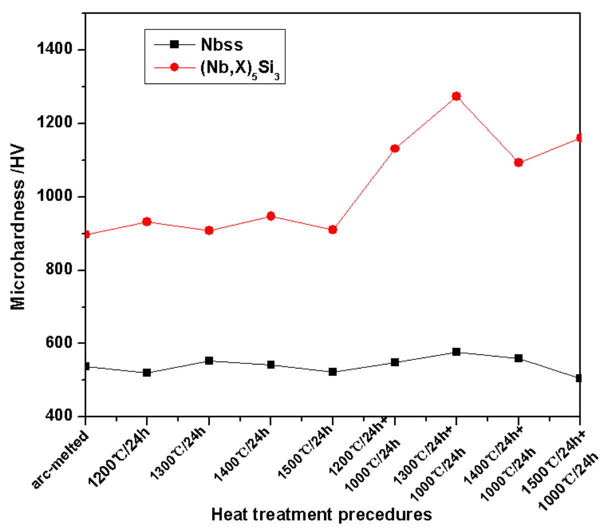


Figure 10: Variation of microhardness of both Nbss and $(Nb,X)_5Si_3$ after homogenizing and aging treatments

As shown in Figure 10, the microhardness of Nbss phase changed less evidently than that of $(Nb,X)_5Si_3$ after heat treatments. The maximum values of the microhardness of Nbss are obtained aging treatment at 1300°C/24h+1000°C/24h. From the microstructures of aging treatment, it can be concluded that the finer precipitates Laves phase which were present in Nbss matrix led to the increase in the microhardness of Nbss. The microhardness of the Nbss increased with increase in homogenized temperature due to the higher Cr concentration in the Nbss matrix. From Figure 10, it can be seen that the microhardness of the Nbss and $(Nb,X)_5Si_3$ reached maximum value at 1300°C/24h+1000°C/24h, so 1300°C/24h+1000°C/24h was an optimum heat treatment process.

4. CONCLUSION

The major conclusions arising from this study on microstructure and microhardness upon the high temperature heat treatment in the multicomponent Nb-Ti-Cr-Si based ultrahigh temperature alloy are as follows:

1. The microstructures of the heat treated were still composed of Nbss, $(Nb,X)_5Si_3$ and Cr_2Nb during the homogenizing and aging treatments. But HfO_2 was present in the microstructure after heat treatment both at 1500°C/24h and 1500°C/24h+1000°C/24h.
2. The Nbss/$(Nb,X)_5Si_3$ eutectic colonies in the arc-melted alloy broke up and formed small $(Nb,X)_5Si_3$ blocks in Nbss matrix with the increase in heat treatment temperatures. The previous Nbss/Cr_2Nb eutectic colonies in the arc-melted alloy broke up and formed coarse Cr_2Nb, but theses coarse Laves phases were dissolved on heat treatment at 1400°C/24h and much finer and crowded Cr_2Nb platelets formed upon cooling. These observations suggest that the coarse Laves phase go into solution at temperatures between 1300 and 1400°C and form on cooling by a solid state reaction in Nbss matrix.
3. Aging of homogenized coupons at 1000°C/24h led to the precipitation of fine needle –like Laves phase with and along the grain boundaries of Nbss matrix.
4. Ti and Cr concentrations decrease with increase in heat-treatment temperatures. Aging at 1000°C for 24h after homogenizing treatments improves the precipitation of fine needle-like Cr_2Nb in Nbss matrix and Cr concentration in Nbss reduces
5. The microhardness of Nbss changed less evidently during heat treatment and the microhardness of $(Nb,X)_5Si_3$ in aging coupons was higher than that of homogenized coupons due to change the partitioning ratios of various elements. The microhardness of Nbss and $(Nb,X)_5Si_3$ reached maximum value at 1300°C/24h+1000°C/24h.

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