Effect of Cr Content on Microstructure and Room Temperature Fracture Toughness of Nb-Si Based Ultrahigh Temperature Alloys: Postprint

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Abstract

Four alloys with nominal compositions of Nb-22Ti-16Si-4Hf-3Al-xCr (x = 0, 3, 5 and 10, at.%) were prepared by vacuum non-consumable arc melting and subjected to homogenization treatment at 1450℃ for 50 h, to investigate the influence of Cr content on the microstructure of Nb-Si based ultrahigh temperature alloys in both as-arc-melted and heat-treated conditions, as well as the room-temperature fracture toughness in the as-arc-melted state. The results indicate that Cr addition did not alter the crystal structure of the silicides (all being γ(Nb, X)5Si3), but increased their volume fraction with increasing Cr content, while the volume fraction of the Nbss/γ(Nb, X)5Si3 eutectic gradually decreased. Moreover, Cr addition promoted the formation of Nbss/(Nb, X)5Si3/Cr2(Nb, X) three-phase eutectic, whose content increased with increasing Cr content in the alloys. Following heat treatment at 1450℃/50 h, the Nbss dendrites and eutectic structures characteristic of the as-arc-melted state disappeared, and the microstructure became significantly more homogeneous. The alloys containing 0, 3% and 5% Cr consisted of Nbss and γ(Nb, X)5Si3 phases, whereas the alloy with 10% Cr exhibited a three-phase equilibrium microstructure comprising Nbss, γ(Nb,X)5Si3 and Cr2(Nb, X). The room-temperature fracture toughness of the as-arc-melted alloys exhibited a decreasing trend with increasing Cr content.

Full Text

Abstract

Four Nb-Si based ultra-high temperature alloys with nominal compositions of Nb-22Ti-16Si-4Hf-3Al-xCr (x = 0, 3, 5, and 10 at.%) were prepared by vacuum non-consumable arc melting and subsequently heat-treated at 1450°C for 50 h. The effects of Cr content on the microstructure and room-temperature fracture toughness of both as-cast and heat-treated alloys were investigated. The results showed that Cr addition did not alter the crystal structure of the silicide γ(Nb, X)$_5$Si$_3$. However, the volume fraction of γ(Nb, X)$_5$Si$_3$ increased while that of the Nbss/γ(Nb, X)$_5$Si$_3$ eutectic decreased with increasing Cr content. Furthermore, Cr addition promoted the formation of a low-melting-point three-phase eutectic consisting of Nbss, (Nb, X)$_5$Si$_3$, and Cr$_2$(Nb, X), whose volume fraction also increased with Cr content. After heat treatment at 1450°C/50 h, the primary Nbss dendrites and eutectic structures observed in the as-cast condition disappeared, and the microstructure became significantly more uniform. The alloys containing 0, 3, and 5 at.% Cr consisted of Nbss and γ(Nb, X)$_5$Si$_3$, whereas the alloy with 10 at.% Cr exhibited a three-phase equilibrium microstructure of Nbss, γ(Nb, X)$_5$Si$_3$, and Cr$_2$(Nb, X). The room-temperature fracture toughness of the as-cast alloys exhibited a decreasing trend with Cr addition.

Keywords: metallic materials, Nb-Si based ultra-high temperature alloy, arc-melting, heat-treatment, room-temperature fracture toughness, Cr addition

Introduction

Over the past three decades, advancements in aerospace technology have demanded turbine blade materials with higher temperature capability, excellent high-temperature creep resistance, and superior oxidation resistance [1]. However, the currently widely used third-generation Ni-based single-crystal superalloys have reached their temperature limit of approximately 1150°C [2], necessitating the development of new high-temperature metallic structural materials. Nb-Si based ultra-high temperature alloys (i.e., Nb-Si in situ composites) have emerged as promising candidates for aero-engine high-temperature turbine blade applications due to their high melting point (1750°C), low density (6.6-7.2 g/cm^3), and favorable combination of high-temperature strength and room-temperature fracture toughness, with potential service temperatures between 1200°C and 1450°C [3, 4].

The microstructure of Nb-Si based ultra-high temperature alloys primarily consists of niobium solid solution (Nbss) and silicides (including (Nb, X)$_5$Si$_3$ and (Nb, X)$_3$Si). When a certain amount of Cr is added to the alloy, a Cr$_2$(Nb, X) Laves phase may also form [1, 3]. The (Nb, X)$_5$Si$_3$ phase exists in three crystal structures: α-(Nb, X)$_5$Si$_3$ (tetragonal D8$_1$, Cr$_5$Si$_3$-type structure), β-(Nb, X)$_5$Si$_3$ (tetragonal D8 , W$_5$Si$_3$-type structure), and γ-(Nb, X)$_5$Si$_3$ (hexagonal D8$_8$, Mn$_5$Si$_3$-type structure). The niobium solid solution acts as a ductile phase, while the silicides serve as strengthening phases. The eutectic in-situ composite formed by these two phases compensates for their respective deficiencies in mechanical properties at both low and high temperatures, achieving a desirable balance [4]. However, the poor high-temperature oxidation resistance of these alloys limits their applications in aerospace and other fields. To improve the comprehensive properties, researchers have extensively investigated multicomponent alloying of Nb-Si based alloys. Currently, the main alloying elements added to Nb-Si systems include Ti, Al, Sn, B, Mo, W, Hf, V, Cr, Fe, Ta, and

C [5-17]. Among these, Ti addition enhances alloy toughness and ductility while improving high-temperature oxidation resistance [5, 6]; Al and Sn additions improve oxidation resistance but adversely affect ductility and toughness [7-9]; B refines grain structure and stabilizes α-(Nb, X)$_5$Si$_3$ [10, 11]; Mo and W strengthen the alloy through solid solution strengthening [12]; Hf promotes γ-(Nb, X)$_5$Si$_3$ formation and improves oxidation resistance [13, 14]; and V facilitates α-(Nb, X)$_5$Si$_3$ formation and increases room-temperature yield strength [15].

Cr addition improves high-temperature oxidation resistance but is detrimental to ductility and toughness. Moreover, Cr promotes the formation of the Cr$_2$(Nb, X) Laves phase while suppressing (Nb, X)$_3$Si formation [9, 16]. To date, research on Cr alloying has primarily focused on simple ternary or quaternary systems, with relatively few studies on multicomponent alloy systems. Chen et al. [17] investigated the effects of Cr content (0, 10, 14, and 20 at.%) on the microstructure and oxidation resistance of multicomponent Nb-Si based ultra-high temperature alloys, but their study did not address equilibrium microstructures or mechanical properties. Therefore, systematic investigation of Cr content effects on the microstructure and properties of multicomponent Nb-Si based ultra-high temperature alloys is crucial for optimizing alloy composition and achieving balanced comprehensive properties. This work focuses on the effects of Cr content on the microstructure of Nb-Ti-Si-Hf-Al alloys in both as-cast and heat-treated (1450°C/50 h) conditions, as well as on the room-temperature fracture toughness of the as-cast alloys.

1. Experimental Methods

Four Nb-Si based ultra-high temperature alloys with nominal compositions of Nb-22Ti-16Si-4Hf-3Al-xCr (x = 0, 3, 5, and 10 at.%) were prepared by vacuum non-consumable arc melting. Each alloy ingot was remelted six times to ensure compositional homogeneity. Samples measuring 8 mm × 8 mm × 8 mm were cut by electrical discharge machining. Some samples were used for as-cast microstructural analysis, while others were subjected to heat treatment. The heat treatment procedure involved placing the samples in an ultra-high temperature vacuum furnace, evacuating to 1×10^{-3} Pa, and heating at approximately 14°C/min. At 1000°C, the vacuum was released and the furnace was backfilled with high-purity argon for protection. The samples were held at 1450°C for 50 h and then furnace-cooled to room temperature.

The sample surfaces were ground with SiC abrasive papers from 100 to 2000 grit, polished, ultrasonically cleaned in ethanol for 10 min, and dried. Room-temperature fracture toughness was measured using single-edge notched three-point bend specimens on an Instron 3382 universal testing machine. The specimen dimensions were 2.5 mm × 5 mm × 30 mm, and no fatigue precracking was performed.

Microstructural characterization of both as-cast and heat-treated samples was conducted using a Panalytical X' Pert PRO X-ray diffractometer (XRD) and a Supra-55 scanning electron microscope (SEM) equipped with an energy-dispersive spectrometer (EDS). The area fractions of constituent phases were analyzed using Image-Pro Plus 6.0 image analysis software. For convenience, the as-cast and heat-treated samples of the four alloys are designated as xCr-AC and xCr-HT (x = 0, 3, 5, and 10), respectively, where AC denotes as-cast and HT denotes heat-treated.

2. Results and Discussion

Figure 1 [Figure 1: see original paper] shows the XRD patterns of the Nb-Si based ultra-high temperature alloys in both as-cast and heat-treated conditions. The as-cast alloys with 0, 3, and 5 at.% Cr (0Cr-AC, 3Cr-AC, and 5Cr-AC) consisted of Nbss and $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ phases, while the 10Cr-AC alloy contained Nbss, $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$, and $\mathrm{Cr}_2(\mathrm{Nb, X})$. Thus, Cr addition did not affect the crystal structure of the silicide, which remained $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$, but higher Cr content promoted $\mathrm{Cr}_2(\mathrm{Nb, X})$ formation. After heat treatment at 1450°C/50 h, the phase constitutions of all four alloys remained unchanged, as shown in Figure 1b.

2.1 As-Cast Microstructure

Figure 2 [Figure 2: see original paper] presents backscattered electron (BSE) images of the as-cast microstructures of alloys with different Cr contents. All four alloys were hypereutectic, and the microstructure changed noticeably with increasing Cr content. Table 1 lists the compositions of the constituent phases. Based on XRD, BSE, and EDS analyses, the 0Cr-AC alloy microstructure consisted primarily of primary $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ (with approximately hexagonal or irregular "H"-shaped morphologies), Nbss dendrites, and Nbss/$\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ eutectic, as shown in Figure 2a. With Cr addition, the microstructure comprised primary $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ (retaining the approximate hexagonal or "H" shapes, though the proportion of these two morphologies varied with Cr content, as discussed below), Nbss dendrites, Nbss/$\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ eutectic, and a certain amount of dark-appearing constituent, as shown in Figures 2b–d. This dark constituent (indicated by boxes in Figures 2b–d) was distributed mainly in the inter-colony regions of the Nbss/$\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ eutectic, and its content increased significantly with Cr content, reaching approximately 2.5%, 4.2%, and 11.4% (area fraction) in the 3Cr-AC, 5Cr-AC, and 10Cr-AC alloys, respectively.

BSE observations revealed that for alloys with lower Cr contents (0, 3, and 5 at.%), the proportion of "H"-shaped primary silicide among all primary silicides decreased with increasing Cr content, becoming nearly absent in the 5Cr-AC alloy. However, when Cr content increased to 10 at.%, this proportion increased again, likely due to the significant promotion of $\mathrm{Cr}_2(\mathrm{Nb, X})$ formation at higher Cr levels.

Quantitative metallographic analysis indicated that the volume fraction of primary silicide increased with Cr content, measuring approximately 12.5%, 17.5%, 18.3%, and 28.7% (area fraction) in the 0Cr-AC, 3Cr-AC, 5Cr-AC, and 10Cr-AC alloys, respectively. Conversely, the Nbss/γ(Nb, X)$_5$Si$_3$ eutectic content gradually decreased. Lower Cr additions (3 and 5 at.%) increased primary silicide size, while further increasing Cr to 10 at.% reduced the size. With increasing Cr content, the Nbss/γ(Nb, X)$_5$Si$_3$ eutectic colonies exhibited a refinement trend, and their morphology changed noticeably. At 5 at.% Cr, the eutectic tended toward a well-coupled petal-like structure, but at 10 at.% Cr, it became highly irregular, as shown in Figure 2. Notably, transverse cracks perpendicular to the long axis were observed within some large hexagonal primary silicides in the 3Cr-AC and 5Cr-AC alloys (Figures 2b and 2c), but not in the 0Cr-AC or 10Cr-AC alloys. Crack formation may be attributed to the mismatch in thermal expansion coefficients between the large primary silicides with hexagonal structures and the surrounding matrix, combined with the limited slip systems in hexagonal crystal systems. Additionally, the large size impairs cooperative deformation with the surrounding matrix, leading to stress concentration and crack initiation.

Figure 3 [Figure 3: see original paper] shows magnified images of the dark constituent in the 3Cr-AC, 5Cr-AC, and 10Cr-AC alloys. This constituent was identified as a low-melting-point three-phase eutectic. EDS analysis revealed high concentrations of Nb, Ti, Si, and Cr, with Cr contents reaching 13.3%, 16.9%, and 15.9%, respectively. Combined with XRD results (Figure 1), this three-phase eutectic was inferred to consist of Nbss, γ(Nb, X)$_5$Si$_3$, and Cr$_2$(Nb, X). The low content of this eutectic in the 3Cr-AC and 5Cr-AC alloys explains the absence of Cr$_2$(Nb, X) diffraction peaks in their XRD patterns. Clearly, Cr addition was the primary cause for Nbss/γ(Nb, X)$_5$Si$_3$/Cr$_2$(Nb, X) eutectic formation, and the rapid solidification rate promoted Cr enrichment in the remaining liquid, facilitating this three-phase eutectic. Guo et al. [18] observed similar three-phase eutectic structures in the mushy zone of directionally solidified Nb-22Ti-16Si-6Cr-3Al-4Hf (at.%) alloys. Additionally, fine white particles were observed in all four alloys and identified as HfO$_2$ by EDS analysis (Figure 2). These particles formed due to the high reactivity of Hf with trace oxygen dissolved in the melt.

According to Table 1, Ti, Cr, and Al primarily dissolved in Nbss, while Hf and Si were mainly present in the silicides. As Cr content increased, its concentration in both Nbss and silicides increased, with the partitioning ratio of Cr between Nbss and silicide also increasing (approximately 8, 9, and 9.4 for the 3Cr-AC, 5Cr-AC, and 10Cr-AC alloys, respectively). The Hf and Si contents in Nbss decreased with increasing Cr content, while noticeable dendritic segregation was observed at Nbss dendrite edges (indicated by arrows "1" in Figures 2a–d), where EDS analysis revealed high Ti and Cr concentrations.

2.2 Microstructure After 1450°C/50 h Heat Treatment

Figure 4 [Figure 4: see original paper] shows BSE images of the four Nb-Si based ultra-high temperature alloys after heat treatment at 1450°C for 50 h, and Table 2 lists the compositions of the constituent phases. The heat treatment eliminated the primary Nbss dendrites and typical eutectic structures present in the as-cast condition, significantly improving microstructural uniformity. The plate- or flake-shaped silicides in the original eutectic spheroidized and grew, distributing more uniformly in the Nbss matrix. The large primary silicides exhibited noticeable dissolution, with their boundaries becoming smoother. Additionally, the number and size of white HfO_2 particles increased after heat treatment, as shown in Figure 4. Comparison of Tables 1 and 2 reveals that Ti and Cr contents in Nbss increased significantly after heat treatment, while Si content decreased. In the silicides, Ti content decreased slightly while Si content increased. This redistribution during heat treatment indicates that Ti and Cr have high solubility in Nbss, whereas Si does not.

Based on XRD, BSE, and EDS analyses, the 0Cr-HT, 3Cr-HT, and 5Cr-HT alloys consisted of Nbss (light gray matrix) and $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ (dark phase), as shown in Figures 4a–c. Notably, $\mathrm{Cr}_2(\mathrm{Nb, X})$ was no longer observed in the 3Cr-HT and 5Cr-HT alloys, indicating that this phase is a high-temperature metastable phase formed by solidification segregation. Its disappearance is likely due to Cr diffusion into Nbss during heat treatment. In addition to Nbss and $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$, a certain amount of dark phase was observed in the 10Cr-HT alloy and identified as $\mathrm{Cr}_2(\mathrm{Nb, X})$ by compositional analysis. This phase existed in two forms: relatively large blocky particles (approximately 10–20 m) and fine granular particles distributed within Nbss or at Nbss/$\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ interfaces, as shown in Figure 4d. Furthermore, Ti- and Hf-enriched zones were observed at the edges of $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ blocks (indicated by arrows "2" in Figures 4a–d), with their area fraction increasing with Cr content.

The microstructure of the 10Cr-HT alloy suggests that the blocky $\mathrm{Cr}_2(\mathrm{Nb, X})$ particles originated from coarsening of the $\mathrm{Cr}_2(\mathrm{Nb, X})$ phase in the three-phase eutectic during heat treatment, while the fine $\mathrm{Cr}_2(\mathrm{Nb, X})$ particles precipitated from Nbss or at Nbss/$\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ interfaces during cooling after heat treatment. EDS analysis showed that the partitioning ratio of Cr between Nbss and $\gamma(\mathrm{Nb, X})_5\mathrm{Si}_3$ increased significantly after heat treatment, reaching approximately 13.3, 13.3, and 11.7 in the 3Cr-HT, 5Cr-HT, and 10Cr-HT alloys, respectively, compared to approximately 8, 9, and 9.4 in the as-cast condition. However, this ratio decreased with increasing Cr content in the heat-treated alloys, opposite to the trend observed in the as-cast condition. Based on these analyses, the Cr content in Nbss of the 10Cr-HT alloy appears to have reached its solubility limit of approximately 11.7 at.% (Table 2).

2.3 Room-Temperature Fracture Toughness

Room-temperature fracture toughness tests were performed on the as-cast alloys with different Cr contents, and the results are summarized in Table 3 . The fracture toughness values (K_Q) decreased with increasing Cr content, measuring approximately 13.4, 11.9, 11.8, and 10.3 MPa $\cdot$ $m^1/^2$ for the 0Cr-AC, 3Cr-AC, 5Cr-AC, and 10Cr-AC alloys, respectively. Thus, Cr addition degrades the room-temperature fracture toughness of Nb-Si based ultra-high temperature alloys. Figure 5 [Figure 5: see original paper] shows SEM images of the fracture surfaces of the four as-cast alloys. The fracture surfaces were relatively flat, featuring numerous bright tear ridges and a few dimples (representing plastic deformation zones of Nbss or eutectic regions), as well as smooth cleavage facets (fracture of primary silicides). Some regions exhibited rock-candy fracture morphologies, with fine, dense steps on certain cleavage planes forming river patterns. Additionally, a small number of secondary cracks were observed on the fracture surfaces. These features indicate that the room-temperature fracture mode was brittle quasi-cleavage fracture.

The degradation in fracture toughness with Cr addition can be attributed to two factors. First, increasing Cr content increased the volume fraction of brittle silicides while decreasing that of the ductile Nbss phase and eutectic regions. Second, higher Cr content increased the amount of Cr_2(Nb, X) phase, which has low room-temperature fracture toughness (approximately 2-5 MPa $\cdot$ $m^1/^2$ [19]). Fracture in these alloys proceeds primarily through cracking of hard (Nb, $X)_5Si_3$ particles and plastic deformation of Nbss and eutectic regions, exhibiting R-curve behavior. Cracks initiate in the hard particles; as the applied load increases, plastic deformation incompatibility between Nbss and the hard particles creates stress concentrations at their interfaces, leading to interfacial debonding and additional crack formation. With further load increase, cracks propagate until final fracture occurs.

3. Conclusions

1. All four Nb-Si based ultra-high temperature alloys were hypereutectic. The microstructure changed noticeably with increasing Cr content. Cr addition did not alter the crystal structure of the silicide, which remained γ(Nb, $X)_5Si_3$, but its volume fraction increased while that of the Nbss/γ(Nb, $X)_5Si_3$ eutectic decreased. Cr addition also promoted formation of the Nbss/(Nb, $X)_5Si_3/Cr_2$(Nb, X) three-phase eutectic, whose content increased with Cr content.
2. After heat treatment at 1450°C/50 h, the primary Nbss dendrites and typical eutectic structures disappeared, and the microstructure became significantly more uniform. The 0Cr-HT, 3Cr-HT, and 5Cr-HT alloys consisted of Nbss and γ(Nb, $X)_5Si_3$, while the 10Cr-HT alloy exhibited a three-phase equilibrium microstructure of Nbss, γ(Nb, $X)_5Si_3$, and Cr_2(Nb, X).
3. The room-temperature fracture toughness of the as-cast alloys decreased

with increasing Cr content.

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