Materials Science and Engineering A 534 (2012) 323–328

Contents lists available at SciVerse ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Microstructure and mechanical properties of Nb–Ti–Si–Al–Hf–xCr–yV multi-element in situ composite

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ARTICLE INFO

Article history:
Received 18 March 2011
Received in revised form 10 October 2011
Accepted 22 November 2011
Available online 3 December 2011

Keywords:
Intermetallics
Microanalysis
Mechanical characterization
Fracture

ABSTRACT

The objective of the paper is to investigate the microstructure, room-temperature fracture toughness and high-temperature compressive deformation behavior of Nb–Ti–Si–Al–Hf–Cr–V multi-component Nb_{ss}/Nb-silicide in situ composite materials with different Cr and V contents. Nb-silicide presented as the primary phase. Cr and V were assisted to stabilize β-Nb_5Si_3 to room temperature. The highest value room-temperature fracture toughness ($K_Q = 14.7\,MPa\,m^{1/2}$) preformed in the test for the materials with lowest Cr and V composition. Crack bridging and crack deflection observed in the crack propagation path were benefit to improve the room temperature fracture toughness. Higher V contents up to 4 at.% was preferred to increase the strength of the studied multi-component system at 1250 °C. A softening of materials phenomenon occurred after the peak stress during compressive tests at 1250 °C.

1. Introduction

It is well known that high temperature structural materials exhibiting excellent mechanical strength and creep resistance, good microstructure stability and oxidation resistance are essential and widely used in jet turbine engine and electric power system [1]. To improve the thrust and reduce emissions of these turbine systems, much higher turbine inlet temperature is demanded, which presently depends on the temperature capacity of turbine blade made of Ni-based superalloy exclusively [1]. However, the operation temperature of Ni-based superalloy, even the newest generation single crystal superalloy, must be less than 1150 °C [2–4] because of the limitation of its melting temperature. In recent years, Nb_{ss}/Nb-silicide in situ composite material is interested to study as one of potential high temperature materials to replace Ni-based superalloy, due to its higher melting point, excellent high temperature strength and low density [5]. In order to balance the mechanical properties and high temperature oxidation resistance of Nb_{ss}/Nb-silicide in situ composites, many research results have been reported in the past several years including the phase diagrams of multi-component systems [6–12], the roles of alloying elements of Ti, Cr, Al, Hf [13–15], Mo, Ta [16], W [17], V [18–20], B [21,22], Ge [23] and the fabrication methods [2,24] [25–30]. The balance of mechanical and environmental properties, however, is still the most challenge for Nb_{ss}/Nb-silicide materials as a type of expected high temperature structure material. Won-Yong Kim reported that the yield strength of Nb_{ss}/Nb-silicide material increased with the V content, however, the strength of the materials will decreased by the increment of V content when the testing temperature is more than 1500 °C [17]. In our previous work [18,19], the role of V on the room temperature toughness and the high temperature oxidation behavior of Nb_{ss}/Nb-silicide material has been investigated, and the results showed that the addition of V in the Nb_{ss}/Nb-silicide material promote the room temperature fracture toughness and high temperature oxidation resistance. However, there is no report about the role of V addition in the multi-component Nb_{ss}/Nb-silicide materials. Nb–Ti–Si–Cr–Al–Hf system was considered to be one of the most potential systems to offer the balance of mechanical properties and oxidation [31]. It has been found that the Cr content significantly affected the fracture toughness at room temperature [32] and the high temperature oxidation resistance [2,3,33,34]. C14 Cr_2Nb Laves phases formed when Cr content is more than 5 at.% will improve the oxidation resistance.

Therefore, the effect of V and Cr contents on the microstructures, room temperature fracture behavior and high temperature compressive deformation behavior of Nb–Ti–Si–Al–Hf–Cr–V multi-component Nb_{ss}/Nb-silicide materials has been studied in the present investigation.

2. Experimental

The raw materials used in this investigation were the commercial Nb, Ti, Si, Cr, Hf with the purity of 99.8 wt.%, 99.7 wt.%, 98.5 wt.%, 99.9 wt.%, 99.9 wt.% respectively and two master alloy

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doi:10.1016/j.msea.2011.11.076

Table 1
The nominal composition of the investigated materials.

Alloy	Composition (at.%)						
	Nb	Ti	Si	Al	Hf	Cr	V
Cr6V4	49	20	16	3	2	6	4
Cr6V2	51	20	16	3	2	6	2
Cr3V4	52	20	16	3	2	3	4
Cr3V2	54	20	16	3	2	3	2

(Al–63.75 wt.%Si and Al–75 wt.%V). The Nb_{ss}/Nb-silicide in situ composite materials with different compositions were prepared by the arc-melting furnace with non-consumable tungsten electrode. The melting processes were preformed in the water-cooled copper button-type crucible with high purity argon atmosphere. In order to ensure the homogenous of chemical composition, all the in situ composite material buttons were re-melted at least four times. The nominal compositions of four designed materials were listed in Table 1.

X-ray diffraction (XRD) analysis was performed on bulk sample for each alloy to characterize phases using D/max X-ray diffractometer with Cu-K_{α} radiation. Microstructures of the four alloys were observed using JSM-5600HV/LV scanning electron microscope (SEM). The average chemical compositions of the alloys and the phase distributed in the alloys were identified by Link ISIS 6489 energy dispersive spectroscopy (EDS). The samples for the microstructure observation were ground with silica abrasive paper and finally polished with 1 μm diamond powder. The volume fractions of Nb_{ss} and Nb-silicide were determined from the area magnified to 350 times by quantitative image analysis using the software of SISC IAS 8.0.

The samples for the room temperature fracture toughness and high temperature compressive strength tests were cut from the central of the as-cast buttons by wire electro-discharge machine (EDM). And the surfaces of the specimens were ground to grit 600. The final beam specimens for fracture toughness tests were 30 mm long, 6 mm wide (W), 3 mm thick (B). The specimens for compressive strength tests were small cylinders with the diameter of 8 mm and the height of 12 mm.

The quasi-plane-strain fracture toughness, K_Q, measurements were measured using the single-edge notched bending (SE(B)) specimens with a support span of 24 mm (S) by MTS machine. An initial notch with the depth (crack length, a) of 3 mm on the specimen was applied by EDM machine according to the description in the ASTM Standard [35]. Values of room temperature toughness were determined by monotonically loading the specimens to failure at a constant displacement rate of 1 mm/min^{-1}. For SE(B) specimens, K_Q is given in terms of the applied maximum load P_{max} by [35]:

$$K_Q = \frac{P_{max}S}{BW^{3/2}} f\left(\frac{a}{w}\right)$$

where f is a non-dimensional function of the crack length a:

$$f\left(\frac{a}{w}\right) = 3\sqrt{\frac{a}{w}} \times \frac{1.99 - (a/W)(1-(a/W))[2.15 - 3.93(a/W) + 2.7(a/W)^2]}{2[1+2(a/W)](1-(a/W))^{3/2}}$$

All values of room-temperature fracture toughness presented in this paper represent an average of two measurements per configuration. After the fracture toughness tests, the cross sections that parallel to the crack advance direction were examined by SEM.

Compressive tests were performed on thermomechanical simulator (Gleeble 3500) at 1250 °C, under displacement control model with the corresponding strain rate of $5 \times 10^{-3}\ s^{-1}$. With the assumption of uniform deformation and perfect shape remaining for the specimens, the load values were converted to true-stress.

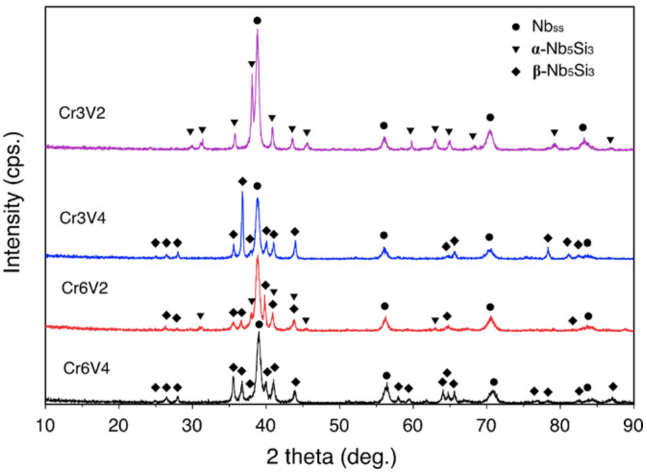

Fig. 1. XRD patterns of Nb–Ti–Si–Al–Hf–Cr–V alloys with different Cr and V composition.

3. Results and discussion

3.1. Phase composition and microstructures

XRD spectra of various investigated alloys are shown in Fig. 1. The XRD analysis suggested that the phase compositions of Cr6V4, Cr3V4 were consisted of Nb_{ss} and β-Nb_5Si_3, and that of Cr3V2 was Nb_{ss} and α-Nb_5Si_3; however, Nb_{ss}, β-Nb_5Si_3 and α-Nb_5Si_3 in small proportion were identified in Cr6V2. C14 Cr_2Nb Laves phase was not found in the four alloys from XRD results. Although the Cr stabilizes Cr-rich Laves phase at bulk composition of greater than ~5 at.% [2,3,34], our previous study [36] showed that the Laves phase did not exist in Nb–Ti–Si–Cr–Al–Hf with 6 at.% Cr content. Thus, in this research, Cr content of no more than 6% is not enough high to form the C14 Cr_2Nb Laves phase. Combined the XRD analysis with the comparison of the nominal compositions in Table 1, it was believed that higher content of V is benefit to slow down the reaction from β-Nb_5Si_3 to α-Nb_5Si_3 or improve the stability of β-Nb_5Si_3 [37] at room temperature. The average chemical compositions of actual alloy and the phases included, which were measured by EDS are shown in Table 2. The results in Table 2 suggested that the type of Nb-silicide may be defined as Nb_5Si_3 other than Nb_3Si, since the content ratios of Nb over Si in Nb-silicide are about 5:3, which is consistent to the results of XRD analysis. Typical back scattered electron images of the four alloys with different V and Cr content are presented in Fig. 2. As indicated by arrows in Fig. 2, the light and dark regions responded to Nb_{ss} and Nb_5Si_3 phases respectively. In Fig. 2a and c, besides the large size β-Nb_5Si_3 phases with the coarse size, the finer Nb_{ss} and β-Nb_5Si_3 were observed. The finer microstructure is similar to the microstructures formed by eutectic reactions and the reaction of $Nb_3Si \rightarrow Nb_{ss} + \alpha\text{-}Nb_5Si_3$, however, α-$Nb_5Si_3$ did not existed in Cr6V4 and Cr3V4 alloys. That might indicate that the finer microstructures in Cr6V4 and Cr3V4 alloys are from the eutectic reaction during the solidification. For Cr6V2 alloy in Fig. 2b, it is hardly to separate the silicide into α-Nb_5Si_3 and β-Nb_5Si_3. However, the ratio of α-Nb_5Si_3 is low which showed by XRD result. Therefore, it might to be believed that most of the finer microstructure in Cr6V2 is still from the eutectic reaction during solidification. In Cr3V2 alloy (Fig. 2d), large size Nb_{ss} and α-Nb_5Si_3 was surrounded by fine Nb_{ss} + α-Nb_5Si_3. However, the size of the fine Nb_{ss} + α-Nb_5Si_3 is larger than the fine Nb_{ss} + β-Nb_5Si_3. In the four pictures, the Nb-silicide phase presented strip or rectangle