Intermetallics 92 (2018) 1–6

Contents lists available at ScienceDirect

Intermetallics

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

Microstructure and fracture toughness of Nb-Si based alloys with Ta and W additions

Yueling Guo, Lina Jia*, Bin Kong, Huarui Zhang, Hu Zhang**

School of Materials Science and Engineering, Beihang University, Beijing, 100191, People's Republic of China

ARTICLE INFO

Keywords:
Nb-Si alloy
Silicide
Directional solidification
Fracture toughness
Crack propagation

ABSTRACT

The alloying effects of Ta and W on the phase composition, microstructure and room-temperature fracture toughness of Nb-Si based alloys were investigated. The nominal compositions of Nb-Si based alloys were Nb-15Si-24Ti-4Cr-2Al-2Hf (base alloy, at.%), Nb-15Si-24Ti-4Cr-2Al-2Hf-1Ta (1Ta alloy, at.%) and Nb-15Si-24Ti-4Cr-2Al-2Hf-1W (1W alloy, at.%). Results showed that the base alloy, 1Ta alloy and 1W alloy were composed of Nb solid solution (Nbss), αNb_5Si_3 and small volumes of hexagonal γNb_5Si_3 phases. The elements of Ta and W were primarily partitioned in the Nbss phases. The addition of Ta improved the fracture toughness of the base alloy from 10.2 to 12.2 MPa $m^{1/2}$, but the addition of W reduced the fracture toughness to 8.2 MPa $m^{1/2}$. The fracture of Nbss phases in the three alloys exhibited a cleavage fracture mode, and river patterns were created on individual facets, while the Nb_5Si_3 phases showed a brittle fracture mode with flat and featureless fracture surfaces. Different with base alloy and 1W alloy, 1Ta alloy involved the occurrence of secondary cracking as well as interface decohesion. The toughening mechanisms of Nb-Si based alloys with Ta and W additions were emphasized.

1. Introduction

As the alternative materials of current Ni-based superalloys for turbine blade applications, Nb-Si based alloys have captured considerable attention, owing to their relatively low densities and outstanding high temperature capabilities [1–4]. Nb-Si based alloys are featured by hard intermetallic Nb_5Si_3 silicides embedded in Nb solid solution (Nbss) matrix or vice versa, depending on their compositions [5–7]. It is known that for turbine blade applications, minimum toughness is required to survive the final assembly into engines and resist damage during service. Though striking achievements have been scored recently, the insufficient fracture toughness of Nb-Si based alloys still limits their industrial application.

Alloying has been considered as an effective method to improve the performance of Nb-Si based alloys. The Nb-Si-Ti-Cr-Al-Hf multi-component system has been recently developed to improve the overall performance of Nb-Si based alloys [8–11]. Ti is added to the Nb-Si based system to improve the intrinsic ductility of the Nbss phase as well as the toughness of Nb-Si based alloys [2,12]. Certain amounts of Cr and Al are alloyed to enhance their oxidation resistance [2,7]. Hf is added as it has a strong solid solution strengthening effect on the Nbss phase [13]. But a further increase in the fracture toughness of Nb-Si based alloys tends to be compromised by the degradation in the high temperature strength or the oxidation resistance [2,14].

The fracture behavior of Nb-Si based alloys at room temperature typically combines the initialization of cracks and the crack-trapping, bowing and bridging processes [15–17]. As in situ composites, the fracture behavior of Nb-Si based alloys may also be dominated by the shear strength and the normal strength of phase interfaces [18]. It has been found that the elements of Ti [12], Hf [19], B [20] and Ga [21] typically benefit the fracture toughness of Nb-Si based alloys, while the elements of Cr [22], Al [2,23], Mo [24], Re [23] and Fe [11] degrade the fracture toughness. Zhang et al. [20] reported a 2 at.% B addition increased the fracture toughness of Nb-22Ti-16Si-5Cr-4Hf-3Al alloy, by refining the eutectic structures and increasing the Ti concentration in Nbss, but higher content of B addition up to 5 at.% or 10 at.% reduced the fracture toughness by increasing the volume fraction of brittle silicides. Zhang et al. [11] found that the Fe addition from 1 at.% to 6 at.% resulted in the phase transition of Nb-Si based alloys from Nbss + $(\alpha+\gamma)Nb_5Si_3$ to Nbss + Nb_4FeSi and the increase in the volume fraction of silicides, leading to the decrease in fracture toughness.

To the best knowledge of the authors, the effect of Ta and W on the fracture toughness of Nb-Si based alloy has not been investigated systematically, which is the aim of the current work. Combined with our

* Corresponding author.
** Corresponding author.
E-mail addresses: jialina@buaa.edu.cn (L. Jia), zhanghu@buaa.edu.cn (H. Zhang).

http://dx.doi.org/10.1016/j.intermet.2017.09.005
Received 10 May 2017; Received in revised form 9 August 2017; Accepted 7 September 2017

Table 1
Nominal compositions of base alloy, 1Ta alloy and 1W alloy (at.%).

Sample	Composition (at.%)
Base alloy	Nb-15Si-24Ti-4Cr-2Al-2Hf
1Ta alloy	Nb-15Si-24Ti-4Cr-2Al-2Hf-1Ta
1W alloy	Nb-15Si-24Ti-4Cr-2Al-2Hf-1W

previous results on the weight gain after oxidation at 1250 °C for 100 h [25], the overall effects of Ta and W on the room/high temperature performance of Nb-Si based alloys processed by directional solidification (DS) were discussed.

2. Materials and methods

Three button ingots with different compositions were prepared by non-consumable arc-melting in an argon atmosphere, respectively. Each button was 1.5 kg in weight. The nominal compositions of base alloy, 1Ta alloy and 1W alloy were listed in Table 1. These buttons were melted five times to ensure chemical homogeneity. Master alloy rods for DS experiment with a diameter of about 13 mm were then prepared by electro-discharge machining (EDM) and assembled in Y_2O_3 crucibles. DS experiments were performed in a liquid-metal-cooled (LMC) furnace, and the melt super-heat temperature was set to be 1980 °C. The furnace chamber started to be heated once the vacuum pressure reduced to 5.0×10^{-3} Pa. High purity Ar (99.99 wt%) was backfilled into the furnace chamber, when the temperature was over 1000 °C. The DS experiments were all carried out at a withdrawal rate of 30 mm/min. The three alloy bars via DS were then heat treated at 1450 °C for 12 h in a high vacuum furnace, followed by furnace-cooling after heat treatment (HT).

According to ASTM E399 [26], fracture toughness measurements (K_Q) were performed on an electromechanical universal testing machine (SANS 5105) using single-edge notched three-point bending specimens (Fig. 1). The calculation of K_Q is presented in Eqs. (1) and (2) in detail [26]:

$$K_Q = \frac{P_Q S}{Bw^{3/2}} \cdot j\left(\frac{a}{w}\right) \tag{1}$$

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

Where P_Q was the maximum force during bend specimen testing, B is the specimen thickness, S is span length, w was the specimen width and a was the notch length. The size of each notch was up to $a/w = 0.5$. The notch, prepared by EDM using a Mo wire with a diameter of 0.2 mm, was normal to the DS direction. Three parallel samples were tested, and the average value as well as the corresponding standard deviation (SD) was obtained.

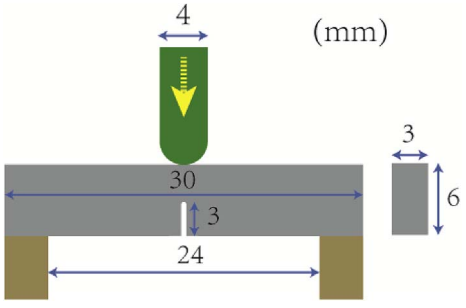

Fig. 1. Schematic drawing of the single-edge notched three-point bending specimen for fracture toughness measurement.

X-ray diffraction (XRD, D/max-2500) with Cu Kα radiation was carried out for phase identification at a 2θ scanning rate of 6°/min. Microstructures of the three Nb-Si based alloys were examined using an electron probe micro-analyzer (EPMA, JXA-8230) equipped with wave-dispersive spectroscopy (WDS). Prior to examination, the samples were gradually grounded using silicon carbide papers up to 1200# and finally mechanically polished. The volume fraction of the Ti-rich Nb_5Si_3 phase was measured by the Image-Pro Plus 6.0 software. At least three images of the longitudinal microstructures were used for the measurement for one alloy, and the average value as well as the corresponding SD was calculated. Secondary electron images of the fracture surfaces were taken by field-emission scanning electron microscopy (SEM, Quanta 200 F), and the corresponding three-dimensional topographies were obtained using a laser confocal scanning microscopy (LCSM, Olympus LEXT OLS4000). The Ti-rich Nb_5Si_3 phases were further investigated using a transmission electron microscopy (TEM, JEOL JEM-2100). The foils for TEM were prepared by a typical cutting, prethinning and final ion thinning process. Ion milling involved bombarding the TEM foils with energetic ions using a Gatan 691 equipment.

3. Results

Fig. 2 shows microstructures of the base alloy, 1Ta alloy and 1W alloy via DS. Combining with XRD results in our previous work [25], the three Nb-Si based alloys are mainly composed of Nbss (light grey in contrast) and αNb_5Si_3 (dark grey in contrast). The base alloy, 1Ta alloy and 1W alloy via DS all exhibit a hypoeutectic microstructure, as the primary large Nbss phases are observed. It can be deduced that during the solidification of the three Nb-Si based alloys, the primary Nbss phases firstly crystallize from the melt, expressed as $L \rightarrow Nbss + L_1$. Then the residual liquid melt reached the first eutectic composition, and the eutectic reaction $L_1 \rightarrow Nbss + \alpha Nb_5Si_3$ is triggered. No significant difference on the microstructure is shown for the three Nb-Si based alloys via DS, suggesting the 1 at.% addition of Ta or W does not significantly alter the microstructure and phase constitution of Nb-Si based alloys. In addition, small volumes of Ti-rich Nb_5Si_3 phases in black contrast are distinguishable in the three alloys. They are frequently formed at the phase boundaries between Nbss and αNb_5Si_3.

Based on the XRD patterns shown in Fig. 3, the main phases of the base alloy, 1Ta alloy and 1W alloy after HT at 1450 °C for 12 h remain to be Nbss and αNb_5Si_3. Typical microstructures of the three alloys are displayed in Fig. 4. It is observed that in the three alloys, the interconnected Nbss phases are the continuous matrix of Nb-Si based alloys embedded with discontinuous silicides. On their longitudinal microstructures, the fibrous silicides are presented paralleling to the DS direction, and the silicides tend to be spheroidized to reduce the interphase boundary area and the total interfacial energy [27]. Considering no obvious chemical segregation is observed in the three alloys after HT, the chemical compositions of Nbss, αNb_5Si_3 and Ti-rich Nb_5Si_3 have been measured by EPMA-WDS, as listed in Table 2. It is observed the addition of Ta or W alters the compositions of the Nbss phases, especially the content of Si in Nbss. The addition of 1 at.% Ta results in an 8% increase in the content of Si in Nbss, while the addition of 1 at.% W leads to an 11% decrease, which is in accordance with other investigations [17,28]. As determined from the Nb-Si and Ta-Si binary phase diagrams [29], the solid solubility of Si in Ta is higher than that in Nb at the same temperature, i.e., the addition of Ta enhances the solid solubility of Si in the Nbss phases. Though the solid solubility of Si in W is also larger than that in Nb according to the W-Si binary phase diagram [29], the addition of W typically reduces the solid solubility of Si in the Nbss phases due to the slow diffusion rate of W in Nb at high temperature [17]. The content of Ti in Ti-rich Nb_5Si_3 is approximately twice as high as that in αNb_5Si_3. The element of Ta is largely partitioned in Nbss phases (1.20 at.%), and the contents of Ta in αNb_5Si_3 and Ti-rich Nb_5Si_3 are merely 0.72 at.% and 0.15 at.%, respectively.