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Effect of Fe additions on microstructure and mechanical properties of a multi-component Nb–16Si–22Ti–2Hf–2Al–2Cr alloy at room and high temperatures

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ABSTRACT

The phase constitutions, microstructural evolutions, and mechanical properties of Nb–16Si–22Ti–2Hf–2Al–2Cr–*x*Fe alloys (where $x = 1, 2, 4, 6$ at.%, hereafter referred to as 1Fe, 2Fe, 4Fe and 6Fe alloys, respectively) prepared by arc-melting were investigated. It was observed that the nominal Fe content affected the solidification path of the multi-component alloy. The as-cast 1Fe alloy primarily consisted of a dendritic-like Nb_{SS} phase and $(\alpha+\gamma)$-Nb_5Si_3 silicide, and the as-cast 2Fe and 4Fe alloys primarily consisted of an Nb_{SS} phase, $(\alpha+\gamma)$-Nb_5Si_3 silicide and (Fe + Ti)-rich region. In addition to the Nb_{SS} phase, a multi-component Nb_4FeSi silicide was present in the as-cast 6Fe alloy. When heat-treated at 1350 °C for 100 h, the 1Fe and 6Fe alloys almost exhibited the same microstructures as the corresponding as-cast samples; for the 2Fe and 4Fe alloys, the (Fe + Ti)-rich region decomposed, and Nb_4FeSi silicide formed. The fracture toughness of the as-cast and heat-treated Nb–16Si–22Ti–2Hf–2Al–2Cr–*x*Fe samples monolithically decreased with the nominal Fe contents. It is interesting that at room temperature, the strength of the heat-treated samples was improved by the Fe additions, whereas at 1250 °C and above, the strength decreased, suggesting the weakening role of the Nb_4FeSi silicide on the high-temperature strength. As the nominal Fe content increased from 1 at.% to 6 at.%, for example, the 0.2% yield strength increased from 1675 MPa to 1820 MPa at room temperature; also, the strength decreased from 183 MPa to 78 MPa at 1350 °C.

1. Introduction

Nb is one of the most attractive base metals to replace Ni-based superalloys for future high-temperature structural materials with an application temperature range in excess of 1200 °C. A balanced combination of properties, including ductility and toughness at low temperatures, strength and creep resistance at elevated temperatures, as well as environmental stability, is the basic requirement for the Nb-based alloys to be applied in advanced aeronautics and astronautics propulsion systems. To enable Nb-based alloys to achieve such balanced properties, the concept of multi-phase alloys [1–10] and the directional solidification [6,8] and hot extrusion technologies [11–14], have been used to design the microstructure and improve the mechanical properties of these alloys. For multi-phase design, the incorporation of the ductile Nb solid solution Nb_{SS} with the stiffening silicide Nb_5Si_3 [1–10] and the oxidation-resistant Laves phase Cr_2Nb [15–17] has been conducted to form an Nb–Si–Cr ternary-based system with a multi-phase $Nb_{SS}/Nb_5Si_3/Cr_2Nb$ microstructure. By optimising the volume fraction and morphology of the Nb_{SS}, Nb_5Si_3 and Cr_2Nb phases in the Nb–Si–Cr based alloys, balanced properties may be obtained that meet the requirements of ultra-high-temperature structural materials.

In addition to the aforementioned microstructural design and processing technologies, alloying is always an effective way to optimise each property. W and Mo are observed to be the strongest solid-solution strengthening elements for Nb [7,8,18], while Ti and Hf have been demonstrated to play a key role in improving the fracture toughness and ductility of the Nb solid solution because these elements decrease the ductile–brittle transition temperature (DBTT) of Nb [19,20]. Al and Fe were observed to reduce oxygen diffusion and solubility in Nb, which is beneficial to the oxidation resistance of Nb [15–17].

Recently, alloying Nb–Si-based alloys with Fe has attracted interest [13,14,21,22] because Fe has a similar effectiveness as Cr at concentrations up to 5 at.% in improving oxidation resistance [15]. Studies have demonstrated that a 5 at.% Fe addition results in

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complex phase constitutions in hypereutectic Nb–18Si–24Ti–5Cr and Nb–18Si–24Ti–5Cr–5Sn alloys, in which a new silicide, Nb_4FeSi, has been observed [21,22]. For as-cast Nb–Si–Fe ternaries, the Nb_4FeSi arises at a Fe content of 2 at.% [23]. A tensile elongation of 2% at room temperature and superplasticity of approximately 512% elongation at 1450 °C were obtained in a Nb–16Si–2Fe alloy prepared by hot pressing sintering [13] due to the formation of a $Nb_4Fe_3Si_5$ phase with a melting-point as low as 1359 °C. To our knowledge, the understanding of how Fe affects the phase and microstructural evolution and mechanical properties at room and high temperatures for Nb–Si based alloys remains poor. The purpose of this research is to determine the basic changes in metallurgy and mechanical behaviour of a multi-component Nb–16Si–22Ti–2Hf–2Al–2Cr with Fe additions under as-cast and heat-treated conditions.

2. Experimental procedures

Button ingots of Nb–16Si–22Ti–2Hf–2Al–2Cr–*x*Fe alloys ($x = 1, 2, 4, 6$ at.%), each with a weight of 150 g, were prepared by melting five or six times using the vacuum arc melting (VAR) method to homogenise the compositions of the ingots. The ingots were then annealed at 1350 °C in vacuum for 100 h, followed by furnace-cooling to ensure quasi-equilibrium microstructures. The purity of all the raw elements used was 99.95% (in mass) or higher. X-ray diffraction (XRD) of the bulk samples was measured to identify the phase constitutions of the as-cast and heat-treated samples using Cu Kα ($\lambda = 0.15405$ nm) radiation at 40 kV and 40 mA (Rigaku D/Max 2500 PC), while Rietveld-type analysis was used to assign the peaks. Back-scattered electron (BSE) images were acquired to investigate the microstructures of the samples and crack paths of the bending tested samples using an SEM (JEOL JXA-8100) equipped with energy-dispersion X-ray spectroscopy (EDS, Oxford Instruments, UK) to analyse the phase compositions, while secondary electron images (SEI) of the fracture surfaces were obtained using an FEI Quanta 200F scanning electron microscope. The phase volume fraction was statistically determined by quantitative metallographic analysis of microstructure images taken in the BSE mode through a software.

Vickers hardness (*Hv*) was measured under an applied load of 0.98 N on a digital HXZ-1000 micro-sclerometer with a loading-maintenance time of 15 s, and an average value was obtained by collecting 10 *Hv* readings for each sample. The *Hv* value was calculated by the formula of $Hv = 0.102 \times (2F \times \sin(\alpha/2))/d^2$, where F is the loading (N), α is the top angle of the diamond indenter (136°), and d is the diagonal length of the indentation. Single-notched three-point-bending plates were employed for the fracture toughness (K_Q) measurements of the bulk alloys. A notch of up to $a/w = 0.5$ (a: notch length, w: specimen width) was introduced at the middle of the longest side of the specimens by electrical discharge machining with a 0.2-mm-diameter Cu wire. The dimensions of the three-point-bending plates were 30 mm × 6 mm × 3 mm, and the bending tests were conducted on a SANS testing machine with a loading span of 24 mm and a cross-head displacement rate of 0.1 mm/min. The compressive tests at room temperature were conducted on a SANS testing machine at a strain rate of $3 \times 10^{-4}\ s^{-1}$ to measure the compressive strength; the size of the rectangular compressive samples was 3 mm × 3 mm × 6 mm. The high-temperature compressive tests at 1250 °C and 1350 °C were conducted in an argon atmosphere at a strain rate of $3 \times 10^{-4}\ s^{-1}$ using a Gleeble 1500 testing machine. The compressive cylinders were 6 mm in diameter and 9 mm in length. All the specimens for the three-point-bending and compressive tests were mechanically polished using SiC paper (1200-grit) with water before testing.

3. Results

3.1. Phase constitutions and microstructural evolution with Fe

Typical X-ray diffraction patterns of the as-cast and heat-treated Nb–16Si–22Ti–2Hf–2Al–2Cr–*x*Fe alloys are presented in Fig. 1. In the as-cast samples, as observed in Fig. 1(a), the characteristic diffraction peaks of the Nb solid solution Nb_{SS}, α-Nb_5Si_3 and γ-Nb_5Si_3 silicides (referred to as the (α+γ)-Nb_5Si_3 phase hereafter) are detected in the 1-4Fe alloys, in which the peak intensities of the γ-Nb_5Si_3 silicide are much lower than those of the α-Nb_5Si_3 silicide, suggesting that the α-Nb_5Si_3 is the dominant silicide. For the 6Fe alloy, the characteristic peaks of the Nb_5Si_3 silicide cannot be detected; instead, the characteristic peaks of a new silicide, Nb_4FeSi, with a b.c.t structure appear in addition to the Nb_{SS} phase. These findings reveal that the Fe additions may promote the generation of the Nb_4FeSi silicide while suppressing the formation of the Nb_5Si_3 phase. After heat treatment at 1350 °C for 100 h, as shown in Fig. 1(b), the 1Fe alloy still contains the characteristic peaks of the Nb_{SS} and (α+γ)-Nb_5Si_3 phases, while the 6Fe alloy exhibits characteristic peaks of only the Nb_{SS} and Nb_4FeSi phases. Heat treatment does not appear to change the phase constitutions

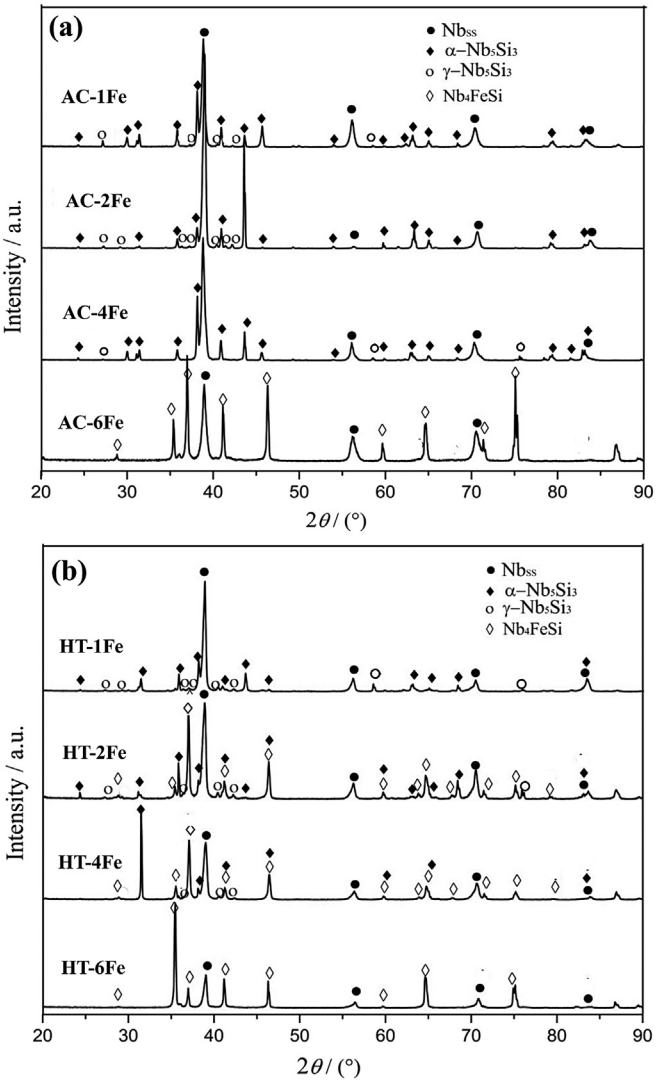

Fig. 1. X-ray patterns of the as-cast (a) and heat-treated (b) 1–6Fe samples.