Intermetallics 90 (2017) 135–139

Contents lists available at ScienceDirect

Intermetallics

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

Mechanical properties and fracture behavior of an Nb-Silicide in situ composite

J.L. Yu[a,*], Xiao Dong Weng[b], N.L. Zhu[c], H. Liu[a], F. Wang[a], Y.C. Li[a], X.M. Cai[a], Z.W. Hu[a]

[a] Refractory Materials Research Center, Northwest Institute for Non-Ferrous Metal Research, Xi'an 710016, China
[b] PLA Rocket Force Representative Office of CAEP, Mianyang 621900, China
[c] Institute of Science and Technology, Hangzhou Dianzi University, Hangzhou 310018, China

ARTICLE INFO

Keywords:
Intermetallics
Tensile properties
Interface strength
Fracture behavior
Plasticity

ABSTRACT

An Nb-Silicide in situ composite with a nominal composition of Nb-16Si-10Ti-10Mo-5Hf (at. %) was fabricated by mechanical alloying followed by hot-pressing sintering. The microstructure consisted of an Nb solid solution, Nb_5Si_3 and a small amount of Nb_3Si. This in-situ composite exhibited good balance of strength between ambient temperature and high temperatures; the ultimate tensile strength was 413 and 496 MPa at room temperature and 1200 °C, respectively. The tensile fracture behavior was dominated by cleavage of the Nbss and Nb_5Si_3 at 1200 °C and lower temperatures. However, the fracture behavior was governed by ductile rupture of Nbss at 1300 °C and higher temperature, which was ascribed to both the increased ductility of Nbss and the decreased interface strength. At 1400 °C and higher temperature, the material exhibited extensive plasticity or superplasticity; the dominant deformation mechanism was grain boundary sliding at 1400 °C and higher temperature.

1. Introduction

The Nb_5Si_3 is currently considered as potential replacements of Ni based superalloys in certain applications in gas turbine engines owing to its low densities, high melting point and high temperature strength [1]. However, the intrinsic brittleness of Nb_5Si_3 at ambient temperature inhibits its practical applications. To overcome this critical issue, ductile Nbss (Nb solid solution) in equilibrium with Nb_5Si_3 up to very high temperatures has been incorporated into an Nb_5Si_3 matrix in an in situ manner to produce composites based on the concept of ductile-phase toughening of brittle compounds [2,3]. The typical examples were reported by Mendiratta et al. [4] and Yu et al. [5] using hot-extruded in situ composites consisting of Nbss and Nb_5Si_3. They show much higher room temperature fracture toughness exceeding $20MPam^{1/2}$, as compared with monolithic Nb_5Si_3. Accordingly, Nb-Silicide in situ composites rather than monolithic Nb_5Si_3 are promising for such applications.

However, the binary alloys exhibited poor oxidation resistance, and, in general, any alloying addition to improve the high temperature oxidation leaded to deterioration of the high temperature mechanical properties of these materials [6]. Thus, a multicomponent approach was necessary to counterbalance the negative effects of one alloying by the other [7]. The results of previous studies [8] indicated that partial substitution of Nb by Ti leaded to appreciable improvement in the oxidation resistance of these composites. The Ti also improved fracture toughness, but on the other hand reduced the melting point and the high temperature strength. Therefore, to counteract the negative effect of Ti, element such W, Mo, Hf and Ta et al. were added, as these provided strong solid solution strengthening of the Nb and silicide phase [9,10]. The Nb-Silicide in situ composites strengthened by Mo and W solution elements exhibited extremely high compressive strength at temperature ranging from room temperature to 1500 °C [11,12]. Furthermore, the balance between high temperature compressive strength [13,14] and room temperature fracture toughness [15,16] of Nb-Silicide in situ composites have been also achieved.

However, only a few reports are currently available on the tensile properties and fracture behavior of Nb-Silicide in situ composites at elevated temperature. Therefore, understanding of the tensile properties and fracture behavior are essential. Jin-Hak Kim et al. [17] reported that the tensile properties of the Nb-18Si-5Mo-5Hf-2C (at. %) in composite fabricated by arc melting method. Excellent tensile strength of 460 MPa was obtained at 1470 K. However, this composite did not exhibit good plasticity even at elevated temperature; the tensile elongation reached only about 0.8% at 1470 K. Here we introduced a new metallurgical manufacturing route, i.e. mechanical alloying (MA) followed by hot-pressing sintering which fabricated a fine-grained Nb-Silicide in situ composite. Mechanical alloying (MA) have been used to synthesize supersaturated solid solution, nano-crystalline, metastable compounds and amorphous solids [18,19]. Due to the reduced diffusion

* Corresponding author. Tel./fax: +86 029 86231082.
E-mail address: yujilang@sina.com (J.L. Yu).

http://dx.doi.org/10.1016/j.intermet.2017.07.014
Received 8 March 2017; Received in revised form 13 July 2017; Accepted 20 July 2017
Available online 24 July 2017

path and the increased free energy, it was easy to produce materials with ultra-fine and homogeneously distributed phase. Therefore, the MA process was introduced as a new process to develop Nb-16Si-10Ti-10Mo-5Hf in situ composites with ultra-fine and homogeneously distributed phase. The main objective of this study was to investigate the microstructure, tensile property, fracture behavior, and mechanism of plastic deformation for the Nb-silicide in situ composite, which was fabricated by MA followed by hot-pressing sintering.

2. Experiments

The raw materials used in the present study were Nb (+200 mesh), Si (+325 mesh), Ti (+200 mesh), Hf (- 200 mesh) and Mo (+130 mesh) powders with purity of 99.9%, 99.9%, 99.9%, 99.9% and 99.9% (wt. %), respectively. The Nb-silicide in situ composite was prepared by a powder metallurgy (PM) technique using elemental powders. The powders with the nominal composition of Nb-16Si-10Ti-10Mo-5Hf (at. %) were ball milled with ball-to-powder volume ratio of 6:1 in argon atmosphere for 50 h until a powder with a nanostructured microstructure was obtained. In order to avoid cold welding, 1 wt % stearic acid was added as a surfactant. The powders after MA were sintered at 1500 °C and 40 MPa for 1 h by hot-pressing sintering in the argon atmosphere.

To identify the constituent phases, X-ray diffraction (XRD) measurements using a Cu-Kα line were carried out, scanning from 20° to 150°. The specimens for microstructure observations were machined, mechanically polished to a mirror surface using water proofed SiC papers, and then used diamond particles with distilled water on platens. Scanning electron microscopy (SEM) studies using backscattered electron image were conducted to reveal the distribution and size of constitution phases.

The tensile specimens with 3 mm × 1 mm cross-section and 10.5 mm gauge length were cut by an electro-discharge machining (EDM) from the as-sintered materials. Then, the surfaces of the specimens were polished so that microstructure investigations of tested specimens were enabled. The tensile tests were carried out in an Instron-type material-testing machine. The test at ambient temperature was carried out with an initial strain rates $2.31 \times 10^{-4}s^{-1}$ in air. The tensile tests were performed at temperature ranging from 1200 °C to 1500 °C under a constant crosshead speed with an initial strain rate $2.31 \times 10^{-4}s^{-1}$ in vacuum (of less than 5×10^{-2}MPa). Finally, SEM fractography was carried out to characterize the fracture modes.

3. Results

3.1. Material characterization

A typical backscattered electron image of Nb-16Si-10Ti-10Mo-5Hf in situ composite was presented in Fig. 1. The microstructure consisted of an Nb solid solution, β-Nb_5Si_3 and a small amount of Nb_3Si, and appeared as a (nearly) continuous Nbss matrix with uniformly dispersed intermetallic phases. In Fig. 1, the bright phase was Nbss; the gray phase was Nb_3Si and the dark phase was the Nb_5Si_3 phases. This composite had a fine microstructure, and the grain shape was nearly equiaxed. The EDX analyses revealed the chemical composition of the constitute phases, which were summarized in Table 1.

Fig. 2 showed a XRD profile of the as-sintered composite. The Nb_5Si_3 was found to be a beta phase. The lattice parameters, a and c, were 0.6581 and 1.1874 nm, respectively, which were smaller than those of binary Nb_5Si_3 (a = 0.65685 nm and c = 1.18815 nm) [20], and the volumetric shrinking due to the Ti and Mo alloying. The lattice parameter of Nbss 0.3284 nm is about 0.6% smaller than that of pure Nb (0.3304 nm) [20].

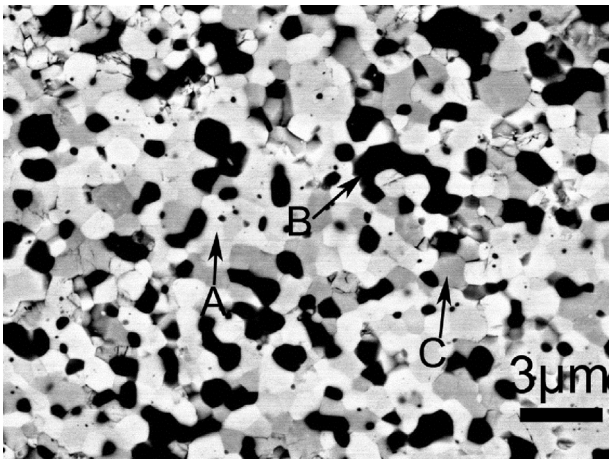

Fig. 1. Typical microstructure of the Nb-16Si-10Ti-10Mo-5Hf in situ composite was prepared by hot pressing sintering. Grains A, B and C represent Nbss, Nb_5Si_3 and Nb_3Si, respectively.

Table 1
Chemical composition of Nb-Silicide in situ composite and constituent phases.

	Composition (mol %)				
	Nb	Si	Ti	Mo	Hf
Composite	59	16	10	10	5
Nbss	66.9	0.7	12.3	17.5	2.6
Nb_3Si	67.1	23.6	5.1	1.6	2.6
Nb_5Si_3	46.5	35.1	7.6	3.1	7.7

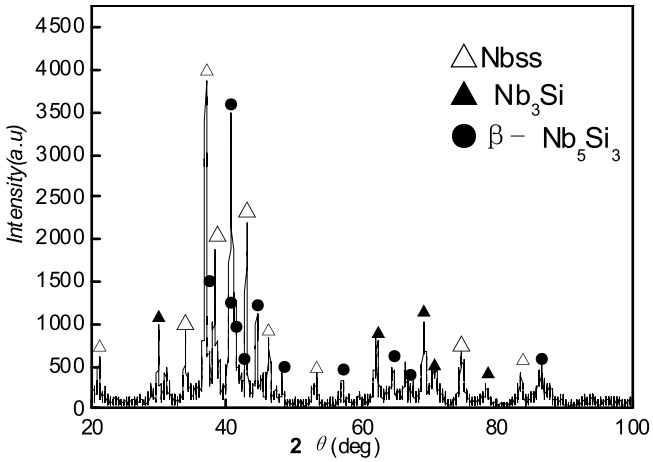

Fig. 2. XRD pattern of the Nb-16Si-10Ti-10Mo-5Hf composite.

3.2. Tensile deformation behavior

The typical tensile stress-strain curves of the Nb-16Si-10Ti-10Mo-5Hf composite at ambient and elevated temperature were presented in Fig. 3. At 1300 °C and higher temperatures, the flow curves showed a peak followed by a gradual decrease.

The tensile properties at ambient and elevated temperature (1200, 1300, 1400, 1500 °C) were shown in Table 2. Three tensile test specimens were determined at room temperature and high temperatures. The measured elongation and tensile strength values were average value. At room temperature, the composite exhibited no elongation and ultimate tensile strength (UTS) was lower than that at 1200 °C, in spite of the large volume fraction of Nbss. As for Nbss with composition of Nb-0.7Si-3Al, since the yield strength of about 450 MPa and a true strain over 0.15 had been reported at room temperature and an initial strain rate of $1.7 \times 10^{-4}s^{-1}$, embrittlement of the composite would be