Materials Science and Engineering A 510–511 (2009) 317–321

Contents lists available at ScienceDirect

Materials Science and Engineering A

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

Effect of microstructure on the high-temperature deformation behavior of Nb–Si alloys

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ARTICLE INFO

Article history:
Received 20 November 2007
Received in revised form 25 March 2008
Accepted 3 April 2008

Keywords:
Nb
Silicide
Eutectic solidification
Eutectoid decomposition
Compression test

ABSTRACT

Deformation behavior of Nb–Si–Zr alloys is investigated at various temperatures ranging from R.T. to 1670 K. The master alloy ingots composed of Nb–18.1 at% Si–1.5 at% Zr doped with Mg are Ar-arc-melted. The ingots contain Nb rods (radius: 1 μm) in Nb_3Si matrix formed by eutectic reaction. Alloys are subjected to heat treatments at 1923 K for 4–100 h to obtain a large Nb network structure with small silicide (α-Nb_5Si_3) particles by decomposing Nb_3Si matrix into Nb and Nb_5Si_3 through a eutectoid reaction. Compression tests are conducted at room temperature in air and at elevated temperatures in Ar atmosphere. At 1471 K the maximum strength is 500 MPa and compressive ductility is higher than 10% with a strain rate of $1.0 \times 10^{-4}\ s^{-1}$, while at room temperature the maximum strength is over 1500 MPa and compressive ductility is about 1.5%. The high-temperature deformation obeys a power-law type equation. The stress exponent n is evaluated to be 4.8 and the apparent activation energy is 350 kJ/mol. The Vickers indentation at room temperature revealed that the crack propagation at room temperature is suppressed effectively by ductile Nb. This suggests that the Nb aggregate in the network structure acts as a large Nb grain containing fine Nb_5Si_3 particles, which might be beneficial for ductility at low temperatures.

1. Introduction

As energy consumption is an imminent problem in mass production, development of a more efficient combustion system is anticipated as the solution and a new class of heat-resisting materials are required. Although the Ni-based superalloys have been the superior heat-resistant materials, the melting point does not reach 1700 K which is the combustion temperature of the current high-performance jet engine. The melting point of Nb is about 1000 K higher than that of Ni, while its density is lower than that of Ni and any other refractory metals such as Mo, W and Ta. So, Nb-based material is one of the most promising materials. Since the 1960s, various Nb-based alloys have been developed for high-temperature applications [1], but further improvement of both strength and oxidation resistance at elevated temperatures were required. Since the mid-1980s, Nb–Nb_3Al two-phase alloys have been investigated [2], and also a broad research on Nb-Nb_5Si_3 two-phase alloys has been conducted because the Nb_5Si_3 intermetallic compound shows high strength at high temperature, which is suitable for the dispersoid in Nb solid solutions [3–6]. However, the maximum solid solubility of Si in Nb terminal solid solution is as small as 3.5 at.% which provides a very limited fraction of α-Nb_5Si_3 precipitate phase in the Nb matrix through any heat treatment. So far, it is quite a natural consequence to utilize the eutectic reaction to obtain metal-silicide in situ composite [4]. On the other hand, the authors have paid attention to combine a eutectic reaction at 2188 K (L – >Nb + Nb_3Si) and a eutectoid reaction at 2043 K (Nb_3Si – >Nb_5Si_3 + Nb) to obtain a microstructure with a high-volume fraction of fine Nb_5Si_3 dispersed in Nb grains through a sequence depicted in Fig. 1 [7–9]. Keys for the microstructure development are as follows.

(1) Fine Nb rods in the same eutectic cell have the same crystallographic orientation.
(2) The formation of Nb plates through the eutectoid reaction starts at Nb-rod/Nb_3Si–matrix interface. In the process Nb plates have the same crystallographic orientation with that of Nb-rods. Consequently, a eutectic microstructure turns into a Nb network without Nb grain boundaries.

For this microstructure control, Zr and Mg addition were found to be inevitable. Zr accelerates the eutectoid decomposition, and Mg accelerates the spheroidization of Nb_5Si_3 (Fig. 1) [7–10]. Although a similar microstructure was also reported by Kim et al. obtained by

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0921-5093/$ – see front matter © 2009 Published by Elsevier B.V.
doi:10.1016/j.msea.2008.04.123

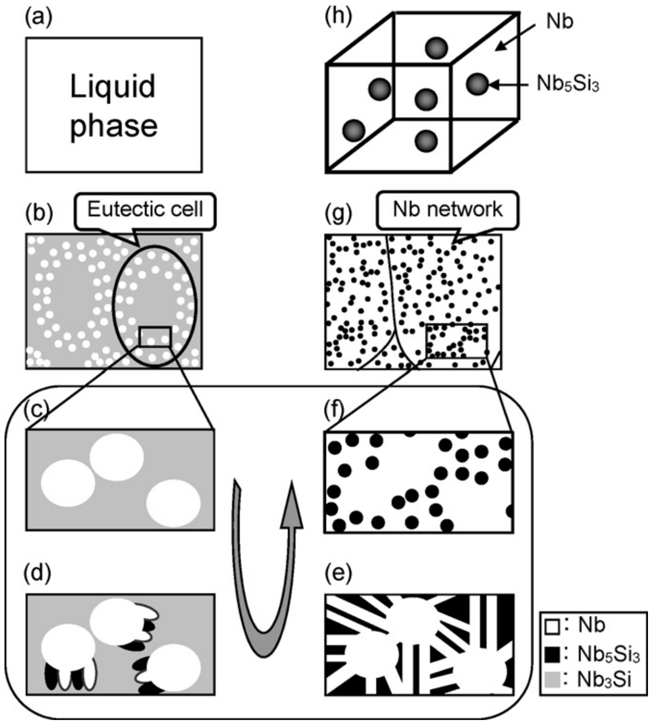

Fig. 1. A schematic illustration of the microstructure evolution from (a) liquid phase to (b) eutectic cells as the result of eutectic solidification, followed by the eitectoid reaction and spheroidization shown in (c)–(f), resulting in the Nb network with fine Nb_5Si_3 dispersoids ((g) and (f)).

extremely high temperature heat treatment of Nb–Mo–Si alloy, the alloys contain no Nb_3Si phase and the principles of the microstructure evolution might be different.

With the present microstructure composed of small Nb_5Si_3 dispersoids embedded in ductile Nb solid solution, the propagation of cracks initiated in the brittle Nb_5Si_3 during the deformation at room temperature is expected to be suppressed, and the small dispersoids act as effective obstacles for dislocation motion at high-temperature region. The purpose of this study is to understand the relationship between the newly developed microstructure and mechanical properties such as strength at high temperatures and toughness at R.T.

2. Experimental procedure

Recently, it was found that the microstructure which resembles a binary eutectic microstructure can be obtained in Nb–Si–1.5 at.% Zr alloys with Si composition around 18.1 at.% [11]. Several alloy ingots with a fixed composition of Nb–18.1Si–1.5Zr + 100ppm Mg are arc-melted. Heat treatments of alloy ingots wrapped by Ta foils were conducted under a high-purity Ar-flow atmosphere at 1923 K for 4–100 h. Microstructure observations were conducted on specimen surfaces carefully polished with colloidal SiO_2 powders (40-nm diameter) using both an electron probe microanalysis (EPMA, JEOL-JXA-8900 M) and an electron backscatter diffraction (EBSD) analysis attached to a field emission scanning electron microscope (JEOL-JSM-6500F) with TexSEM Laboratories-orientation imaging microscopy (OIM) software ver. 4.5.

Compression test specimens with 3 mm × 3 mm × 6 mm in dimension were cut from the heat-treated specimens by a wheel cutter and polished with emery papers. An Instron testing machine (model 5584) was used for the compression tests at various temperatures ranging from room temperature to 1670 K with an initial strain rate of $1.0 \times 10^{-4}\,s^{-1}$ or $1.0 \times 10^{-5}\,s^{-1}$. At high temperatures the compression tests were conducted under Ar atmosphere. Test temperature was measured by an R-type thermocouple near the specimen. In some of the compression tests the strain rate was alternately changed several times from the initial strain rate of $1.0 \times 10^{-4}\,s^{-1}$ to $1.0 \times 10^{-3}\,s^{-1}$ and $1.0 \times 10^{-5}\,s^{-1}$ during plastic deformation. Specimens compressed at a constant strain rate were investigated by scanning electron microscope (SEM) to understand the evolution of microstructure during high-temperature compression. Vickers test at room temperature with a constant load of 50 kg (490 N) for 15 s were conducted on specimens heat-treated for 100 h.

3. Results and discussion

3.1. Microstructure

The microstructures of as-cast and heat-treated specimens are shown in Fig. 2. The constituent phases are examined by EPMA. In Fig. 2(a), bright and gray areas are Nb and Nb_3Si, respectively, and the areas surrounded by circles are eutectic cells. In Fig. 2(b), bright and dark areas are Nb and Nb_5Si_3, respectively. The bright Nb phase has a tendency to connect each other, forming Nb networks. It was also confirmed by using EBSD analysis that each Nb area forming a network has an identical crystallographic orientation relationship [7], and the Nb network had a size almost the same with that of eutectic cells in as-cast specimens shown in Fig. 2(a).

3.2. Mechanical properties

3.2.1. Compression behavior at high-temperature range

Compressive stress–strain curves of heat-treated specimens with a strain rate of $1.0 \times 10^{-4}\,s^{-1}$ are shown in Fig. 3. In the

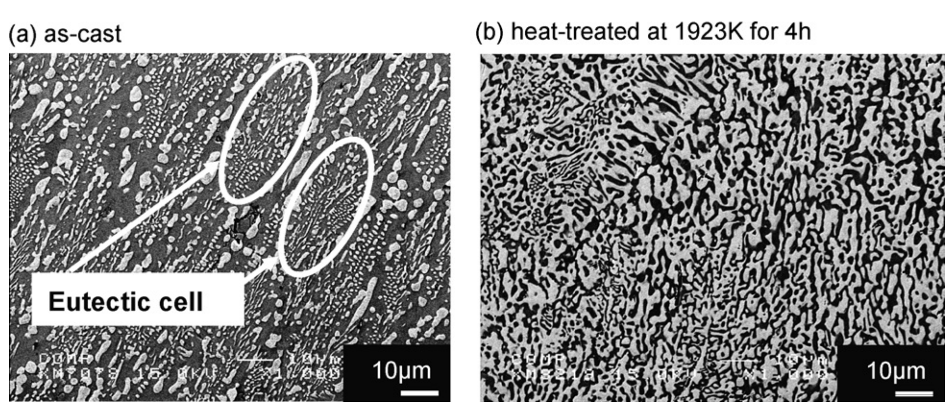

Fig. 2. The microstructure of (a) as-cast alloy and (b) heat-treated alloy at 1923 K for 4 h. Bright phase is Nb, and dark phase in (a) and in (b) are Nb_3Si and Nb_5Si_3, respectively, as determined by EPMA.