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Dislocation character and operative slip systems in α-Nb_5Si_3 tested at 1673 K

N. Sekido [a,*], S. Miura [b], Y. Yamabe-Mitarai [a], Y. Kimura [c], Y. Mishima [c]

[a] National Institute for Materials Science, Tsukuba 305-0047, Japan
[b] Division of Materials Science and Engineering, Hokkaido University, Sapporo 060-8628, Japan
[c] Department of Materials Science and Engineering, Tokyo Institute of Technology, Yokohama 226-8502, Japan

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ABSTRACT

Dislocation character and operative slip systems in α-Nb_5Si_3 were examined by transmission electron microscopy. Two-phase alloys comprised of (Nb) and α-Nb_5Si_3 were used in this study. Although few dislocations were present in the α-Nb_5Si_3 phase of a pre-deformed alloy, many developed after 15% of compressive deformation at 1673 K. Two types of the Burgers vectors were identified for α-Nb_5Si_3: <100] and 1/2 < 111]. The glide planes of dislocations were defined by the cross products between the Burgers vectors and the line vectors of the dislocations, by which the slip systems that operate in α-Nb_5Si_3 at 1673 K were determined as {011)<111], {100)<010], and {001)<100].

1. Introduction

Significant performance improvement of aircraft engines and land-based gas turbine engines requests the development of a new class of heat resistant materials with temperature capability beyond Ni-based superalloys. Refractory metal silicides are considered as candidate materials since some of them possess high strength and capability of forming protective oxide layers at elevated temperatures [1–3]. In terms of structural applications, however, major drawback is the brittleness at ambient temperatures in monolithic form. For this, a number of studies have demonstrated that one effective approach is an alloy design to develop multiphase microstructures with ductile phase(s), by which the brittle silicide matrices are toughened to enhance the resistance against crack propagation. At the same time, long-term stability of multiphase microstructure under elevated temperature regime is a matter of consideration. For example, attempts to increase the toughness of $MoSi_2$ by incorporating ductile metals, such as Mo, W, Nb, have had a limited success due to the severe interfacial reactions during annealing [4]. In this context, M_5Si_3-type silicides (where M is a refractory metal) are attractive since they are often equipped with high melting temperatures, densities lower than those of Ni-based alloys [5], excellent high temperature strength, and potential availability of ductile phase toughening with robust microstructures up to elevated temperatures.

Three types of the crystal structures that commonly appear in M_5Si_3-type silicides are $D8_l$ (I4/mcm, Cr_5B_3 type), $D8_m$ (I4/mcm, W_5Si_3 type), and $D8_8$ ($P6_3$/mcm, Mn_5Si_3 type). It has been demonstrated that $D8_8$ compounds show low creep resistance at elevated temperatures [6]. $D8_8$ and $D8_m$ compounds exhibit high anisotropy in thermal expansion coefficients and elastic moduli [7–10]. On the other hand, some $D8_l$ compounds possess high strength at elevated temperature [11–13], and nearly isotropic thermal expansion [8,14] and elastic property [8,15]. Despite such advantages, the $D8_l$ structure seems to be the least stable between the three structures in the binary systems. As depicted in a structure map for binary M_5Si_3 compounds shown in Fig. 1, the $D8_8$ structure most often formed, and the $D8_l$ structure only appears in Nb and Ta. Moreover Nb_5Si_3 and Ta_5Si_3 exhibit a polymorphic transformation from the high-temperature phase $D8_m$ to the low-temperature phase $D8_l$ in the binary systems, which complicates the preparation of $D8_l$ single crystals. Therefore, although multiphase alloys containing α-Nb_5Si_3 have been widely studied for potential high temperature structural application so far [12,17,18], limited information is available for physical and mechanical properties of α-Nb_5Si_3. Due to such restriction, we attempt here to characterize the deformation behavior of α-Nb_5Si_3 by using multi-phase

* Corresponding author. 1-2-1 Sengen, Tsukuba 305-0045, Japan. Tel.: +81 29 859 2467.
E-mail addresses: sekido.nobuaki@nims.go.jp (N. Sekido), miura@eng.hokudai.ac.jp (S. Miura), mitarai.yoko@nims.go.jp (Y. Yamabe-Mitarai), kimura.y.ac@m.titech.ac.jp (Y. Kimura), mishima.y.ab@m.titech.ac.jp (Y. Mishima).

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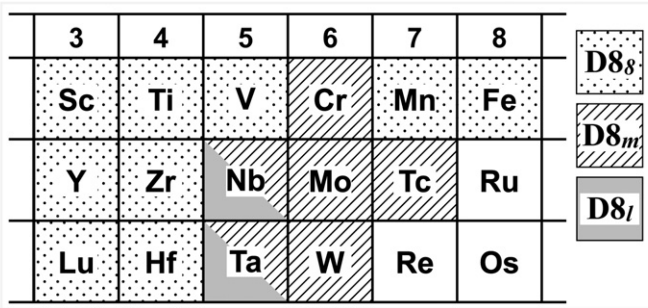

Fig. 1. Structure Map for M_5Si_3 compounds in M–Si binary systems.

polycrystalline alloys comprised of (Nb) and α-Nb_5Si_3. The slip systems that operate in α-Nb_5Si_3 are examined through characterization of dislocations developed during compression tests at 1673 K.

2. Experimental procedures

Rod ingots (φ11 mm × 70 mm) with the nominal composition of Nb–10at.%Ti–17.5at.%Si were preliminary prepared by arc-melting of high purity raw materials under an Ar atmosphere. These ingots were unidirectionally solidified in an optical floating zone furnace with the solidification rate of 10 mm/h under an Ar atmosphere. The effect of solidification rates on microstructures has been already discussed elsewhere [16]. Previous studies have also demonstrated that the decomposition of the high temperature phase Nb_3Si into the equilibrium (Nb)/α-Nb_5Si_3i two phases through an eutectoid reaction is so sluggish that the Nb_3Si phase is retained in the as-solidified condition [17,18]. Thus the directionally solidified alloys were annealed at 1673 K for 500 h, by which retained high temperature phase Nb_3Si completely transforms into (Nb) and α-Nb_5Si_3 [19,20]. High temperature compression tests were conducted under vacuum at temperatures up to 1673 K with a nominal strain rate of $1.0 \times 10^{-4}\ s^{-1}$. The specimens (2 mm × 2 mm × 5 mm) were prepared by EDM to tailor the loading axis to the solidification direction. Microstructures of the alloys were characterized by scanning electron microscopy and transmission electron microscopy. TEM foils were perforated with an ion-miller with the acceleration voltage of 4 kV.

3. Results

As has been reported previously [16,19], an as-directionally-solidified Nb–10Ti–17.5Si alloy exhibits a two-phase microstructure comprised of the (Nb) phase and the high temperature phase Nb_3Si. After annealing at 1673 K for 500 h, the Nb_3Si phase decomposes into (Nb)/α-Nb_5Si_3 lamellae through a eutectoid reaction. Fig. 2 shows a typical microstructure of the annealed Nb–10Ti–17.5Si alloy, where fine-scaled (Nb)/α-Nb_5Si_3 lamellae develop with large (Nb) grains that are grown along the solidification direction. This alloy has been found to show some compressive deformability at 1673 K, where no macroscopic cracks were visible on the surface of the specimen after 15% of compressive deformation [19].

Fig. 3 shows the bright-field images of (Nb)/α-Nb_5Si_3 lamellae before and after the compressive deformation. In the pre-deformed sample, few dislocations are observed in α-Nb_5Si_3, as shown in Fig. 3a. After the compression, a number of dislocations are found to develop as shown in Fig. 3b. This indicates that α-Nb_5Si_3 can plastically deform at 1673 K. Dissociation of dislocations was not observed in the α-Nb_5Si_3 phase.

The Burgers vectors of dislocations that developed in α-Nb_5Si_3 were determined by the weak-beam thickness fringe method [21].

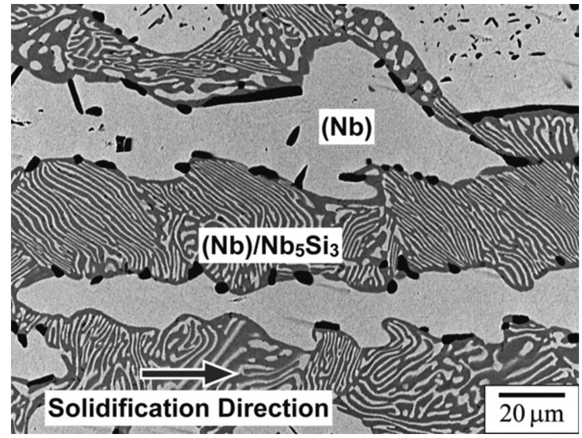

Fig. 2. A SEM micrograph of the Nb–10Ti–17.5Si alloy directionally solidified at 10 mm/h, followed by annealing at 1673 K for 500 h.

The diffracting vector, ***g***, the Burgers vector of a dislocation, ***b***, and the number of fringes terminated at the dislocation end, *n*, satisfy the following equation:

$$\boldsymbol{g}\cdot\boldsymbol{b} = n \tag{1}$$

Thus, the Burgers vector of a dislocation can be identified by counting the excess thickness contours terminating at the end of each dislocation line imaged in weak-beam dark-field mode under, at least, three non-coplanar reflection vectors. Fig. 4 shows weak-

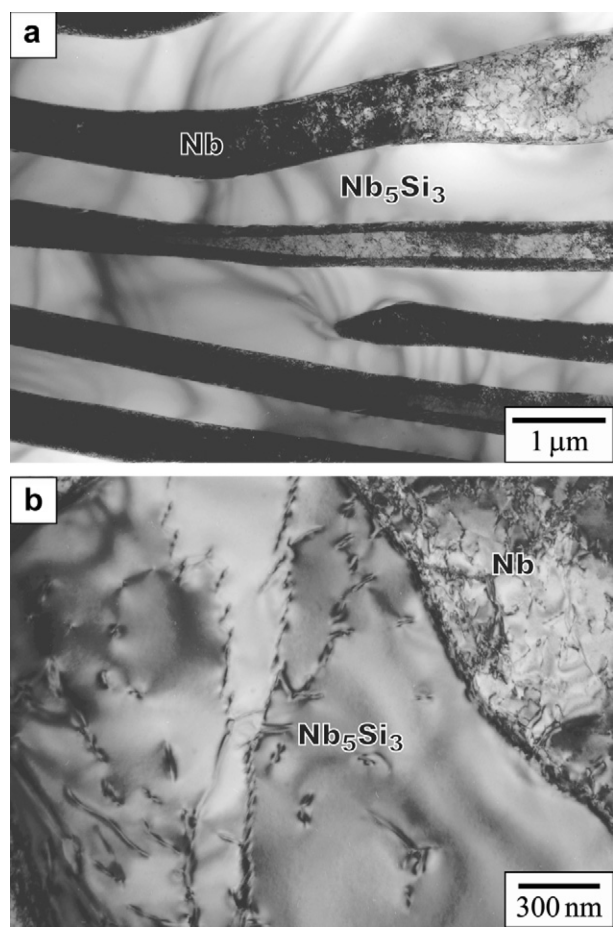

Fig. 3. Bright-field images of (Nb)/α-Nb_5Si_3 eutectoid lamellae (a) before and (b) after compression at 1673 K.