



Full length article

# Creep-deformation behavior of $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ lamellar-structured C40/C11<sub>b</sub> two-phase crystals

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## ABSTRACT

The creep-deformation behavior of  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  crystals that were composed of C40 and C11<sub>b</sub> two-phases with an oriented lamellar microstructure was examined over 1200–1400 °C under compression. The creep behavior of the crystals varied greatly depending on the orientation of the loading axis with respect to the lamellar interfaces. The steady-state creep strain rate (SSCR) when the loading orientation was parallel to the lamellar interfaces (0°-orientation) was approximately 2 orders of magnitude lower than that when the loading orientation was inclined by 45° (45°-orientation). Creep tests were performed also with single crystals of C40-single-phase  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ , and proved that the C40-phase effectively acts as a strengthening phase in the creep behavior of the C40/C11<sub>b</sub> duplex-phase crystals. The variant-1-type C11<sub>b</sub> phase grains, which have a loading orientation parallel to [001], were also found to act as an effective strengthening component. The SSCR of the 0°-orientation was succeeded to be further decreased by controlling the lamellar microstructure via the addition of Cr. The SSCR of the 1 at.%-Cr-added lamellar-structured crystal under 300 MPa in the 0°-orientation was approximately a third of that of the non-added ternary crystal.

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## 1. Introduction

There is a great demand for materials that can tolerate operating temperatures greater than 1200 °C, i.e., higher than that of Ni-based superalloys. Transition-metal disilicides (TMDs) have received much attention because they are promising candidates for such ultra-high-temperature (UHT) structural materials.  $\text{MoSi}_2$  is one of the most promising TMDs because of its high melting temperature, good oxidation resistance, relatively low density, and high thermal conductivity [1–5].  $\text{MoSi}_2$  crystallizes in the C11<sub>b</sub> structure, which is based on a tetragonal unit cell. The relatively high crystal symmetry of this structure means that many slip systems, such as {110}<111] and {013}<331], can be operative [1,4]. Here, the mixed notation of {hkl} and <uvw> differentiates the first two indices from the third index, which does not play the same role as the first two because of the tetragonality of the C11<sub>b</sub>- $\text{MoSi}_2$  system. Owing to the operation of those slip systems,  $\text{MoSi}_2$  exhibits some level of plasticity even at room temperature [4,5]. However, the serious drawbacks of a low fracture toughness at low temperatures and

insufficient high-temperature creep strength must be improved before  $\text{MoSi}_2$  is used in practical applications.

We have proposed to combine different TMDs that have similar properties as a facile approach to overcome the aforementioned problems [6,7]. In particular, C40-structured  $\text{NbSi}_2$ , which has a hexagonal unit cell, is the most promising candidate for strengthening C11<sub>b</sub>- $\text{MoSi}_2$  [3,6–16]. The C11<sub>b</sub> and C40 crystal structures are closely related to each other because the {110} atomic arrangement in the tetragonal C11<sub>b</sub> structure is almost identical to the (0001) arrangement in the hexagonal C40 structure. The structures only differ from each other in the stacking sequences of the common atomic layers, with the C11<sub>b</sub> and C40 structures based on two-fold and three-fold stacking sequences, respectively. By focusing on the similarity of the crystal structures, we succeeded in producing a C40/C11<sub>b</sub> duplex-phase crystal with an oriented lamellar microstructure [7]. Fig. 1(a) shows the microstructure of the resulting duplex-phase crystal. The oriented lamellar microstructure was developed by annealing a Mo-oversaturated  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  single-crystal with the C40 structure, which was grown via the floating-zone (FZ) method. Owing to the similarity of the aforementioned crystal structures, C11<sub>b</sub> phases were precipitated in the C40 single crystal during the annealing process so as to their

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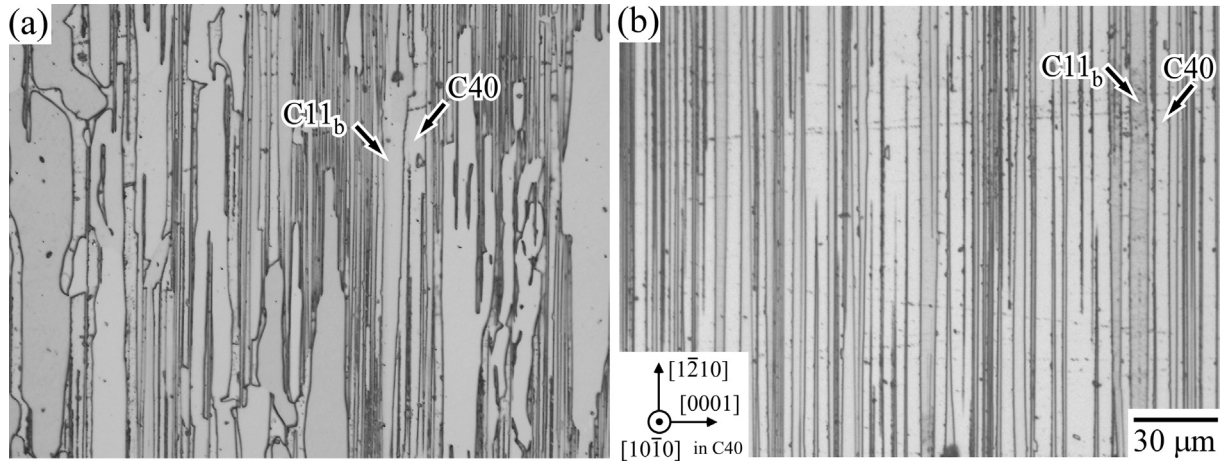


Fig. 1. OM images of the microstructures of FZ-grown crystals after being annealed at 1400 °C for 168 h (a)  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  and (b) 1 at.% Cr-added crystals.

common planes are aligned parallel to each other at the lamellar interfaces. Hence, the C40 and C11<sub>b</sub> phases exhibited distinct crystallographic orientation relationships of  $(0001)_{\text{C40}}//\{110\}_{\text{C11}_b}$  and  $\langle\bar{1}2\bar{1}0\rangle_{\text{C40}}//\langle\bar{1}\bar{1}0\rangle_{\text{C11}_b}$ . However, on the common plane, there was a slight distortion in the atomic arrangements; the [110] axis of the C11<sub>b</sub> structure and [0001] axis of the C40 structure possess two-fold and six-fold rotational symmetries, respectively. Therefore, three variants of the C11<sub>b</sub> phase, which were designated variants 1 (V1), 2 (V2), and 3 (V3), were formed within the C40 single-crystal, and are represented by the following orientation relationships.

$$\text{V1: } (0001)_{\text{C40}}// (110)_{\text{C11}_b}, \quad [\bar{1}2\bar{1}0]_{\text{C40}}// [1\bar{1}0]_{\text{C11}_b}, \quad [10\bar{1}0]_{\text{C40}}// [001]_{\text{C11}_b} \quad (1)$$

$$\text{V2: } (0001)_{\text{C40}}// (110)_{\text{C11}_b}, \quad [2\bar{1}\bar{1}0]_{\text{C40}}// [1\bar{1}0]_{\text{C11}_b}, \quad [0\bar{1}10]_{\text{C40}}// [001]_{\text{C11}_b} \quad (2)$$

$$\text{V3: } (0001)_{\text{C40}}// (110)_{\text{C11}_b}, \quad [\bar{1}\bar{1}20]_{\text{C40}}// [1\bar{1}0]_{\text{C11}_b}, \quad [\bar{1}100]_{\text{C40}}// [001]_{\text{C11}_b} \quad (3)$$

This was actually confirmed by the selected area electron diffraction (SAED) pattern analysis in the transmission electron microscopy (TEM) observation [7], as indicated in the appendix Fig. A.1(a–c). These crystallographic orientation relationships (hereinafter referred to as variant orientation relationships) are schematically drawn in appendix Fig. A.1(d). The compositions of the C40 and C11<sub>b</sub> phases in the lamellar crystal were evaluated to be approximately  $(\text{Mo}_{0.79}\text{Nb}_{0.21})\text{Si}_2$  and  $(\text{Mo}_{0.94}\text{Nb}_{0.06})\text{Si}_2$ , respectively [6].

The development of a lamellar microstructure effectively increases the room-temperature fracture toughness of  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  crystals [10,15]. In addition, C40/C11<sub>b</sub> crystals exhibit superior high-temperature strength compared to the polycrystalline  $\text{MoSi}_2$  [6,11]. Thus, lamellar-structured  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  crystals have a potential as a next-generation UHT structural material. It must be focused here that both the fracture toughness and high-temperature strength were found to exhibit strong orientation dependencies with respect to the lamellar interfaces [10,11,15]. Thus, controlling the microstructure and loading orientation in the C40/C11<sub>b</sub> duplex-phase crystals is essential for obtaining superior mechanical properties.

Regarding the control over the lamellar microstructure, we found a problem with its thermal stability. As shown in Fig. 1(a), some coarse C11<sub>b</sub>-phase grains were developed in addition to the fine lamellar-structured C11<sub>b</sub> grains during the long-term annealing of the ternary  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  crystals. These coarse C11<sub>b</sub>-phase grains do not exhibit the aforementioned variant orientation relationships, and they are thought to form as a way of releasing the residual strain accumulated at the lamellar interfaces from the formation of fine lamellae [7]. We have recently shown that the thermal instability of the fine lamellar microstructure is drastically improved by the small addition of Cr [14,16]. As shown in Fig. 1(b), the almost all of coarse C11<sub>b</sub> grains without having the variant orientation relationship disappeared, and the Cr-added duplex-phase crystal was entirely composed of the fine lamellar microstructure. The lamellar microstructure was confirmed to be stable even after annealing for 504 h at 1400 °C [17]. The origin of the improved thermal stability of the microstructure, accompanied by the segregation of added Cr atoms on the lamellar interface, has been discussed with the help of computational analyses [18,19].

In the present study, we examined the creep properties of C40/C11<sub>b</sub> duplex-phase crystals for the first time. The creep-deformation behavior was investigated via compressive creep tests at temperatures above 1200 °C, with the orientation dependence examined in detail. By observing the deformation microstructure, the factors controlling the creep behavior were identified. In addition, the effects of the Cr addition on the creep-deformation behavior were also examined by focusing on the influence of variation in lamellar microstructure.

## 2. Experimental methods

Mother alloys with chemical compositions of  $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$  and  $(\text{Mo}_{0.85}\text{Nb}_{0.15})_{0.97}\text{Cr}_{0.03}\text{Si}_2$  (the numbers are in atomic fractions; the alloys will hereinafter be referred to as ternary and Cr-added alloys, respectively) were prepared by arc-melting high-purity Mo, Nb, Si, and Cr under an Ar atmosphere. Single crystals were grown from the mother alloys with an optical floating-zone furnace (SCI-MDH-20020, Canon Machinery) at a growth rate of 2.5 mm/h under an Ar gas flow. The Laue X-ray diffraction method was used to confirm that the single crystals were composed of a C40-single-phase. The C40-phase single crystals were then annealed at 1400 °C for 168 h under an Ar atmosphere to develop the oriented lamellar microstructure, which is composed of C40 and C11<sub>b</sub> phases as

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