Single-crystal growth and plastic deformation behaviour of a Ti-15Mo-5Zr-3Al alloy for biomedical application

S-H Lee, K Hagihara, M-H Oh and T Nakano

1Division of Materials and Manufacturing Science, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan
2Department of Information & Nano Materials Engineering, Kumoh National Institute of Technology, 1 Yangho-dong, Gumi, Gyeongbuk 730-701, Korea
E-mail: nakano@mat.eng.osaka-u.ac.jp

Abstract. A Ti-15Mo-5Zr-3Al alloy with a bcc structure are promising materials for biomedical application and were examined. The focus of this study was on the effect of heat treatment on microstructure and plastic deformation behaviour using a single crystal. The single crystal was successfully obtained by a floating zone method at a crystal growth rate of 2.5 mm/h. A slip at the <111> dislocation was present irrespective of heat treatment at 573K or 673K for 1.2 ks or 300 ks. The yield stress at the [149] loading axis varied significantly depending on microstructure, especially from the precipitation of the α phase. Al addition suppresses generation of the ω phase and increases the yield stress at the same time.

1. Introduction
Recently, β-type titanium alloys with a bcc structure have received a great deal of attention as structural and biomedical materials because of their excellent mechanical properties, good cold formability, low Young’s modulus etc. [1]. Various types of β-Ti alloys have been developed for engineering parts and also biomedical applications [2, 3]. Ti-15Mo-5Zr-3Al (wt.%) alloy is one of the most promising materials for biomedical applications with outstanding bio-compatibility as well as mechanical characteristics [4]. The alloy was accepted for commercial use by being registered in ISO in 2007. Many studies on the effect of heat treatment on microstructure and deformation behaviour for this alloy have been reported. However, to obtain more comprehensive knowledge such as the crystal orientation dependence of plastic and elastic behaviour, further studies using single crystals are required. Hanada et al. previously examined plastic deformation behaviour of Ti-15Mo-5Zr (wt.%) using a single crystal [5, 6, 7] that had a similar composition to Ti-15Mo-5Zr-3Al. Concerning the effect of Al-addition to the microstructure in β-Ti alloys, Ikeda et al. reported a Ti-V system [8] wherein Al-addition enhances precipitation of the α-phase during annealing since Al is an α-phase stabilizing element. In addition, they determined that Al-addition hinders the formation of the athermal and the isothermal ω-phase. They also investigated the effect of Al-addition in Ti-15Mo-5Zr-3Al using a polycrystal [9] and made similar conclusions. Their experiment was not done by the direct observation by TEM but through measuring the variation in electric resistivity during annealing.

In this study, the influence of heat-treatment on the microstructure and the plastic deformation behaviour in a Ti-15Mo-5Zr-3Al alloy was examined using a single crystal. The results will be compared with those for the Ti-15Mo-5Zr reported by Hanada et al. and the effect of Al-addition to this alloy system will also be discussed.
2. Experimental procedure

The mother alloy with a composition of Ti-15Mo-5Zr-3Al (wt.%) was supplied by Kobe Steel Co. Ltd. The single crystal was grown by an optical floating zone melting method in high-purity Ar gas flow at a crystal growth rate of 2.5 mm/h [10]. The obtained single crystal was solution treated (ST) at 1108K for 3.6 ks in an Ar atmosphere, followed by water quenching. In some of the ST specimens, heat-treatment was further carried out at 573K and 673K for 1.2 ks and 300 ks, which is the same as that reported by Hanada et al. for Ti-15Mo-5Zr [5, 6]. The constituent phases in specimens were examined by transmission electron microscopy (TEM). For compression tests, rectangular specimens with dimensions of 2×2 mm×5 mm were cut out. The loading axis was selected to be parallel to [T49], where the Schmid Factor (SF) for the (101) [111] primary slip had a maximum value of 0.5. The tests were carried out at room temperature at a nominal strain rate of 1.67×10⁻⁴ s⁻¹ in vacuum.

3. Results

Figure 1 shows electron diffraction patterns (DPs) along the [113] direction and corresponding dark-field images in ST and annealed specimens. In the ST specimen, only the spots derived from the β-phase was observed and no other extra spot was detected, although very weak streaks were observed along the <12\bar{T}> direction as shown in Fig. 1(a). This indicates that the ST specimen is mainly composed of the β-phase. No significant difference was observed in the DPs for specimens from short-time annealing (1.2 ks both at 573K and 673K) as seen in Fig. 1(c), (e). No clear precipitates were observed from the dark-field observation (Fig. 1(d), (f)) although a slight increase in the intensity of the streaks was detected, especially at 673K. On the other hand, a large amount of ω-precipitates were formed and clear spots, derived from the four ω-variants, appeared after long-time annealing for 300 ks at 573K as shown in Fig. 1(g). For the specimen annealed at 673K, the observed zone DP was strongly distorted because of the large amount of precipitates. Many extra spots appeared between the fundamental spots of the β-matrix phase and these were identified as spots that derived from the α-phase and not from the ω-phase. Indeed, in the dark-field image the large lath-like shapes of α-precipitates were abundant as shown in Fig. 1(j).

Figure 2 shows the change in room temperature yield stress according to different heat-treatment conditions and compressed at the [T49] loading axis. The yield stress in the as-ST specimen was about 670 MPa. The yield stress increased as the annealing time and temperature increased but the increment was small for the short-time annealing at 1.2 ks both at 573 and 673K. The increment in stress became relatively large and was about 800 MPa for the long-time annealing specimen at 300 ks and 573K. In the specimen annealed at 673K for 300 ks the yield stress showed a significant increment and was about 1285 MPa.

![Figure 1](image_url)

**Figure 1.** Electron diffraction patterns along the [113] direction (a, c, e, g, i) and corresponding dark-field images (b, d, f, h, j) in ST (a, b) and annealed specimens at 573K for 1.2 ks (c, d), 673K for 1.2 ks (e, f), 573K for 300 ks (g, h) and 673K for 300 ks (i, j). Spots used for the dark-field observation are indicated by circles in the DPs.
With regard to ductility of the specimens, at least 20% of the compressive strain could be obtained in all specimens except for the specimen annealed at 673K for 300 ks, which showed very high stress but it broke at a strain of less than 10%.

Figure 3 shows the slip traces of the specimens deformed to 1% plastic strain as observed on their (I2I) and (I15I) side surfaces. The morphology of deformation markings indicates that the deformation occurred by slips in all specimens, irrespective of heat-treatment. The contrast of slip traces is strong on the (I15I) but is very faint on the (I2I) face, indicating that the Burgers vector of dislocations is parallel to [111]. By a two-face slip analysis the slip plane of the [111] dislocation was determined to be nearly (312), which is a slight deviation from the (101) maximum resolved shear stress plane at the [149] loading axis. Such a deviation of the slip plane from (101) toward (211) was also reported for Ti-15Mo-5Zr single crystals [5] and it is due to a dislocation core structure effect in the bcc-structured crystal.

Although the same [111] slip system was operative, independent of annealing conditions, the morphology of the traces was slightly different for each specimen. Slip traces were introduced relatively homogeneously to the ST specimen and no large difference in features was observed for specimens of short-time annealing at 1.2 ks and 573K or 673K, as shown in Fig. 3 (c, d and e, f). However, in the specimen annealed at 573K for 300 ks, in which a large amount of \( \omega \)-phases precipitated, the distribution of slip traces became very coarse and localized. In the specimen aged at 673K for 300 ks, in which formation of \( \alpha \)-precipitates occurred, the contrast of slip traces became very faint and difficult to identify.

Figure 3. Optical microscope images of slip traces after compression tests to 1% plastic strain as observed on (I2I) (a to i) and (I15I) side surfaces (b to i) after ST (a, b). Annealed specimens at 573K for 1.2 ks (c, d), 673K for 1.2 ks (e, f), 573K for 300 ks (g, h) and 673K for 300 ks (i, j). The loading axis was chosen as the horizontal direction in the figure.
4. Discussion

We clearly determined that the microstructure and plastic deformation behaviour of a β-type Ti-15Mo-5Zr-3Al single crystal depends on annealing conditions. In this section, our result is discussed by comparison to that of Ti-15Mo-5Zr as reported by Hanada et al. and the effect of Al addition on this alloy system is also considered. Our first focus was on the difference between microstructures. The observed microstructure showed a significant difference between our Al-addition alloy and the Ti-15Mo-5Zr alloy, even though heat-treatments were conducted under using equivalent conditions for both single crystals.

The large difference was due to a suppression of ω-phase precipitation in Ti-15Mo-5Zr-3Al. For Ti-15Mo-5Zr it was reported that precipitation of the ω-phase occurred even at short-time annealing for 1.2 ks and 573K. The ω-phase precipitation was clearly recognizable at 673K. For Ti-15Mo-5Zr-3Al, the ω-phase precipitation was not clear after the short-time annealing, even at 673K, and the alloy was only composed of the β-phase although a slight increase in intensity of streaks along <121> was accompanied by annealing. These results are in good agreement with previous reports by Komatsu et al. [9] and Hanada et al. [11]. Al acts as a strong inhibitor for the formation of the ω-phase.

These changes in microstructure gave a strong influence on the plastic deformation behaviour. In Ti-15Mo-5Zr-3Al, the yield stress did not show a significant change after the short-time annealing compared with that of the ST specimen. This obviously indicates that the suppression of ω-phase precipitation in Ti-15Mo-5Zr-3Al reduces an increase in yield stress by annealing. However, in case of annealing to 300 ks at 573K the annealing led to ω-phase precipitation even in Ti-15Mo-5Zr-3Al resulting in a relatively large increase in yield stress. On the other hand, in the specimen annealed for a long-time at 300 ks and 673K large amounts of the α-phase precipitation occurred. This caused a significant increase in yield stress, although α-phase precipitation was accompanied by a strong reduction in ductility.

5. Conclusions

A single crystal of Ti-15Mo-5Zr-3Al (wt.%) was successfully grown by a floating zone method at a growth rate of 2.5 mm/h in a high purity Ar gas atmosphere. The microstructure and plastic deformation behaviour of the Ti-15Mo-5Zr-3Al single crystal varied significantly depending on the heat-treatment. For Ti-15Mo-5Zr-3Al the suppression of ω-phase precipitates compared with that of Ti-15Mo-5Zr and the effect of Al was clearly confirmed by TEM observations. The variation in microstructure leads to a large change in yield stress and this depends on the annealing condition.

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References
[1] Niinomi M 2003 Biomaterials 24 2673
[2] Inamura T, Hosoda H, Wakashima K, and Miyazaki S 2005 Mater. Trans. 46 1597
[3] Matsumoto H, Watanabe S, and Hanada S 2005 Mater. Trans. 46 1070
[4] Okazaki Y, Ito Y, Kyo K, and Tateishi T 1996 Mater. Sci. Eng. A 213 138
[5] Hanada S, and Izumi O 1980 Titanium’ 80 Science and Technology 691
[6] Hanada S, and Izumi O 1980 Metall. Trans. A 11 1447
[7] Hanada S, and Izumi O 1982 Trans. Jpn. Inst. Metals 23 85
[8] Ikeda M, Komatsu S, Sugimoto T, and Kamei K 1994 J. Jpn. Inst. Light Met. 44 35
[9] Komatsu S, Ikeda M, Sugimoto T, Kamei K, Maesaki O, and Kojima M 1996 Mater. Sci. Eng. A 213 61
[10] Nakano T, Hagihara K et al 2007 Ti-2007 Science and Technology 1437
[11] Hanada S, and Izumi O 1980 Trans. Jpn. Inst. Metals 21 201