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Formation of Microstructures in the Molybdenum-Aluminum-Titanium Ternary System through Eutectoid Decomposition†

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Formation and evolution processes of microstructure of Mo–Al alloys retarded by addition of 10 at%Ti are studied through various heat treatments. Fine acicular Mo_3Al precipitates are observed inside a grain for a specimen quenched at 1673 K as an initial stage of the eutectoid decomposition of bcc phase. It suggests that there is a crystallographic orientation relationship between the bcc matrix and A15 precipitates. By changing heat treatment conditions, fraction of fine lamellar structure decreases and spheroidized area increases. Subsequent aging treatment at lower temperature also provides the spheroidized structure. A coarse equiaxed structure is observed for a specimen quenched at 1673 K then aged at 1173 K.

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Keywords: *lamellar structure, eutectoid decomposition, molybdenum-aluminum system, molybdenum-aluminum-titanium system, molybdenum aluminide*

I. Introduction

Present authors have investigated the mechanical properties of Mo–Al binary alloys with a lamellar structure composed of Mo_3Al and Mo_3Al_8 phases by a eutectoid decomposition of MoAl phase with a bcc structure shown in **Fig. 1**[1], and reported that a coarse lamellar structure with the mean interlamellar spacing of 1–3 μm is effective for suppressing the crack initiation in brittle phases around Vickers indents[2]. It has been suggested that the microstructural control is required to optimize the mechanical properties of this alloy. Due to the quick decomposition process in binary Mo–Al system, however, the knowledge of the formation process of the lamellar structure is seriously lacking. From the information on the ternary phase diagram and DTA examination, it is expected that Ti addition to Mo–Al system is valid for decreasing the eutectoid temperature by stabilizing the bcc phase, which also decomposes to Mo_3Al and Mo_3Al_8 phases. In this study, systematic SEM and TEM observations are conducted on a Mo–Al alloy with 10 at% of Ti to reveal the initial stage of the eutectoid decomposition from the bcc phase to Mo_3Al and Mo_3Al_8 phases as well as the evolution process of lamellar structure.

II. Experimental Procedures

Alloy ingots with a composition of Al–38 at%Mo–10 at%Ti were arc-melted with high purity Mo, Al and Ti in an argon atmosphere. The button ingots were cut by a wheel cutter followed by various heat treatments. The eutectoid temperature for the alloy was measured to be 1558 K by DTA with a heating and cooling rate of 5 K/min in an argon atmosphere. The temperature for homogenization treatment was then determined to be 1673 K, about 100 K higher than the eutectoid temperature measured, and samples were homogenized for 259 ks. Then, some specimens were continuously cooled to 1373 K or 1173 K, with a cooling rate of 20 K/min or 30 K/min followed by quenching into water. Also to study aging behavior, one specimen was aged at 1173 K for 259 ks after homogenization, and the other was continuously cooled from 1673 to 1373 K and aged for 21.6 ks. Samples were polished by conventional metallographic method. Microstructural observations were conducted by a SEM operated at 15 kV and a TEM operated at 200 kV. Compositions of constituent phases were analyzed by EDS attached to the SEM.

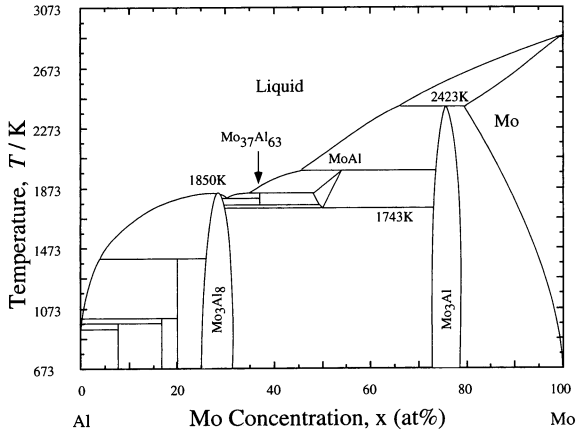


Fig. 1 Mo–Al binary phase diagram.

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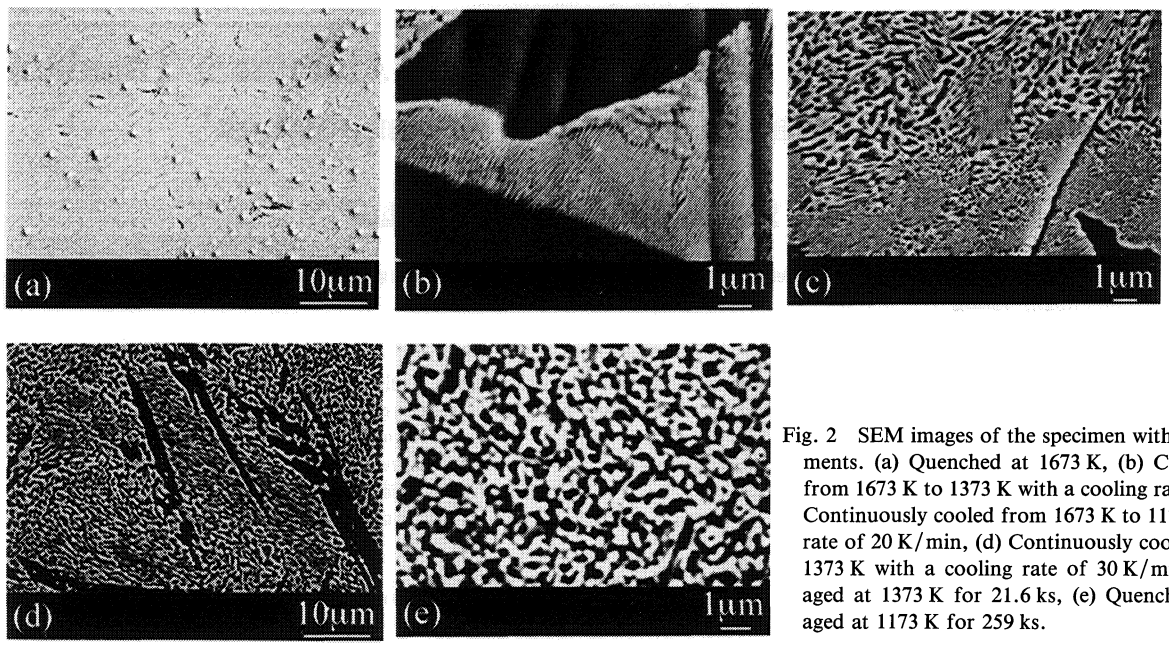


Fig. 2 SEM images of the specimen with various heat treatments. (a) Quenched at 1673 K, (b) Continuously cooled from 1673 K to 1373 K with a cooling rate of 30 K/min, (c) Continuously cooled from 1673 K to 1173 K with a cooling rate of 20 K/min, (d) Continuously cooled from 1673 K to 1373 K with a cooling rate of 30 K/min and successively aged at 1373 K for 21.6 ks, (e) Quenched at 1673 K then aged at 1173 K for 259 ks.

III. Results

1. SEM observation

A series of SEM images in **Fig. 2** show the microstructures of Mo–Al–Ti alloys with various heat treatments. Bright regions are identified as Mo_3Al phase and dark regions as Mo_3Al_8 phase, respectively by EDS. There is no significant difference between the reported microstructures of binary Mo–Al alloys[3] and those of the present alloys. There seems to be a single phase for the specimen quenched at 1673 K as shown in Fig. 2(a). As shown in Fig. 2(b), finely decomposed regions are observed in the specimen rapidly cooled with a rate of 30 K/min. The decomposed region is composed of colonies with fine lamellae. The mean interlamellar spacing is approximately 200 nm. The uniform distribution of fine colonies in the specimen suggests that the eutectoid decomposition in the Mo–Al–Ti alloys start not only at grain boundaries but also in the interior of grains. This microstructure is almost identical to those of binary Mo–Al alloys quenched from the bcc phase region. By a cooling rate of 20 K/min, fine lamellar colonies were also formed and spheroidized regions were confirmed in Fig. 2(c). The mean interlamellar spacing of fine lamellae is almost the same as Fig. 2(b), and the size of spheroidized structures is about 1 μm. With decreasing the cooling rate, the fraction of spheroidized area increases and the lamellar colony size becomes larger. A spheroidized structure is also observed in the specimen cooled with a rate of 30 K/min to 1373 K and continuously aged for 21.6 ks as shown in Fig. 2(d). This morphology appearing in Fig. 2(d) resembles to that of a binary Mo–Al alloy formed by a similar heat treatment[2]-[4]. From these results, it can be concluded that Ti addition slows down the decomposition kinetics without any changes of microstructural formation and evolution path. In the specimen that was quenched at 1673 K followed by aging at 1173 K, it was shown in Fig. 2(e) that the equiaxed grain precipitation is dominant. This suggests grain boundaries do not act as preferential sites in the present alloy.

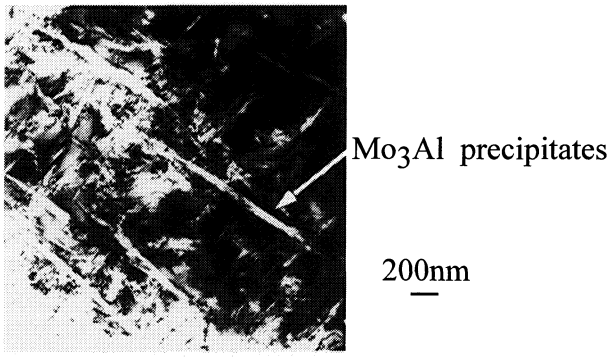


Fig. 3 Microstructure of the specimen quenched at 1673 K. White acicular precipitates are identified to be Mo_3Al phase.

2. TEM observation

To reveal the detail of the precipitation stage prior to the eutectoid decomposition, TEM observation was conducted for the specimen quenched at 1673 K whose microstructure observed by a SEM is shown in Fig. 2(a). As shown in **Fig. 3**, acicular precipitates with a width less than 100 nm are recognized in the matrix and these are identified as Mo_3Al phase by the electron diffraction pattern. The precipitates, which were formed during the quenching sequence from 1673 K to room temperature, align in some crystallographic orientations of the matrix, and misfit dislocations are not observed at around precipitates at least in conventional TEM.

IV. Discussion

1. Microstructural formation

Microstructures shown in Figs. 2(b)–(d) have revealed that the formation and evolution processes of lamellar structure are common in both binary and ternary alloys. According to the isothermal section of Mo–Al–Ti system[5], the addition of Ti to bcc MoAl phase is expected to decrease the temperature for the eutectoid decomposition into Mo_3Al and Mo_3Al_8. This is also confirmed by the present DTA work. Such a reduction of the eutectoid decomposition temperature due to the addition of Ti provides an advantage for observing the early stage of decomposition process of these alloys, because even water quenching can not suppress the decomposition for binary alloys[3]. As shown in Fig. 3, acicular precipitates appear prior to the eutectoid decomposition. Since the bcc matrix and the A15 Mo_3Al precipitates differ both in crystal structure and in composition, one tends to think incoherent precipitation reaction took place. Some orientation relationships between the bcc solid solution and A15 precipitates, however, have been reported in the literature for Nb–Al[6] or Nb–Al–Ti[7] systems. Therefore, it may be suggested that the acicular morphology observed in the present alloy is also caused by a certain crystallographic orientation relationship between the bcc Mo–Al–Ti solid solution and A15 Mo_3Al precipitates. Before drawing any reliable conclusions, one needs detailed diffraction analysis, which still remains as a future subject.

In view of the comparable size of an acicular precipitate and a fine lamellar, the latter is expected to be formed from the former. When an acicular Mo_3Al precipitates from the disordered bcc phase, a solute-rich atmosphere is formed around the precipitate by a short-range bulk diffusion, a subsequent precipitation of Mo_3Al_8 phase in the vicinity of the acicular precipitate is expected. At this stage it is speculated that a contribution of the interfacial energy between Mo_3Al and Mo_3Al_8 becomes more dominant against the strain energy contribution, which allows the platelet growth to form a lamellar structure in the alloy.

In the previous study, we pointed out lamellar has a tendency to spread out from a primary Mo_3Al[3] in the binary alloy. This can be explained by the early precipitation of Mo_3Al from primary phase acting as a preferential site, which leads to the formation of lamellar structure.

2. Microstructural evolution

In this section, the evolution of microstructure composed of only Mo_3Al and Mo_3Al_8 phases will be discussed. In the eutectoid decomposition of the present alloy, an induced strain contribution and interfacial properties such as crystallographic relation at the lamellar interface become important. Fine lamellar colonies were observed in Fig. 2(b) with the faster cooling rate and partially spheroidized regions were also confirmed in Fig. 2(c) with the slower cooling rate. The microstructure also easily changes from a fine lamellar structure shown in Fig. 2(b) to a spheroidized one shown in Fig. 2(d) by an aging treatment for 21.6 ks. These morphological changes imply not only coarsening but also spheroidization of fine lamellae, and a lamellar structure of this alloy is intrinsically unstable, which leads spheroidization in the fast manner.

V. Conclusion

The formation and evolution processes of microstructure in Mo–Al–Ti alloys are investigated. Fine acicular Mo_3Al precipitates appear in the interior of a bcc grain prior to the eutectoid decomposition and their morphology suggests a crystallographic relationship is held between the matrix and precipitates. During the formation of the lamellar structure from these acicular precipitates, interfacial properties between Mo_3Al and Mo_3Al_8 may play an important role. A fine lamellar structure composed of Mo_3Al and Mo_3Al_8 is formed by a continuous cooling from the temperature above the eutectoid point. Fine lamellae undergo spheroidization under a slow cooling and during aging at 1373 K for 21.6 ks. This morphological evolution process implies the instability of lamellar structure in this alloy.

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