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Grain boundary hardening and triple junction hardening in polycrystalline molybdenum

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Abstract

The grain boundary and triple junction hardenings in molybdenum with different carbon content were studied in connection with the character and the connectivity of grain boundaries at triple junctions by the micro-indentation test. The triple junction hardening is smaller at the junctions composed of low-angle and Σ boundaries than at the junctions composed of random boundaries. This difference in the hardening depending on the grain boundary connectivity becomes more significant with a decrease in carbon content in molybdenum.

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Keywords: Molybdenum; Micro-indentation test; Grain boundary hardening; Triple junction hardening; Grain boundary character; Grain boundary connectivity

1. Introduction

Molybdenum has been used as a high temperature structural material and its use is also anticipated in the substrates of integrated circuit and micromachines. However, molybdenum often shows severe brittleness due to intergranular fracture around room temperature, which limits its practical applications [1–5].

The grain boundaries and triple junctions play important roles in the development of various properties of polycrystalline materials. For example, when polycrystalline materials are subjected to deformation, the compatibility stresses are often generated in the vicinity of grain boundaries and triple junctions, causing crack nucleation. To date, many efforts have been made concerning grain boundary hardening in connection with grain boundary character [6,7] and grain boundary segregation [6,8–12]. From these results, it is known that grain boundary hardening strongly depends on the grain boundary character. To our knowledge, however, the effect of triple junctions on hardness has scarcely been studied so far.

Triple junctions can be classified into two types, I-line and U-line, based on the O-lattice theory by Bollmann [13–15]. Palumbo and Aust [16] demonstrated the first experimental evidence that U-lines in nickel show preferential corrosion pit nucleation. Also, Randle [17] reported that U-lines are susceptible to cavitation in a superplastically deformed Al–Li alloy, whereas I-lines are more resistant to cavitation. On the other hand, Fortier et al. [18] classified the triple junctions by grain boundary connectivity. This is based on how to connect the grain boundaries with different characters at the triple junctions. We have observed the preferential sites of cavitation accompanying superplastic deformation in an Al–Li alloy, and showed that the triple junctions where

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more than two random boundaries are connected become preferential sites of cavitation [19].

The purpose of this work is to explore the relationship between the triple junction hardening and the type of triple junction in "intrinsically brittle" molybdenum with different purities. In addition, we have studied grain boundary hardening particularly taking account of the deviation from the exact coincidence misorientations.

2. Experimental procedure

Two kinds of specimen of molybdenum with different carbon content were used. The Type A specimen containing 3 ppm carbon was grown by a radio frequency floating zone method, and then uniaxially compressed and annealed at 1873 K for 7.2 ks. The Type B specimen containing 14 ppm carbon was also grown by a radio frequency floating zone method, and then hot-forged at 1593 K in hydrogen atmosphere and annealed at 1773 K for 14.4 ks. The oxygen content was 6 ppm in both types of specimen. The grain sizes of Type A and Type B specimens were about 300 and 550 μm, respectively.

The micro-indentation tests were made for grain boundaries and triple junctions whose characters were previously determined by orientation imaging microscopy (OIM) [20,21] by a Vickers micro-hardness tester (Akashi MVK-E) with a rotation specimen stage. The indentation tests were performed at a small force of 0.098 N and a loading time of 15 s. The degrees of grain boundary hardening ΔH_{gb} and of triple junction hardening ΔH_{tri} were evaluated by Eqs. (1) and (2), respectively

$$\Delta H_{\mathrm{gb}} = \left(H_{\mathrm{gb}} - \frac{H_{\mathrm{G1}} + H_{\mathrm{G2}}}{2}\right) \Big/ \left(\frac{H_{\mathrm{G1}} + H_{\mathrm{G2}}}{2}\right), \tag{1}$$

$$\Delta H_{\mathrm{tri}} = \left(H_{\mathrm{tri}} - \frac{H_{\mathrm{G1}} + H_{\mathrm{G2}} + H_{\mathrm{G3}}}{3}\right) \Big/ \left(\frac{H_{\mathrm{G1}} + H_{\mathrm{G2}} + H_{\mathrm{G3}}}{3}\right), \tag{2}$$

where H_{gb} is the hardness on the grain boundary, H_{tri} is the hardness on the triple junction and H_{G1}, H_{G2}, H_{G3} is the hardness of the adjoining grains. The grain boundary hardening and the triple junction hardening were standardized by the hardness of the grain interior, because the hardness of the grain interior depends on the grain orientation.

Moreover, "the excess triple junction hardening ΔH_{TRI}", probably attributed to the interaction among three grain boundaries connecting at a triple junction, was evaluated by the difference between triple junction hardening and the average grain boundary hardening of three grain boundaries connecting at a triple junction as follows:

$$\Delta H_{\mathrm{TRI}} = \Delta H_{\mathrm{tri}} - \alpha\left(\frac{\Delta H_{\mathrm{gb1}} + \Delta H_{\mathrm{gb2}} + \Delta H_{\mathrm{gb3}}}{3}\right), \tag{3}$$

where α is a constant which compensates for the difference in the total length of grain boundary segments between those under an indent positioned at the center of triple junction and those under the indent aligned along a single grain boundary. The value α was estimated to be 1.2–1.3 in the experiments in which the grain boundary hardening was measured.

In this work, two opposite corners of the indentation were aligned along the grain boundary in order to obtain the higher possible grain boundary hardness. On the other hand, in the case of triple junction hardness, the center of the indentation was located at the triple junction. For the measurements of the grain interior hardness, the indentation was located about 80 μm away from the grain boundary in order to exclude grain boundary effect. Fig. 1 shows indentations on grain boundaries and a triple junction, indicating that indentations were accurately made on the grain boundaries and the triple junction.

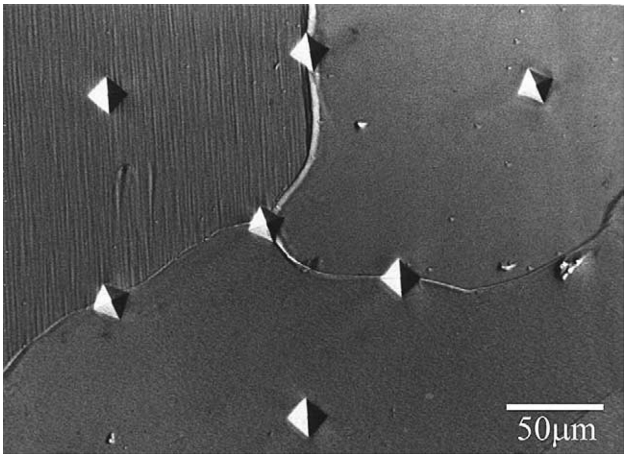

Fig. 1. Vickers indentations on grain boundaries and a triple junction.

The deformation dislocation structure produced by indentation on the grain boundary was observed by using a transmission electron microscope (TEM, JEOL JEM2000CX). In preparing the TEM thin foils, the indented surface was protected by covering with aluminum foil during thinning by electrolytical polishing in order to prevent corrosion from the indentations.

3. Results and discussion

3.1. Effect of grain boundary character on hardness change across grain boundary

Figs. 2(a)–(e) show hardness–distance profiles for grain boundaries with different characters in the Type A specimen containing 3 ppm carbon. The hardness increases on approaching a grain boundary and shows a peak at the grain boundary irrespective of grain bound-

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