Effects of Sintering Conditions on the Properties of Sintered Molybdenum

Takanori Kadokura[1,a], Yutaka Hiraoka[2,b], Seiji Nakabayashi[1,c] and Yoshiharu Yamamoto[1,d]

[1] Material Research Dept., A.L.M.T.Corp., 2 Iwasekoshi-machi, Toyama-shi 931-8543, Japan
[2] Faculty of Science, Okayama University of Science, 1-1 Ridai-cho, Okayama-shi 700-0005, Japan
[a]takanori-kadokura@allied-material.co.jp, [b]hiraoka@dap.ous.ac.jp,
[c]seiji-nakabayashi@allied-material.co.jp, [d]yoshiharu-yamamoto@allied-material.co.jp

Abstract

Effects of sintering conditions such as sintering temperature and heating rate on oxygen content, density, microstructure and toughness of sintered Mo were investigated. The oxygen content of the sintered Mo significantly depended on the sintering conditions. The oxygen content of the primary sintered(below 1673 K) Mo influenced the densifications. The number of pores at grain boundaries of the secondary sintered(at 2073 K)Mo depended on the oxygen content of the primary sintered Mo. Grain growth of the secondary sintered Mo was inhibited by the existence of pores at the grain boundaries. The secondary sintered Mo having larger number of pore and smaller grain size demonstrated higher strength.

Keywords : molybdenum, sintering, oxygen content, microstructure, toughness

1. Introduction

Molybdenum (Mo) is a suitable material for high-temperature structural applications because of its high melting point (2890K), high thermal conductivity, low thermal expansion coefficient, ease of fabrication and excellent compatibility with liquid oxide.

In general, Mo product is manufactured by powder-metallurgy. In order to improve properties such as heat-resistance and strength of Mo product, it is important to clarify the mechanism of sintering process. Therefore, there are many reports on the Mo sintering behavior from viewpoints of sintering conditions such as dew point, sintering atmosphere and particle size [1-3].

In the present study, press body of Mo powder weresintered at various sintering temperature and heating rate, and the effects of sintering conditions on properties such as oxygen content, density, microstructure and toughness of the sintered Mo were investigated.

2. Experimental and Results

Mo powder of 3.8 μm that contains about 1000 mass-ppm oxygen was pressed by cold isostatic pressing. From the pressed body, specimens with dimensions of 40 x 40 x 20 mm were cut out. Then the specimens were sintered at a sintering temperature between 773 and2173 K with heating rate between 2 and 20 K/min in hydrogen atmosphere.

Properties of the sintered Mo were evaluated as follows.The density was determined by the Archimedes measurement, but some sintered Mo with low density were evaluated by the size and weight measurement. The oxygen content was analyzed by LECO TC600. Microstructure was observed by an optical microscope and average grain size was measured by the Full-man method [4].

Three-point bending tests were carried out at a temperature between 173 and 473 K at a crosshead speed of 0.0083 mm/s (initial strain rate is $1.95\mathrm{x}10^{-4}\ \mathrm{s}^{-1}$). The specimen dimensions were 3.5 mm wide, 25 mm long and 1.0 mm thick. Recrystallized rolled pure Mo sheet (designated as Mo(R)) was also used for comparison. From temperature dependence of the yield (σ_y) and the maximum (σ_m) strengths, two parameters, critical stress (σ_c) and critical temperature (T_c) were determined [5]. σ_c is the stress at T_c (DBTT) where σ_y is equal to σ_m. The fracture surface of the specimens which failed below T_c were examined by a scanning electron microscope and the percentage of the intergranular fracture area to total fracture area (PIF) was evaluated.

Figure 1. shows the effect of sintering temperature (heating rate is 2 K/min) on oxygen content and relative density. The oxygen content passed through three stages and decreased to a level as low as 30 mass-ppm. Distinct

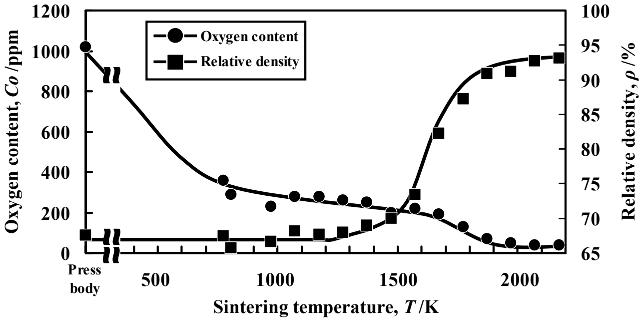

Fig. 1. Effect of sintering temperature on oxygen content and relative density.

densification occurred at a temperature above 1673 K. In this study, primary sintered Mo at a temperature below 1673 K was designated as Mo(P). The oxygen content of Mo(P) depended on the heating rate. The faster the heating rate was, the lower the oxygen content was.

Oxygen content of Mo(P) influenced the densification. Mo(P) with lower oxygen content demonstrated higher density after secondary sintering at 2073K. In this study the secondary sintered Mo at 2073 K was designated as Mo(S).

In Mo(S), there were two kinds of pores that exist at grain boundaries and inside the grain. Number of pores at grain boundaries depended on the oxygen content of Mo(P), whilst size of pores were almost the same regardless of the oxygen content. There was a linear relation between number of pores at the grain boundaries and grain size of Mo(S). This results suggest that grain growth of Mo(S) is inhibited by the pores relating to the oxygen content of Mo(P).

Figure 2. shows the effect of grain size on σ_c of Mo(S). The specimen with a smaller grain size exhibited a higher σ_c according to the Hall-Petch relationship, in spite of lower density and more pores. It is noted that σ_c of Mo(R) with almost the same grain size was higher by about 170 MPa than that of Mo(S). Grain size dependence of strength of Mo materials without large pores was already reported [6,7]. Mo(S) with pores demonstrated a lower strength and a less significant grain size dependence than Mo materials without pores. This result seems to be attributed to the weakness of grain boundaries by a number of pores.

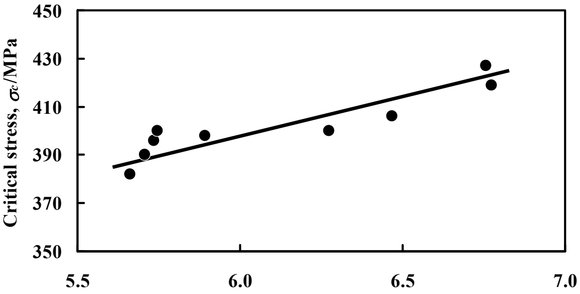

Fig. 2. Effect of grain size on critical stress of Mo(S).

T_c of Mo(S) was about 293 K regardless of the grain size, and was much higher than that of Mo(R) (208 K).

Figure 3. shows typical fractography of Mo(S) and Mo(R). Fracture mode of Mo(S) was primarily the intergranular fracture due to weakness of the grain boundaries, and PIF was about 98 %. On the other hand, PIF of Mo(R) was about 80 %.

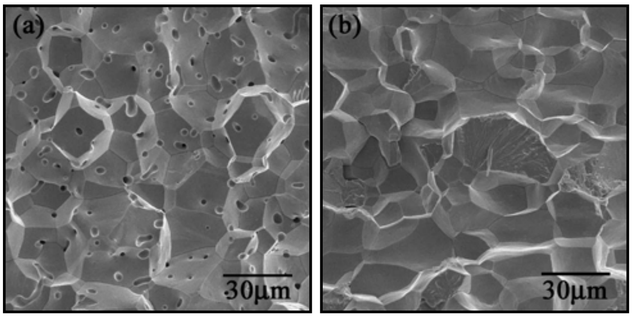

Fig. 3. Typical fractography of Mo(S)(a) and Mo(R)(b).

3. Summary

(1) Oxygen content of the sintered Mo significantly depended on the sintering temperature and the heating rate.
(2) Oxygen content of Mo(P) influenced the densification and the number of pores of Mo(S).
(3) Grain growth of Mo(S) was inhibited by pores at the grain boundaries.
(4) Mo(S) with a larger number of pores and/or with a smaller grain size demonstrated a higher strength, whilst its ductility was almost equivalent.

4. References

1. C.Stallybrass, G.Leichtfried and A.Kneissl, Proc. of the 15th International Plansee Seminar'01, eds. G.Kneringer, P.Rodhammer and H.Wilder, Plansee Holding AG, Reutte, Vol.1(2001), p.267.
2. H.S.Huang and K.S.HWANG, Metall. Trans. A, 33A (2002), p.657.
3. D.Uskokovic, B.Novakovic, V.Petrovic and M.M.Ristic, Proc. of the 5th Int. Round Table Conference on Sintering, Material Science Monographs, 14(1981), p.69.
4. **Jounal of Metals, 3(1953), p.447.**
5. Y.Hiraoka, Mater.Trans., JIM, 31(1990), p.861.
6. Y.Hiraoka, M.Okada and H.Irie, Quart. J. Jpn. Weld. Soc., 5(1987), p.522.
7. Y.Hiraoka, M.Okada and H.Irie, J. Nucl. Mater., 155-57 (1988), p.381.