High Temperature Deformation Behavior of Nano Grain W Produced by SPD-PM Process

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Abstract

In this study, nano grain W is fabricated by Severe Plastic Deformation-Powder Metallurgy (SPD-PM) process. W powder and W-Re powder mixtures are processed by SPD-PM process, a Mechanical Milling (MM) process. As results, a nano grain structure, whose grain size is approximately 20nm, is obtained in W powder after MM for 360ks. A nano grain W compact, whose grain size 630nm, has excellent deformability above 1273K. A nano grain W-10Re compact is composed of equiaxed grain, a grain growth is restrained and has low dislocation density after the large deformation; therefore it is considered that W-Re compact shows superplasticity.

Keywords : tungsten, rhenium, nano grain structure, superplasticity, mechanical milling.

1. Introduction

Tungsten (W) is a useful material because of its high performances such as high melting temperature (T_m: 3695 K) and high strength (Young's modulus: 403 GPa). However, its high melting temperature requires high temperature for deforming; therefore an improvement of deformability is important for W. Various methods are used to improve the deformability in W, and one of useful methods is grain refinement. Grain refinement improves strength, sinterability, and high temperature deformability[(1)].

Moreover, the nano grain materials show excellent superplasticity. It is considered that superplasticity enables to notably improve deformability in W. In the present study, the nano grain W and W-Re were prepared by Mechanical Milling (MM) process, whose is one of Severe Plastic Deformability-Powder Metallurgy (SPD-PM) processes. In addition, our previous papers have shown that Re solid solution is able to retard the grain growth by segregation of Re at the grain boundaries in the nano grain W[(1, 2)].

Objective of the present study is to fabricate the nano grain W and clarify the high temperature deformation behavior of W and W-Re compacts with the nano grain.

2. Experimental Procedures

W (purity 99.9mass %) and Re (purity 99.9mass %) powders were blend to the composition of W, W-3mass% Re (W-3Re) and W-10mass%Re (W-10Re), and sealed into a vial under an Ar atmosphere. MM was carried out for 360ks by Fritsch P-5 planetary ball mill using a SKD11 vial and SUJ2 balls. The vial and balls were coated by W prior to the milling to avoid contamination.

MM powders were sintered by Spark Plasma Sintering (SPS) system, and the high temperature deformability of sintering compacts was evaluated. Deformability was also examined at a constant load by using the SPS. Columnar specimens of ϕ 4 mm $\times$ 6.4 mm were cut out from these sintering compacts, and examined under the following conditions; load: 200MPa, holding time: 1.8ks and heating rate: 1.67 Ks^{-1}. The sample was heated by ohmic heat, and the temperature was measured by the radiation thermometer.

The strain-rate sensitivity, m-value, was obtained by Precision Universal Tester (Shimadzu AG-5kNE) by a constant strain rate test. The size of specimens was the same as stated above, and test conditions are the following; initial strain rate: between 2.6×10^{-5}～7.7×10^{-4}, compression temperature: 1473 K, heating rate: 0.5 Ks^{-1}, atmosphere: Ar gas. The sample was heated by infrared image furnace, and the temperature was measured by thermocouple.

Powders and compacts were characterized by means of XRD, SEM, and TEM/ EDS.

3. Results and discussion

Fig.1 shows TEM micrograph and Selected Area Diffraction Pattern (SADP) of W/MM 360ks powders. W/MM powder is the equiaxed nano grains, whose grain

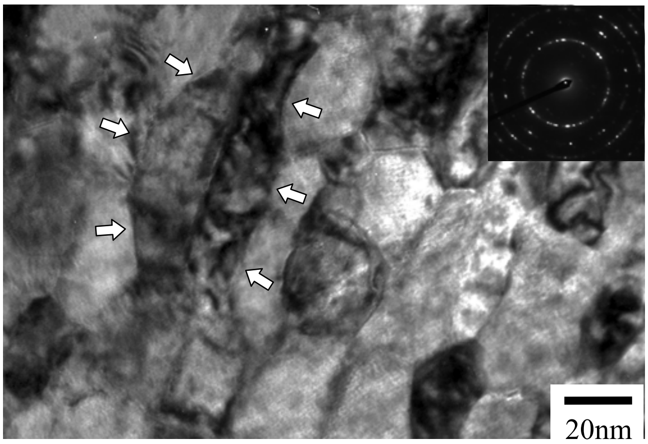

Fig. 1. TEM and SADP images of W/MM 360ks powder.

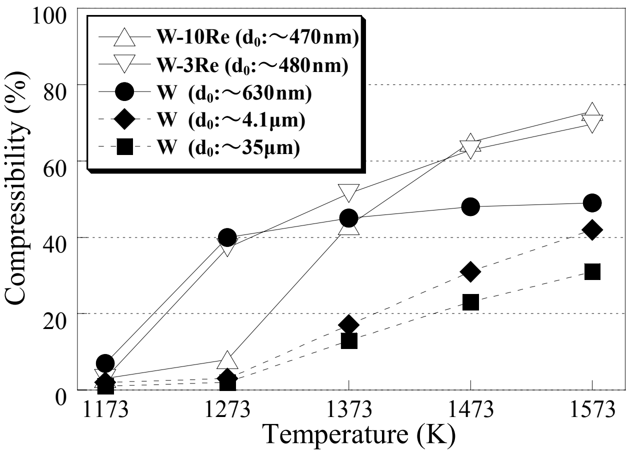

Fig. 2. Relationship between compressibility and tem peratures in W and W-Re sintering compacts.

size approximately 20 nm. Although there are elongated nano grains in part, as can be seen by white arrow in Fig.1, these elongated nano grains are divided into two or more nano subgrains by strain contrast. The sizes of these nano subgrains are similar to those of the equiaxed nano grains, so it is considered that the equiaxed nano grains are formed by a division of elongated nano grain. SADP of the nano grain structure shows ring profile, which implies these nano grains have high angle boundary.

Fig.2 shows change of the sintering compacts height, i.e., compressibility, at various compression temperatures. Compared with to W compacts with coarse grain size, the nano grain W compact well deformed above 1273K ($0.34T_m$). This result indicates that the high temperature deformability of W is improved by grain refinement. Deformabilitys in the W-3Re and W-10Re compacts are highly improved up to over 60% at 1473 K ($0.40T_m$). Re addition into W controls the grain growth of nano grain W alloy[1], therefore, it is considered that this grain growth control improved the deformability in W-Re compacts.

Fig.3 shows that compression test at various strain rates was performed to obtain m-value of W-10Re compact at 1473 K. m-value of 0.41 is obtained and this value satisfied

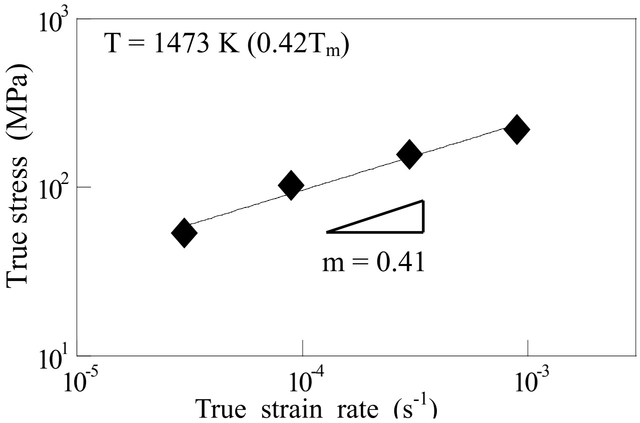

Fig. 3. Relationship between true stress and true strain rate at 1473 K.

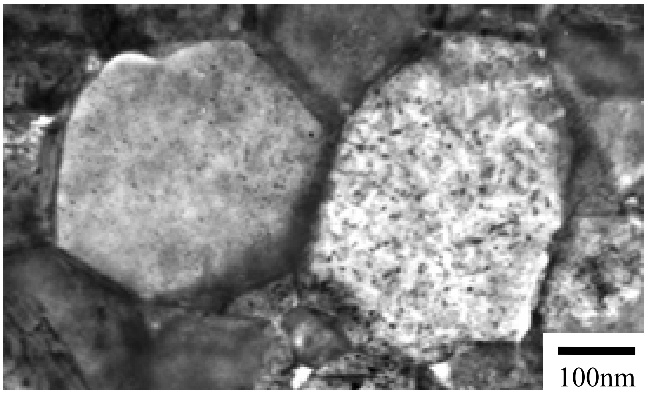

Fig. 4. TEM micrograph of W-10Re compact compressed at 30% at 1473K initial strain rate of 7.7 $\times 10^{-3}s^{-1}$.

the general requirement for superplasticity, namely m > 0.3. Fig.4 shows TEM micrograph of the W-10Re compact after compressed at 30% at 1473 K at initial strain rate of $7.7\times 10^{-3}s^{-1}$. After compression test, W-10Re compact has an equiaxed nano grain structure with no dislocations. These results, as shown in Figs.3 and 4, suggest that the W-10Re compact shows superplasticity at 1473 K, which is 0.42T of solidus temperature. Already reported superplasticity temperature of tungsten is around $0.54T_m$[3]. Therefore, such an extremely low superplastic temperature in this study must be due to the grain boundary sliding of the nano grain structure as well as the enhanced thermal stability by solid solution of Re.

4. References

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