THERMAL CHANGE OF MICROSTRUCTURE AND MECHANICAL PROPERTIES OF DISPERSION STRENGTHENED TUNGSTEN

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Abstract

Using a Mechanical Alloying (MA)-Hot Isostatic Pressing (HIP) method, new dispersion strengthened tungsten (DS-W) alloys containing TiO2 particles on grain boundaries have been developed for improvement of Plasma Facing Materials (PFM) for divertor components of fusion reactors. In this study, effects of the dispersion strengthening on the thermal change of microstructure and the thermal conductivity at high temperature were investigated. Within contrast to Pure Tungsten, the DS-W did not show significant microstructural change or a drastic decrease of the bending strength after annealing at high temperature.

1. INTRODUCTION

In the designs of the tokamak DEMO and the helical reactor (FFHR-d1), tungsten (W), having high thermal conductivity, high melting point and low tritium retention, plays a key role as Plasma Facing Material (PFM) of divertor components [1, 2]. W is known to become brittle by recrystallization when subjected to high heat loading. In case of design of the helical divertor, it is expected that surface temperature would achieved 1600 °C on heat input of 30MW/m^2. [3] The recrystallization embrittlement, degrading the mechanical property, is considered to be a critical issue necessary to be resolved for the divertor components development [4]. As a solution of the problem, we have developed an Oxide Dispersion Strengthened Tungsten (ODS-W) which can avoid embrittlement even after recrystallization [5]. The idea is an application of Carbide Dispersion Strengthened Tungsten (W-TiC alloy) developed by Dr. Kurishita [6]. The present ODS-W have been dispersion-strengthened by the TiO_2 particles which are known to be stable relative to the TiC carbide particles at high temperature [7]. The TiO_2 particles were formed because the TiC added initially reacted with impurity oxygen in tungsten matrix during the succeeding process. As the starting study of the ODS-W, effect of the diameter of Mechanical Alloying (MA) balls on the mechanical properties after Hot Isostatic Pressing (HIP) treatment have been investigated. The MA with 3.0 mm balls resulted in a rapid progressing of MA relative to that of 1.6 mm balls, fine dispersion of TiO_2 particles, and improvement of mechanical properties after HIP treatment [8]. In this study, effects of the post-HIP heat treatment on microstructure and mechanical properties were investigated in comparison with pure tungsten (ITER-Pure W), focusing the effect of dispersion strengthening particles on thermal change.

2. EXPERIMENTAL PROCEDURE

In the preparation of Dispersion Strengthened tungsten (DS-W) alloys, the initial materials used were powders of pure-tungsten and titanium carbide supplied by New Metals and Chemicals Co. LTD. and JAPAN NEW METALS Co. LTD., respectively. The data of the powders supplied by the vendors are listed in Table 1.

TABLE 1. The data of the powders

Materials	Purity (%)	Particle size (μm)
Tungsten	99.95	1.0
Titanium Carbide	98.00	1.8

The initial materials were mixed and mechanically alloyed in a planetary-type ball mill using tungsten carbide MA balls of 1.6 mm and 3.0 mm in a 250 cc tungsten carbide MA pot with ball-powder ratio of 2:1. The alloying was carried out with a rotating rate of 360 rpm for 64 hrs in a pure argon (below 1 ppm) gas atmosphere. The rotation was repeated by an automated program (20 min milling + 3 hr pause) to avoid excess temperature increase of the pot. The mechanically alloyed powders were compressed by cold isostatic pressing (CIP), and were then pre-sintered in hydrogen gas atmosphere at high temperature (Preliminary sintering). The pre-sintered W alloys were then sintered by HIP at 1750 °C for 1.5 hr with a pressure of 186 MPa. For comparison, a pure tungsten supplied by the A.L.M.T. Corp. hereafter named ITER-Pure W, was prepared. The HIPed DS-W with 1.6 mm and 3.0 mm MA balls and ITER-Pure W were annealed at 1600-1800 °C for 1.5 hr in vacuum. The annealed three tungsten materials were characterized by four-point bending tests. The microstructures were also characterized by Electron Back Scatter Diffractometer (EBSD), Transmission Electron Microscope (TEM) and Scanning Electron Microscope (SEM). The thermal conductivity of the materials was analyzed by Flash method. HIPed pure W fabricated by same HIP process as that of DS-Ws were prepared [9].

3. RESULTS

Fig.1 shows EBSD map images before and after annealing. The ITER-Pure W and the DS-W before annealing exhibited the texture of fine grains by rolling and the equiaxed-fine grains of non-texture by HIP treatment, respectively. As seen in EBSD color map of the ITER-Pure W and the DS-W before annealing, misorientation of the crystal grains of DS-W before annealing is larger than that of ITER-Pure W, indicating that the grain boundary is close to the recrystallized state. Grains of ITER-Pure W after annealing at 1600, 1800 °C coarsened drastically, indicating completion of the secondary recrystallization. The grains of DS-W with 1.6 mm MA balls after annealing at 1600 and 1800 °C were also coarsened significantly. On the other hands, grains of DS-W with 3.0 mm MA balls after annealing at 1600 and 1800 °C showed limited coarsening.

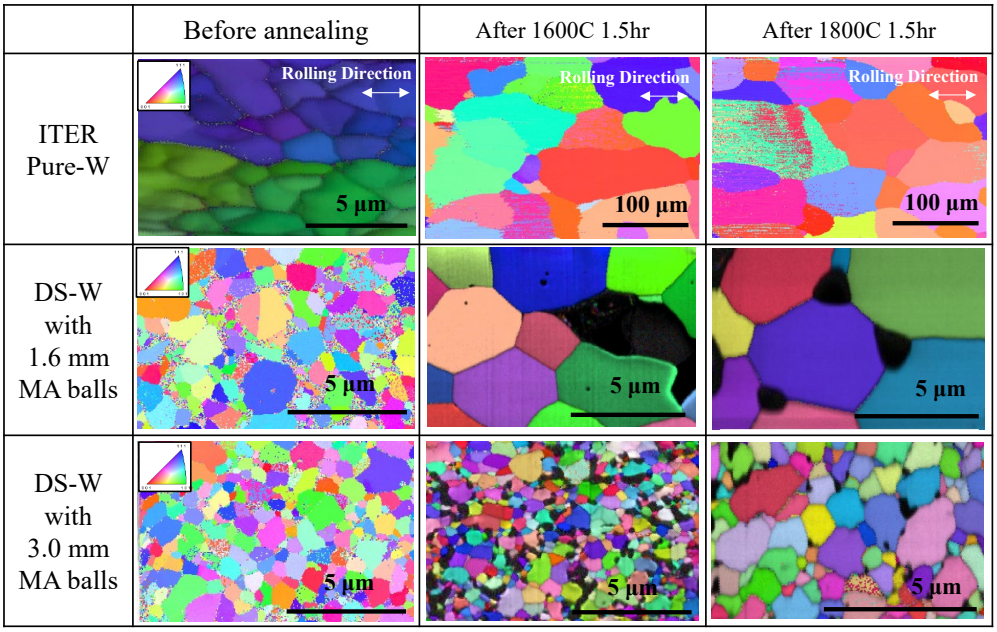

Fig.1 EBSD map images of the ITER-Pure W and the DS-Ws before and after annealing

Figu.2 shows crystal grain size and four- point bending strength of the ITER-Pure W and the DS-Ws before and after annealing at 1600, 1800 °C. Broken lines in the figure show the grain sizes before and after annealing. The grains of ITER-Pure W after annealing at 1800 °C was 31 times as large as that before annealing. On the

other hands, grain size of DS-W with 3.0 mm MA balls was maintained after annealing at 1600, 1800 °C in contrast to significant coarsening for DS-W with 1.6 mm MA balls. As seen in the previous study [7], TiO_2 particles in DS-W with 3.0 mm MA balls were more finely dispersed that those in DS-W with 1.6 mm MA balls before the annealing. The present result suggested that the dispersed particles of TiO_2 is stable to 1800 °C , maintaining the pinning effect on grain boundaries [8]. As a reference of the similar effect in previous research, the suppressing effect of K-doping on grain growth at high temperature was reported [10]. These results of indicates that both of bubble dispersion strengthening by potassium and oxide dispersion strengthening by TiO2 can suppress the grain coarsening of secondary recrystallization.

Solid lines in Fig.2 show the fracture strength derived by four-point bending tests before and after annealing. Fracture strength of ITER-Pure W decreased drastically with grain coarsening after annealing. The fracture strength of ITER-Pure W after annealing is almost same as that of DS-W with 1.6 mm MA balls. This is interpreted as the result of the fact that The DS-W with 1.6 mm MA balls did not have pinning effect due to insufficient dispersion of TiO_2.On the other hands, the fracture strength of DS-W with 3.0 mm MA balls after annealing decreased slightly, but was still higher than that of ITER-PURE W and DS-W with 1.6 mm MA balls.

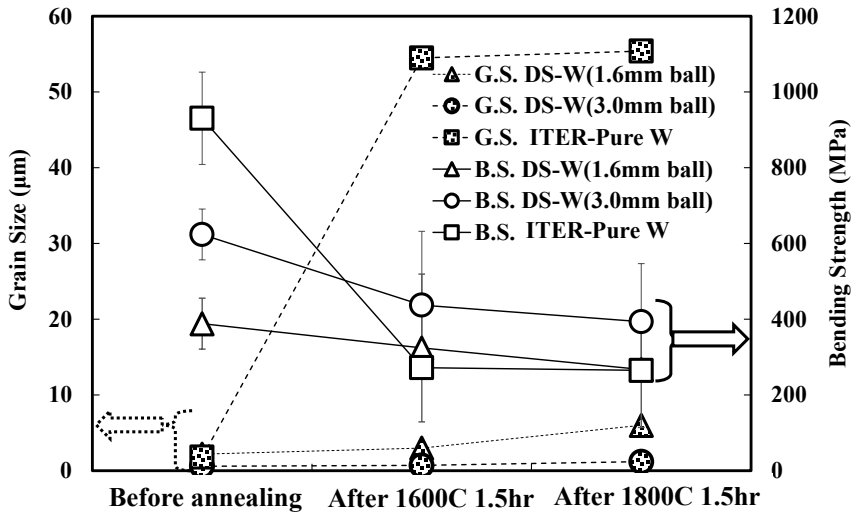

Fig.2 Grain size and bending strength of the ITER-Pure W and the DS-Ws before and after annealing

Fig.3 shows the fracture surfaces of four-point bending test samples after annealing at 1800 °C for 1.5 hr. As shown in Fig.3-(a), the fracture surface of DS-W with 1.6 mm MA balls exhibits large voids in grain boundaries. On the other hands, the fracture surface of DS-W with 3.0 mm MA balls shown in Fig.3-(b) exhibited dense microstructure, in agreement with the higher strength of DS-W with 3.0 mm MA balls than that with 1.6 mm MA balls.

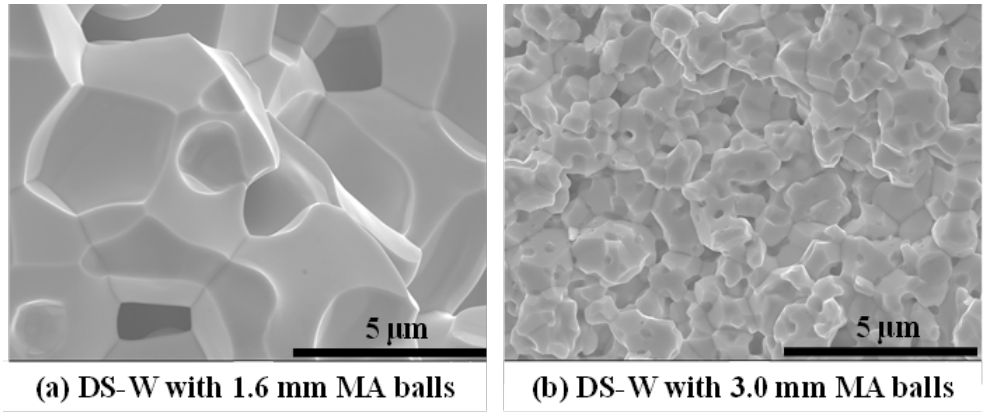

Fig.3 Fracture surfaces of four-point bending test samples after annealing at 1800 °C for 1.5 hr.

Fig.4 shows the thermal conductivity of DS-W with 1.6 and 3.0 mm MA balls, and the HIPed-pure tungsten from room temperature to 1250 °C. The thermal conductivities of HIPed-pure tungsten and DS-W with 1.6 mm MA balls significantly decreased from room temperature to 1250 °C. On the other hands, the thermal conductivities of DS-W with 3.0 mm MA balls decreased from room temperature to 1250 °C only slightly, indicating effect of different heat diffusion path in contrast to the HIPed-pure tungsten and the DS-W with 1.6 mm.

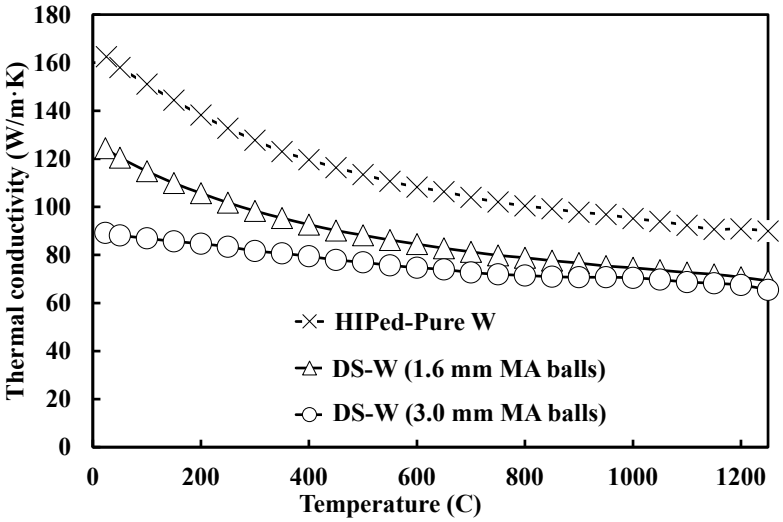

Fig.4 Thermal conductivity of DS-W with 1.6, 3.0 mm MA balls and HIPed-pure tungsten from room temperature to 1250 °C

4. DISCUSSIONS

Fig.4 shows that the thermal conductivity of pure tungsten significantly decreases with temperature. However, considering the high temperature environment in which the PFM-W will be used, thermal conductivity at high temperature is more important than that at low temperature such as room temperature. Thermal conductivity of HIPed-pure W at room temperature is superior to one at high temperature. However, the difference of thermal conductivity between DS-W pure W becomes smaller at high temperature. Thus, improvement of thermal conductivity of DS-W at high temperature to some extent would enhance largely the attractiveness of the material. Table 2 shows densities of the DS-Ws with 1.6, 3.0 mm MA balls and HIPed-Pure W. The densities of DS-Ws with 1.6, 3.0 mm MA balls is lower than that of HIPed-Pure W in spite of the same HIP treatment condition. This result indicates possibilities of improvement of thermal conductivity by densification for DS-W with 3.0 mm MA balls.

TABLE 2. Densities of the DS-Ws with 1.6, 3.0 mm MA balls and the pure tungsten fabricated by HIP treatment

Materials	Density (g/cm^3)
HIPed-Pure W	18.3
DS-W with 1.6 mm MA balls	15.4
DS-W with 3.0 mm MA balls	16.6

5. CONCLUSIONS

Using a Mechanical Alloying (MA)-Hot Isostatic Pressing (HIP), we have developed a new dispersion strengthened tungsten (DS-W) in which recrystallization does not cause embrittlement. In this study, thermal

changes of microstructure and mechanical properties on the two kind of DS-Ws and the pure tungsten (ITER-Pure W and HIPed-W) after annealing was evaluated. The main results are as follows:

・Fracture strength of the ITER-Pure W decreased drastically after annealing with thermal change of microstructure from texture of fine grain to non-texture of coarsened grain.
・DS-W with 3.0 mm MA balls exhibit only a moderate decrease of fracture strength with the thermal change of microstructure after annealing. This was attributed to the effect of fine dispersion of TiO_2.
・Thermal conductivity of the DS-W with 3.0 mm MA balls decreased moderately from room temperature to 1250 °C in contrast to the significant decrease for HIPed-Pure W, suggesting that the grain boundaries are the dominant heat diffusion path for the DS-W.
・Considering low density of DS-W with 3.0 mm MA balls, it is expected that the densification by improved processes can contribute to enhancing its thermal conductivity.

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