

Formation Mechanism of Nano-Strengthening Particles in Dispersion Strengthened W-Ti Alloys*

Hiroyuki NOTO, Yoshimitsu HISHINUMA, Takeo MUROGA and Hideo BENOKI¹⁾

National Institute for Fusion Science, Toki, Gifu 509-5292, Japan

¹⁾*Nippon Tungsten Co., Ltd., Saga 841-0203, Japan*

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The realization of advanced fusion reactors rests upon improvements of plasma-facing materials for divertors. The toughness of tungsten is a critical issue for these improvements. As a solution, we developed dispersion-strengthened (DS) W-Ti alloys using a new process which is based on a combination of mechanical alloying (MA) and hot isostatic pressing. In this paper, the microstructure of new DS-W-Ti alloys and the formation mechanism of nano-strengthening particles are investigated. The produced DS-W-Ti alloys are dispersed with TiO₂. The chemical analysis shows that new precipitation processes occurred during the MA based on redox reactions between carbon and oxygen.

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Tungsten alloys are a candidate plasma-facing material for divertor components due to their high thermal conductivity, high melting point, and low tritium retention. However, W alloys possess low toughness at high temperatures, which limits their application. As a possible solution to this issue, we propose a strengthening concept based on dispersion strengthening (DS). In previous studies of dispersion-strengthened tungsten (DS-W), W-1.1wt% TiC alloys were investigated [1], which used titanium carbide to decrease the density of grain boundaries, which are known to act as a driving force of recrystallization. The W-1.1wt% TiC alloys examined in previous studies [1] exhibited high resistance to heat loading and their ductile brittle transition temperatures were below room temperature. These materials were fabricated by 3MPDA (three mutually perpendicular directions agitation) ball mill and grain boundary sliding-based microstructural modification treatment. However, this process has some critical issues such as its complexity and the limited volume of the products. Therefore, we adopted a new fabrication process based on mechanical alloying (MA) and hot isostatic pressing (HIP) methods, which is applied to oxide dispersion strengthened ferritic steels, for the production of new DS-W alloys. In this study, the dispersed particles are investigated, and the formation mechanism of the dispersed particles is discussed.

In the production of our DS-W-Ti alloys, W and TiC were mixed and mechanically alloyed in a planetary-type ball mill using tungsten carbide balls of 1.6 mm Φ and

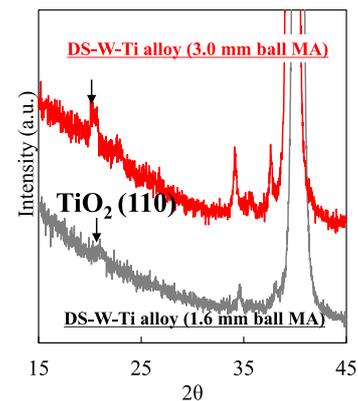


Fig. 1 X-ray spectra of DS-W-Ti alloy alloys made with 1.6- and 3.0-mm MA balls.

3.0 mm Φ in a 250-cc MA pot with a ball-powder ratio of 2:1. Alloying was carried out with a rotating rate of 360 rpm for 64 hr in a pure argon gas atmosphere. The mechanically alloyed powders were compressed by cold isostatic pressing, and were then presintered in a hydrogen-gas atmosphere at high temperature. The presintered DS-W-Ti alloys were then sintered by HIP at 1750°C for 1.5 hr under a pressure of 186 MPa. The DS-W-Ti alloys were then characterized by X-ray diffractometry, chemical analysis, transmission electron microscopy (TEM), and energy dispersive X-ray spectroscopy (EDS).

Figure 1 shows X-ray diffraction spectra of the DS-W-Ti alloys. It can be seen that titanium oxides formed in the W matrix. As a reason for the formation of titanium oxides, it is expected that the titanium oxide which has a

author's e-mail: noto.hiroyuki@nifs.ac.jp

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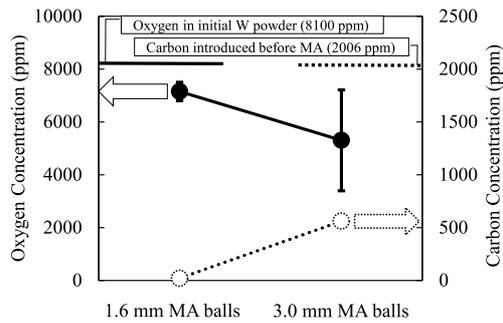


Fig. 2 Chemical analysis of the HIPed-W alloys with 1.6- and 3.0-mm MA balls, initial tungsten powder and carbon before MA.

lower formation energy than titanium carbide precipitated preferentially during the sintering [2].

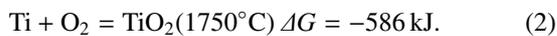
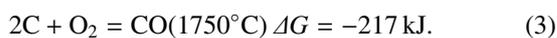


Figure 2 shows the results of our chemical analysis of the HIPed-W alloys formed via the 1.6 and 3.0-mm MA balls. The oxygen concentration of the HIPed-W alloys made using the 1.6-mm MA balls was lower than that of the initial pure W (8100 ppm). The carbon concentration of the same HIPed-W alloys was almost zero, although 2006 ppm of carbon was introduced before MA. The decrease in the oxygen and carbon concentrations implies the redox reaction in the MA process for which carbon oxide has a lower formation energy than titanium carbide.



Considering the Gibbs free energy of formation values shown in Eqs. (1 - 3), it is seen that TiC easily reduced to Ti. It is predicted that the TiO_2 arose because the oxygen in matrix combined with the pure Ti. The oxygen concentration of the HIPed-W alloys made with the 3.0-mm MA balls was lower than that made with the 1.6-mm MA balls, while the carbon concentration of former alloys was higher than that of the latter.

It was shown that MA processing using 3.0-mm balls promoted alloying more than 1.6-mm balls in a previous study [3]. The promoted alloying occurred due to heavy contact between the powders and the WC balls. Thus, it is suggested that the MA processing introduced more carbon from the larger 3.0-mm WC balls to the powders than when using the smaller 1.6-mm balls. Such contamination of carbon by the MA process when using the 3.0-mm balls has the potential to promote redox reactions between carbon and oxygen. In general, gettering processes that

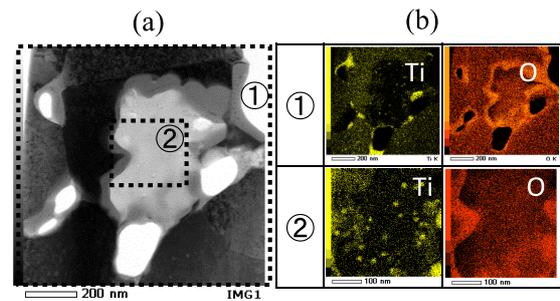


Fig. 3 TEM-EDS analysis of the W alloy made with 3.0-mm MA balls. (a) TEM image of the grain boundary ① and the grain interior ②, (b) The EDS analysis.

use matrix impurities are known as scavenging processes, which have been applied as a strengthening technique for vanadium alloys [4]. The redox reactions seen in this study are attributed to scavenging by the impurity carbon.

Figure 3 shows TEM-EDS analysis of the W alloy made with the 3.0-mm MA balls. It is seen that TiO_2 precipitated into the grain boundaries significantly and a pure Ti phase also formed in the grain's interior. Considering that TiC was added initially, the pure titanium phase was likely a residue from the reduction of TiC to pure Ti.

In this work we successfully produced a DS-W alloy with Ti using a new process that combined MA and HIP. In this study, the formation mechanism of the strengthening particles was investigated. The main results are as follows:

1. Formation of TiO_2 particles in the matrix through the MA and HIP processes was confirmed. This result implies a different reaction mechanism to that suggested in a previous study on W-1.1wt% TiC alloys [1].
2. The formation of TiO_2 was caused by a redox reaction between excess oxygen in the W matrix and carbon material in the MA mill balls.
3. TiO_2 and pure Ti phases were observed in grain boundaries and the grain's interior, respectively. The pure Ti phase is thought to have formed due to the reduction, the residue of which was not oxidized to TiO_2 .

In future work, we plan to introduce a new MA process that includes increased MA times and additional MA ball diameters. It is expected that the inhomogeneity of the microstructure and the agglomeration of dispersed particles will be suppressed by this procedure.

- [1] H. Kurishita *et al.*, *Materials Transactions* **54**, No.4, 456 (2013).
- [2] The Japan Institute of Metals, *Physical Chemistry of Metals*, ISBN4-88903-011-5.
- [3] H. Noto *et al.*, *PRICM-10 proceedings* (2019) pp.158-162.
- [4] M. Koyama *et al.*, *J. Nucl. Mater.* **329-333**, 442 (2004).