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Mechanical properties and thermal stability of pure W and W-0.5 wt%ZrC alloy manufactured with the same technology

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ABSTRACT

The mechanical properties, microstructure and thermal stability of hot rolled pure W and W-0.5 wt%ZrC alloy manufactured using the same technology are systematically studied. The results of Vickers hardness and tensile tests show that the W-0.5 wt%ZrC alloy has a higher hardness, ultimate tensile strength, ductility and thermal stability compared to pure W. The recrystallization temperature of W-0.5 wt%ZrC alloy is about 1300 °C which is 100 °C higher than that of hot rolled pure W (1200 °C). The ductile to brittle transition temperature (DBTT) of W-0.5 wt%ZrC alloy is 50 °C lower than that of the rolled pure tungsten. The above results indicate that at the fully same fabrication technology, the trace ZrC does improve the mechanical properties and thermal stability of W. The possible micro-mechanisms were investigated and proposed.

1. Introduction

Tungsten and tungsten based materials are considered as top candidates for various fusion applications, solid targets in spallation neutron sources as well as critical components in rockets and missiles due to their supreme high temperature properties. Such properties include high melting point, high creep resistance, high temperature strength, good thermal conductivity, low sputtering yield, low vapor pressure and good erosion resistance [1–3]. However, in contrast to these excellent high-temperature properties, the main disadvantage of the proposed material is its brittleness including low-temperature brittleness and irradiation and recrystallization induced brittleness. In addition, the high operation temperatures (~ 1200 °C) in fusion applications during service can alter the microstructure of pure tungsten by recovery, recrystallization and grain growth [4,5]. Thus, problems might arise during application because the ductile to-brittle transition temperature (DBTT) can be rather high. This is clearly seen in the results of Charpy tests performed on pure recrystallized W plate material with the crack orientated perpendicular to the rolling direction, where the DBTT goes up to 1000 °C and beyond. Therefore, the degradation of mechanical properties during the high work temperature should be solved by increasing the stability of microstructures as well as inhibiting recrystallization [6–9].

In recent studies, toughness improvement via manufacturing nanostructured tungsten materials has been attracting lots of attention. These process routes rely on producing precursor powders within nano-sized oxide dispersions by a wet chemical method and then consolidation [9,10]. The addition of nano-sized oxide such as La_2O_3, Y_2O_3 processed by powder metallurgy methods can effectively inhibit the grain growth and thus reduce the grain size [11–14]. However, the coarsening of dispersoids to the micron scale after high temperature sintering would on the other result in mechanical performance degradation, because these coarse dispersoids act as sites for crack initiation by stress concentrations [15]. In addition, the toughness of tungsten is also strongly sensitive to the concentration of interstitial gaseous impurities such as O, C, N, etc., which tend to segregate along the grain boundaries (GBs), weaken the GBs, and ultimately lead to inter-granular failure under a tensile load. Therefore, compared with oxide dispersion, carbides not only could inhibit the grain growth and reduce the grain size but also could diminish free O impurities through bonding with O to form stable compounds as dispersions to strengthen GBs [16–19]. It was reported that carbide dispersion strengthened W alloys may reduce the DBTT to about 200 °C, and raise their crystallization temperature to 1600 °C [17].

It is well known that properties of tungsten materials vary greatly from one product to another, depending on different production route. For example, hot-rolling or hot forging can generate very different microstructure and lead to different performances even for the same kind of materials. Even for the hot-rolling technology, the different rolling reductions, rolling pass and rolling temperature also determine

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Table 1
Chemical composition of the as-received tungsten powders (wt%).

	Cr	Ti	Fe	O	C	P	S	N	W
W powder	0.0005	0.0005	0.001	0.24	0.0029	0.0005	0.0005	–	Bal

different properties. In our previous work, the rolled W-0.5 wt%ZrC alloy with superior properties was fabricated and exhibits superior properties [17,20–23]. However, it is not fully certain that the superior performance is determined by trace ZrC addition or fabrication technology. Therefore, in order to clarify this issue and further investigate the effects of trace ZrC on the properties of W alloys, an overall study for the properties discrepancy between pure tungsten and W-0.5 wt% ZrC alloys should be discussed under the same work process.

In this work, we manufactured 6 mm thick pure W and W-0.5 wt% ZrC plates by the completely same technology. The mechanical properties and microstructure of W and W-0.5 wt%ZrC plates were investigated and compared. The tensile tests, Vickers hardness and microstructure analysis are used to determine effects of dispersed ZrC particles on the mechanical properties and thermal stability.

2. Experimental details

2.1. Starting materials and fabrication methods

Pure tungsten samples, W-0.5 wt% ZrC alloy were fabricated from pure W powders (purity > 99.9% trace metals basis, purchased from Xiamen Tungsten Reagent Co., LTD., chemical content listed in Table 1) and nano-sized ZrC powders (average particle size 50 nm, purity > 99%), respectively. Powders were ball milled in a planetary ball mill for 4 h in argon atmosphere (oxygen (O) < 0.1 ppm, H_2O < 0.1 ppm) with ball-to-powder weight ratio of 8:1 and a rotation speed of 300 rpm. Tungsten carbide balls and containers were used to minimize the possible impurity contamination. Subsequently, the milled powders were sintered at 1800 ℃ for 20 h in vacuum to get sintered billets. Then the sintered slab was hot rolled into a plate with thickness of 6 mm (see Fig. 1(a), from original thickness of 21 mm), width of 150 mm and length of 220 mm. For both alloys, small specimens of about 2 mm (TD) × 6 mm (ND) × 8 mm (RD) and tensile sample of about 4 mm (TD) × 16 mm (RD) were cut from the plate using wire cut electrical discharge machining. RD, TD and ND are short for rolling direction, transverse direction and normal direction, respectively.

2.2. Vickers microhardness and tensile test

In order to investigate the influence of annealing on the microstructures and mechanical properties of these two W materials, specimens were annealed at temperature ranging from 1000 °C to 1600 °C, and the W-0.5 wt%ZrC alloys were annealed from 1000 °C to 1800 °C for 1 h in vacuum (10^{-4} Pa), respectively. Then the annealed samples were subjected to Vickers microhardness test at room temperature on RD-ND surface with a load of 100 g and a dwell time of 15 s. Each examined sample was indented for at least 8 times in different areas.

Dog-bone-shaped specimens with a thickness of 0.75 mm for tensile tests were cut from the rolled pure W and W-0.5 wt%ZrC plates by electrical discharge machining. The gauge length and width of the tensile samples are 16 and 4 mm, respectively (see Fig. 1(b)). The as-rolled and annealed tensile specimens were all along the rolling direction. The tensile tests were carried out using an Instron-5967 machine using a constant speed of 0.06 mm/min at various temperatures from RT to 500 °C. Three tensile specimens were tested to eliminate errors.

2.3. Microstructure analysis

The metallography of samples was obtained using an optical microscope (ZEISS-AX10) after polishing and etching (10% potassium ferricyanide with 10% sodium hydroxide aqueous solution). And then grain orientation of as-rolled and recrystallization specimens were measured by Electron Backscatter Diffraction Pattern (EBSD), mappings equipped on a Zeiss SIGMA field emission scanning electron microscope, with an acceleration voltage of 20 kV and collected using a CRYSTAL detector (Oxford Instruments, Oxfordshire, UK). Specimens for EBSD were obtained using electrolytic polishing in 5% sodium hydroxide aqueous solution at RT with a constant voltage of 11 V and a current density of 3 mA/mm. The morphology and microstructures of nano-sized dispersions was characterized by Transmission Electron Microscope (TEM, JEM-2000FX). Specimens for TEM were prepared by twin-jet in Tenuple-5. Specimens for OM, EBSD and TEM were cut from the RD-ND surface which is the most direct viewing of recrystallization compared with other surfaces (see Fig. 1(a, c)).

3. Results and discussions

3.1. Vickers hardness and metallographic analysis

The hardness results of the rolled pure W and W-0.5 wt%ZrC after annealing treatments are presented in Fig. 2. The Vickers micro-hardness of the as-rolled pure W samples is 448 HV as shown in Fig. 2(a). For the pure W, the microhardness slightly decreases to 441 HV after annealing at 1200 °C for 1 h. And a sharply decrease in the micro-hardness occurs when the annealing temperature (T_a) rises above 1200 °C. For example, the hardness of pure W is just 346 HV after annealing at 1300 °C for 1 h. This hardness decrease after annealing

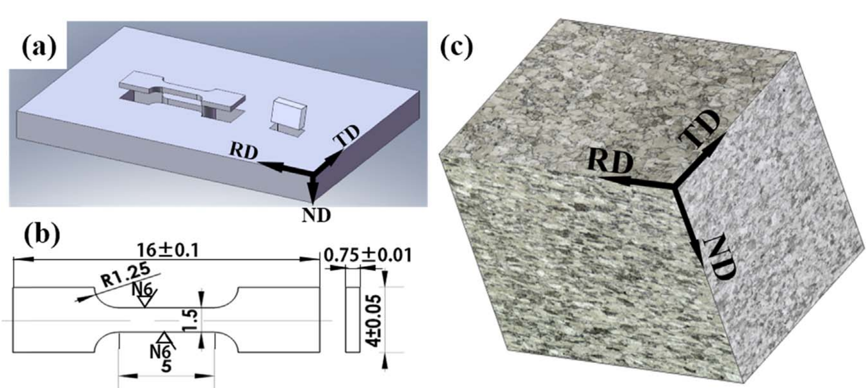

Fig. 1. Geometry and dimensions of the (a)tensile, (b)EBSD test samples, (c) optical micrographs of pure tungsten RD-ND, RD-TD, TD-ND surface.