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Preparation of TiC-doped W-Ti alloy and heat flux performance test under laser beam facility

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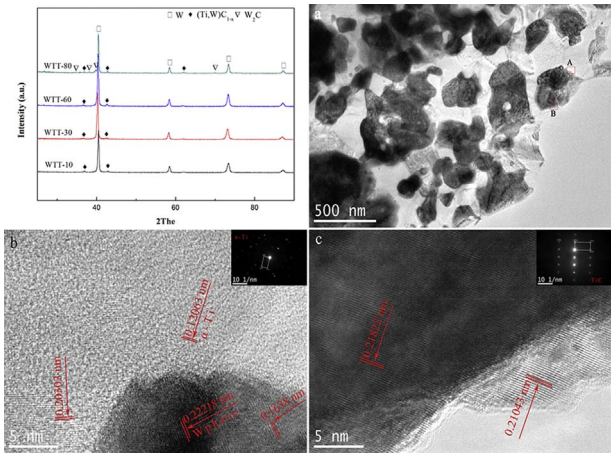
GRAPHICAL ABSTRACT



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ABSTRACT

TiC-doped W-Ti composites were prepared by mechanical alloying and spark plasma sintering. The influence of milling time on the microstructure and properties of TiC-doped W-Ti composite material was investigated. Results show that with increasing ball milling time, powder particles undergo a process of fracture-cold welding-fracture. Powder grains are continuously refined to 25.1 nm, and micro-strain continuously increases from 0.324% to 0.724%. With the extension of ball milling time, the sintered composite material exhibits a declining trend in density and an initial decrease and later increase in grain size. The minimum grain size is noted in the specimen WTT-60 at 0.35 μm. Microhardness increases first then decreases, and the microhardness of specimen WTT-60 reaches the highest value (1139.27 Hv) among those tested. Heat-shock resistance of the specimens was researched though a transient heat load experiment at pulse width 1 ms, current 60 A, and laser beam spot diameter 0.6 mm (44 ~ 47 MW/m^2).The bulk prepared by powder sintering from 60 h of ball milling presents the best thermal shock resistance.

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1. Introduction

At present, developing nuclear fusion devices has been a focus and yet faces serious challenges, particularly, in plasma facing materials (PFM) [1–3]. Tungsten, which is characterized by high melting point, high thermal conductivity, and low sputtering corrosion rate and hydrogen retention ratio, has become a main candidate first-wall material [4–9]. Meanwhile, many problems exist in the application of tungsten materials in the nuclear fusion environment.

One problem of tungsten materials is their exposure to considerable heat load conditions, including stable state (heat load up to 10 MW/m^2), slowly transient (up to 20 MW/m^2), and transient heat loads (up to 1 GW/m^2 and above) [10–15]. Except for steady heat load even in the case of relief, the edge local area model in ITER (ELMs) still bears a heat load of 0.5 MJm^{-2} for less than 1 ms. The resulting crack, surface melting, evaporation, recrystallization, grain growth, and other problems cause device damage, severely influencing the lifetime of the device [16–20].

In addition to bearing the heat load, another problem is the need of PFM to tolerate the strong effect of hydrogen isotopes, helium, and neutrons. This effect may cause vacancies, bubbles, and cracking on the material surfaces [21–25]. Moreover, PFM likely causes hydrogen retention within a certain temperature range [26–28].

Many studies have shown that alloying tungsten, nano-structure tungsten or dispersing strengthening tungsten matrix using carbide oxide is an effective means for improving the performance of tungsten materials [29–32]. Therefore, the performance of tungsten materials in poor fusion environments is worth discussing. Meanwhile, mechanical alloying, a processing technology for preparing balanced metastable phase powder, continues to face many obstacles, such as uneven distribution and aggregation of the second phase [33–35]. This study uses nano-TiC, titanium hydride powder, and tungsten powder to achieve mechanical alloying and adapts spark plasma sintering (SPS) to prepare TiC-doped W-Ti composite material. These procedures are accomplished to study the influence law of different milling times on the microstructure and properties of TiC-doped W-Ti composite material. We also assess the effect of heat load of composite materials prepared under different milling times.

2. Experimental process

The original powder used in our study is pure tungsten powder (grain size 1.2 μm). Micro-titanium hydride powder (45 μm grain size) and nano-titanium carbide powder (20 nm grain size) are also utilized. The mass ratios of titanium hydride and titanium carbide in the tungsten materials are 15 and 1.5 wt%. All the powders are placed in an agate jar with carbide to achieve tungsten carbide grinding at a ball material ratio of 20:1. To avoid powder oxidation, argon is used as a kind of protective gas in the ball milling process. Powder collection is also conducted in a glove box. The ball milling time is set to 10, 20, 30, 40, 60, or 80 h in the ball milling process, with a rotation rate of 400 rpm. At the end of ball milling, the powders generated after 10, 30, 60, and 80 h ball milling are adopted for use in SPS at sintering temperature of 1600 °C for 3 min under a maximum sintering pressure of 43.6 MPa. Four sintered bulks are named in this paper as WTT-10, WTT-30, WTT-60, and WTT-80, respectively. Fig. 1 shows the technical process of SPS.

The powder obtained after ball milling and the sintered bulk are characterized by X-ray diffraction (XRD). The tissue and components of the sintered bulk are analyzed by using field-emission scanning electron microscopy (FE-SEM) and energy dispersive X-ray spectrometry (EDS). The microstructural morphologies of the powder and bulk are analyzed by transmission electron microscopy. The density is measured by the Archimedes drainage method. The load and time of Vickers microhardness are 200 gf and 10 s, respectively.

During the laser beam heat-shock experiment, the sintered specimen is first cut into a rectangular block with dimensions 2 mm × 4 mm by line cutting. Then, experimental surface is polished to eliminate the influence of a rough surface. The laser beam heat-shock device is then used to accomplish a transient heat load experiment at pulse width 1 ms, current 60 A, and laser beam spot diameter 0.6 mm. The estimated power density range is 44 MW/m^2 to 47 MW/m^2.The experiments were carried out in atmosphere of Ar, sprayed from the source of the laser beam gun and mainly covering the central zone.

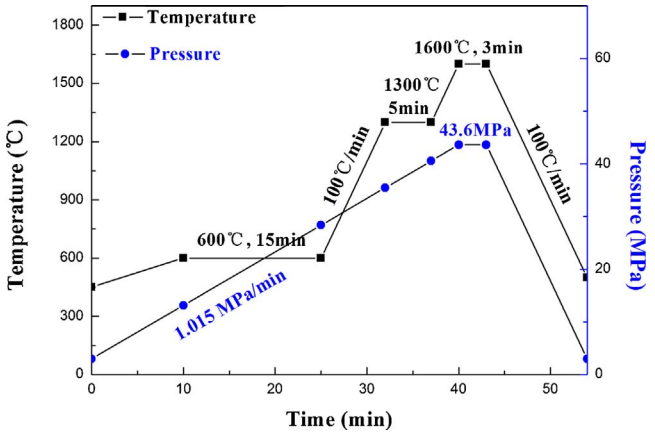

Fig. 1. Schematic of the technical process of SPS.

3. Result and analysis

3.1. Powdery analysis

Fig. 2 shows the scanning electron microscopy images of composite powders in different ball milling times. A-f refer to the milling times of 10, 20, 30 40, 60, and 80 h. Powder particles show irregular shapes with rough surfaces and a uniform grain size distribution (Fig. 2a-b). We speculate that the fracture mechanism in this period is stronger than that by cold welding. Cold-welding aggregation of the powder can be clearly observed with increasing large ball particles (Fig. 2c-e). In Fig. 2c, small quantities of large particles are observed. However, a greater number of large particles are found in Fig. 2e. Furthermore, the high surface area enables the small particles to adhere together and form a large ellipsoidal particle. However, when the ball milling time increases to 80 h, grain size begins to shrink relative to the size produced after ball milling for 60 h. In this case, fracture mechanism again predominates.

Fig. 3 shows the XRD peak of composite powder in different ball milling times. In the early stage of ball milling (Fig. 3a-b), not only the W peak, but also the $TiH_{1.9}$ peak is detectable. With increasing ball milling time, the $TiH_{1.9}$ peak becomes undetected because of the shrinking of titanium hydride powder particles, which widens the peaks, or titanium hydride becomes decomposed [36]. When the ball milling time is 60–80 h, the peak of (Ti, W)C solid solution (Fig. 3e-f) can be observed. We speculate that after long-term ball milling, Ti can react with WC to form (Ti, W)C solid solution [37]. Moreover, with increasing ball milling time, tungsten diffraction peak width and strength are significantly reduced. The continuous refinement of tungsten particles and the formation of solid solution can be surmised. Fig. 4 shows the changing trend of grain size and micro-strain of composite powder with increasing ball milling time, calculated through the XRD pattern in Jade program. Since the start of ball milling to 40 h, the grain size of the powder is greatly refined to up to 26.3 nm. Then, from 40 h to 80 h, grain size becomes smaller to up to 25.1 nm. From 10 h to 40 h ball milling time, the micro-strain rapidly changes from 0.324% to 0.56%, and from 40 h to 80 h, slowly increases from 0.56% to 0.724%. Long time milling could dramatically increase the micro-strain of the composite powders because continuous severe deformation and broken