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# Preparation and microstructure characterization of W–0.1 wt.%TiC alloy via chemical method

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ABSTRACT

W–0.1 wt.%TiC materials with and without the addition of PVP were fabricated by wet-chemical method and spark plasma sintering. The microstructures were characterized by FESEM and TEM. The results revealed that the addition of PVP remarkably improved the uniform dispersion of TiC particles during the synthesis process. W–TiC without PVP showed dispersoids with size of 100–1000 nm were distributed unevenly in the tungsten matrix; only a few particles with the size of about 80–300 nm were located in tungsten grain interior. Conversely, the microstructure of W–TiC with PVP showed that dispersoids were dispersed well and kept about 80 nm. Moreover, the majority of them were uniformly distributed in the grain interior. The EDS analyses revealed that the dispersoids were composed of Ti, C, O and W. TEM observations further verified that the dispersoids were TiC particles. W–TiC with PVP showed better mechanical properties when compared with the samples of W–TiC without PVP and pure tungsten.



## 1. Introduction

Tungsten and its alloys have attracted extensive interest all over the world due to its excellent mechanical and thermal properties such as high melting point, high thermal conductivity, high hardness, low thermal expansion coefficients, high strength at elevated temperature, and low sputtering yield [1]. They are ideal candidate materials for high temperature applications, such as turbines [2], kinetic energy penetrators [3], and heating elements. Furthermore, tungsten has been chosen as the primary candidate material for the divertor and first wall plasma facing components (PFCs) in future nuclear fusion reactors such as ITER and DEMO [4–7]. However, the susceptibility of tungsten to brittle crack under recrystallized and irradiated conditions limits its application [1,8–9]. Dispersive particles (like $La_2O_3$, $Y_2O_3$, TiC) [10–12] reinforced tungsten materials that have been reported to mitigate this problem effectively. Among these dispersive particles, TiC with high melting temperature (~3067 °C), high hardness, excellent high temperature strength, and good corrosion resistance [13] would be an ideal choice to strengthen tungsten matrix.

Nanostructured W–TiC materials which were prepared by mechanical alloying (MA), hot isostatic pressing (HIP) and hot plastically working exhibited high bending strength, desirable impact ductility, excellent high temperature tensile strength, and outstanding irradiation resistance [14–18]. Most recently, UFGR (ultra-fine grained, recrystallized) and TFGR (toughened, fine grained, recrystallized) W–TiC materials [19–20] with nano TiC particles dispersed at the grain interior and grain boundary showed even better mechanical properties. This indicated that nano TiC particles can significantly strengthen tungsten alloys. To avoid long milling time and enlarge mass production, new methods to prepare nanostructured W–TiC materials have been explored.

Recently, our group prepared well-dispersed TiC/W core-shell nanoparticles with uniform diameters about 100 nm [21]. Unfortunately, a few large TiC particles (300–1000 nm) were located at the grain boundaries, even though a majority of fine TiC particles (80–300 nm) were distributed inside the tungsten grains. We assume that these large TiC particles were derived from the poor dispersion of primary nano TiC powders in AMT (ammonium metatungstate) solution. In this paper, we further developed this process by using PVP (Polyvinylpyrrolidone) as dispersion agent to avoid the aggregation of nano TiC particles. The synthesized precursor powders were reduced and then sintered by SPS. The expected microstructure with uniformly distributed nano TiC particles in the grain interior was obtained. And the microstructure and mechanical properties of the as fabricated W–0.1 wt.%TiC materials were characterized.

## 2. Experimental procedure

### 2.1. Precursor preparation, reduction and sintering

The details of precipitation-coating method to synthesize the W–0.1 wt.%TiC composite powders with core-shell structure were presented elsewhere [21]. 1.0 wt.% PVP (Polyvinylpyrrolidone, K30) was added into the suspension solution of TiC powders before ultrasonic process to avoid TiC aggregation. The precursor was calcined in an electric oven under 550 °C to decompose PVP. The reduction process was

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performed under 800 °C for 1 h. The other fabrication processes were still the same as the reference [21]. The reduced powders were subjected to SPS sintering at 1600 °C and 50 MPa for 1 min. The heating rate was 150 °C/min from room temperature to 700 °C, 80 °C/min from 700 °C to 1200 °C, and 150 °C/min from 1200 °C to 1600 °C. Besides, commercial pure tungsten (CPW) powder with average Fisher particle size of 1.5 μm which was purchased from Beijing Tian-Long Tungsten & molybdenum CO., Ltd. were sintered under the same route.

### 2.2. Characterization

The crystal structures were identified by X-ray diffraction (XRD). FESEM and TEM were used to observe the microstructure and morphology. TEM specimens were prepared by mechanical grinding and ion thinning.

The density of sintered samples was measured by the Archimedes method. Vickers micro-hardness tests were performed under a load of 1.96 N for 10 s. Three point bending test was conducted at room temperature to measure the fracture strength. The specimens with dimensions of 3 × 2 × 16 mm were used. The working span was 10 mm and the constant loading rate was 0.5 mm/min.

## 3. Results and discussion

Fig. 1 shows the FESEM images of the as-received nano TiC powders (a) and commercial pure tungsten powders (b). The average diameter of the TiC particles was about 80 nm. The TiC particles gathered into agglomerates revealed the basic characteristics of nano TiC particles. The commercial pure tungsten powders with an average diameter of 1.5 μm (Fig. 1(b)) displayed aggregate morphology. Fig. 2 shows the FESEM images of the reduced W-TiC powders with and without the addition of PVP. Both kinds of powders showed similar aggregate morphologies with primary particles size of 30–700 nm which demonstrated the addition of PVP had negligible influence on the reduction of W–TiC precursors.

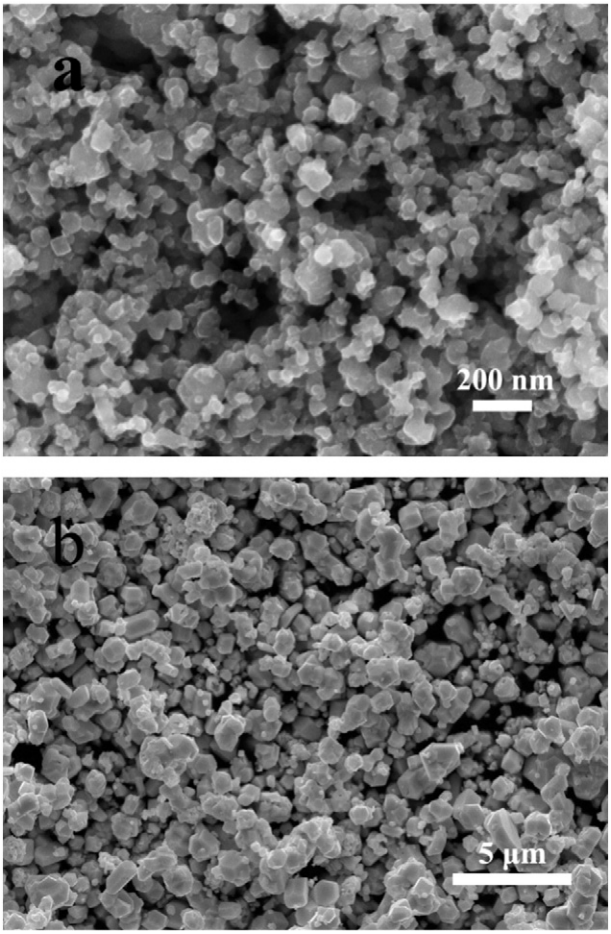


**Fig. 1.** FESEM images of (a) nano TiC powders and (b) commercial pure tungsten powders.

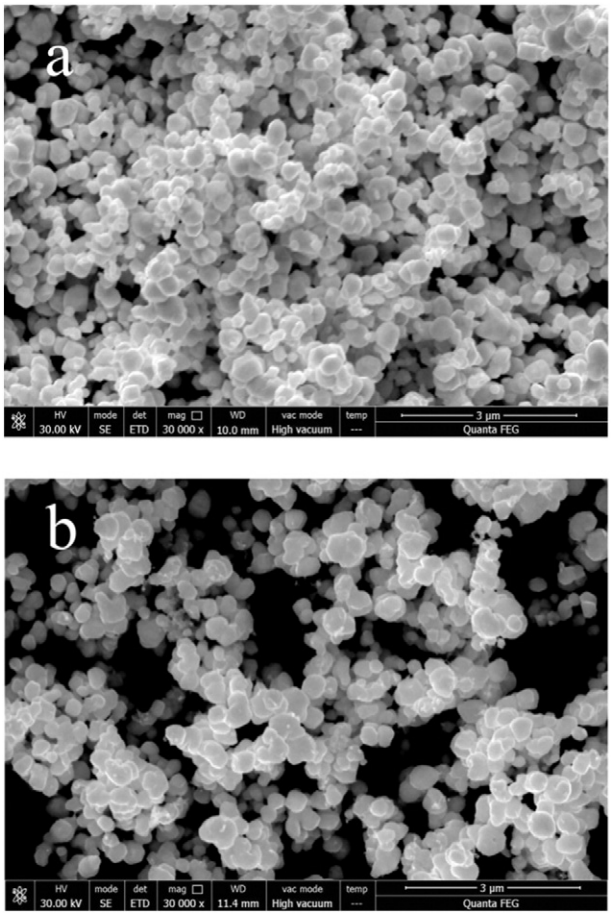


**Fig. 2.** FESEM images of reduced W–TiC powders (a) without PVP and (b) with PVP.

The XRD patterns of the sintered pure tungsten, W–TiC samples and pure TiC powders are shown in Fig. 3. All the peaks of the W–TiC

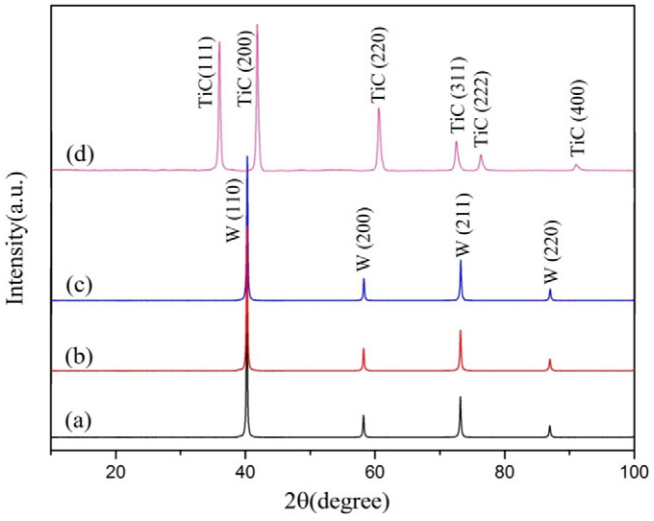


**Fig. 3.** XRD patterns of the sintered (a) pure tungsten, (b) W–TiC without PVP, (c) W–TiC with PVP, and (d) pure TiC powders.