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Preparation and thermal shock characterization of yttrium doped tungsten-potassium alloy

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ABSTRACT

Novel tungsten-based W-K-Y alloys were sintered by spark plasma sintering (SPS) method using fine-grained yttrium doped tungsten-potassium (W-K) powder. The relative density, microstructure, hardness and the resistance to thermal shock damage of the sintered samples were characterized. With the enhanced Y doping, the grain size decreased and the hardness increased. Thermal shock test under 0.37 GW/m^2 heat load showed that low yttrium doping (0.05 wt%, 0.1 wt% and 0.5 wt%) in W-K-Y alloys can improve the resistance to thermal shock damage comparing with traditional commercial W-K, while high yttrium doping (1 wt%) easily leads to crack formation. This study will provide helpful information to optimize the preparation of tungsten-based plasma facing materials through composite tuning.

1. Introduction

Tungsten and tungsten-based alloys are important materials in lighting, electronics, military and in particular fusion devices due to their good thermal conductivity, high melting point, low erosion rate and low tritium retention under the extreme service environment (high thermal load, high flux H/He plasma and neutron irradiation) [1,2]. It is regarded as one of the promising candidates for plasma facing materials (PFMs) in fusion devices and fusion reactors and has been widely studied in recent years. Some properties such as its low ductile-brittle transition temperature (DBTT), irradiation embrittlement and irradiation swelling are not excellent enough serving as PFMs to date, and thus need to be further improved [3]. Especially, resistance to thermal shock is considered as one of the most important properties for PFMs, because high steady state heat load (10–20 MW/m^2) will be superimposed with transient heat loads in the sub-ms and ms-range (at low and high frequencies; up to the GW/m^2 range) on the first wall during plasma operation [4,5]. Such heat load may cause surface melting, cracking, recrystallization and droplet ejection on the surface of PFMs, which will seriously shorten their service life [6]. Therefore, it is of great significance to develop high-thermal-shock resistant tungsten alloy as PFMs.

Tungsten-potassium (W-K) alloy is a traditional material with high thermal shock resistance. The excellent mechanical properties and higher recrystallization temperature of AKS-doped tungsten (also named non-sag tungsten) was found in early 1930's in lamp filament industry [7]. The AKS-doping means the potassium bubbles form from the decomposition of the Al-K-Si dopant particles under certain process [8]. The potassium bubbles were found to perform the similar function to other dispersion strengthening phase [7]. Some studies have been carried out on the thermal shock resistance of commercial W-K. It showed that the surface damage of commercial W-K is largely mitigated compared with pure W, displaying improvement of mechanical properties [9]. Traditional commercial W-K bulk material is fabricated through pressing, vertical sintering and swaging, and finally the potassium bubble size is tuned to 50–200 nm and mainly located on grain boundary [10]. Non-sag W-K with smaller bubble size are fabricated by rotary swaging and high temperature annealing after cold isostatic compaction and vertical sintering, but the final product is tungsten wire which is difficult to be used as PFMs. In our previous studies, SPS sintered AKS-W bulk samples with small size (20–100 nm) and intra-granular potassium bubble was obtained [8].

Besides potassium bubble strengthening, Oxide Dispersion

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Strengthening (ODS) is another common method in alloy strengthening. Especially, La_2O_3 [11,12] and Y_2O_3 are usually used for tungsten alloy strengthening [13]. The high temperature stability of Y_2O_3 makes it suitable for oxide dispersion strengthening in tungsten alloys. People have studied yttrium oxide doped tungsten and high temperature tensile strength and fracture strength were obviously improved [14]. Actually, low amount Y_2O_3 doping can effectively refine the tungsten grain, and enhance its recrystallization temperature and reduce DBTT. Considering free oxygen is regarded as the main source of crack formation to PFMs [15], yttrium can adsorb free oxygen and thus should be more suitable as the dopant rather than yttrium oxide. Veleva et al. have developed a new W-2Y material by mechanical alloying and hot isostatic pressing [16]. The yttrium dopant was finally oxidized into nano size yttrium oxide.

Considering the respective advantages of potassium bubble and yttrium strengthening, there is a high interest in developing W-K-Y ternary tungsten alloy as PFM, and thus need to be systematically studied. In this work, a novel tungsten-based alloy, namely W-K-Y ingots are fabricated by powder metallurgy method to get high homogeneity and fine grain size through spark plasma sintering (SPS) technique. The effects of doping contents on microstructure, hardness, density and resistance to thermal shock were investigated.

2. Experiment details

Commercial AKS tungsten powder (99.9% purity, particle size3–4 μm), yttrium powder (99.99% purity, particle size 30–70 μm) were used as the starting materials. The AKS tungsten powder is fabricated by adding aqueous solutions of Al-K-Si-containing compounds to the tungsten blue oxide (TBO). Hydrogen reduction of the doped TBO leads to a metal powder with potassium-containing dopant phases within the tungsten grains [17]. The AKS component is shown in Table 1.

Five starting powders with different yttrium doping ratio (0, 0.05 wt%, 0.1 wt%, 0.5 wt% and 1 wt%) were used to prepare bulk samples, as shown in Table 2. The powders were put into WC/8Co milling vessel with tungsten carbide balls (diameter of 5 mm and 10 mm with a mass ratio of 2:1) at the ball-to-powder weight ratio of 5:1. After 40 h high energy ball milling under rotation speed of 250 rpm and in hydrogen and argon mixed atmosphere (1:12 in volume ratio, 99.9999%), the powders were sufficiently mixed and the grain size is supposed to be refined in this process according to our previous studies [8,14].

The consolidation of the mixed powder was carried out by SPS method. The mixed powders were fed into a graphite die of 15 mm diameter and a pre-compacting of 80 MPa was done before sintering. The consolidation scheme is shown in Fig. 1. The SPS sintering machine was heated to 1750 °C from room temperature at the heating rate of 100 °C/min and hold for 3 min, the pressure would increase from 20 to 80 MPa with the increasing temperature in two steps. All the consolidation took about 19 min not include the cool down time and were carried out in vacuum (5 Pa). When the heating process was over, the samples were naturally cooling to room temperature and the pressure was removed at the end of the whole sintering process. Before the thermal shock tests, all the samples were polished to mirror-like to reduce the surface roughness. And stress relief treatment was carried out at 1273 K in vacuum (10^{-1} Pa) to obtain better mechanical properties.

Table 1
The component of commercial AKS tungsten powder.

	Si	K	Al	Ni	Fe	Cr	N	O_2	Co	C
Content (ppm)	185	82	30	3	28	3	13	1000	4	10

Table 2
The yttrium doping ratio of starting powder.

	1	2	3	4	5
Doping percentage (wt%)	0	0.05%	0.1%	0.5%	1%

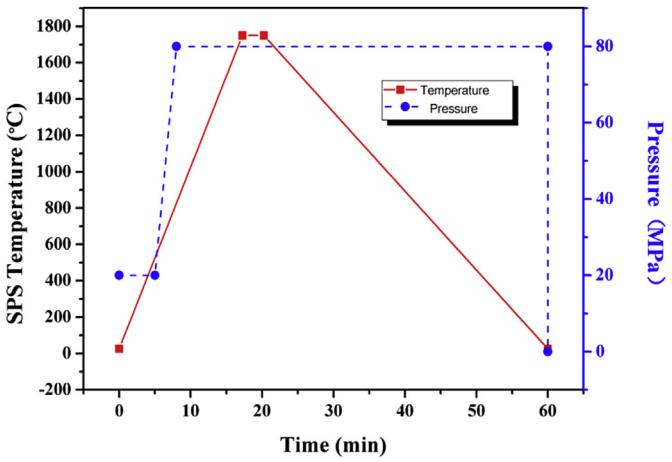

Fig. 1. Schematic heating cycles for sintering process.

Thermal shock tests were performed in the electron beam test facility EMS-60 at Southwest Institute of Physics (SWIP). Single shot and multiple shots (100) tests with different pulse duration (5 ms for single shot and 1 ms for multiple shots) were carried out. The accelerating voltage of electron beam is 120 kV and the average beam current is 90 mA. The heat load scanning area is 4 × 4 mm^2, the heat flux could be calculated as 0.37 GW/m^2 using the formula $P = UI\alpha$. Here, the value of electron absorption coefficient α is 0.55, which is a result of Monte Carlo Simulation. The power density corresponds to a heat flux factor $F_{HF} = P_{abs}\Delta t^{0.5} = 26.2MW/m^2s^{0.5}$ for a single shot and $F_{HF} = P_{abs}\Delta t^{0.5} = 11.7MW/m^2s^{0.5}$ for 100 shots. In addition, the thermal shock tests were performed at room temperature and in a vacuum degree of 10^{-2}Pa.

Material properties were characterized to verify that the manufacturing were successful and can be used to interpret the thermal shock tests. The density was measured by drainage method. The theoretical density of the yttrium doped AKS samples were calculated from the fraction and theoretical density of each component. The thermal shock damage and microstructure were investigated through optical microscopy and scanning electron microscopy (SEM, Hitachi S4800). Vickers hardness tests were performed under a load of 200 g and hold for 10 s.

3.. Results and discussion

The SEM morphologies of initial commercial AKS powder, yttrium powder and mixed powder after 40 h ball milling are displayed in Fig. 2. Comparing Fig. 2a with Fig. 2f, the real size of powder particles are almost the same, but the surface of the ball milled powder is much rougher than that before after ball milling. As shown in Fig. 2d, the powder was crushed into flat particles after 20 h's ball milling. With the ball milling time increasing, the powder particle size slightly decreased. The morphologies of mixed powder after 30 h and 40 h's ball milling are nearly the same, and the extended 10 h is to fully ensure that the yttrium particles are crushed into small size. For inter-metallic powder like yttrium