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Preparation of tungsten coatings on graphite by electro-deposition via Na_2WO_4–WO_3 molten salt system

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HIGHLIGHTS

- Tungsten coatings on graphite were firstly obtained by electro-deposition method via Na_2WO_4–WO_3 molten salt system.
- Uniform and dense tungsten coatings could be easily prepared in each face of the sample, especially the complex components.
- The obtained tungsten coatings are with high purity, ultra-low oxygen content (about 0.022 wt%).
- Modulate pulse parameters can get tungsten coatings with different thickness and hardness.

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ABSTRACT

Tungsten coating on graphite substrate is one of the most promising candidate materials as the ITER plasma facing components. In this paper, tungsten coatings on graphite substrates were fabricated by electro-deposition from Na_2WO_4–WO_3 molten salt system at 1173 K in atmosphere. Tungsten coatings with no impurities were successfully deposited on graphite substrates under various pulsed current densities in an hour. By increasing the current density from 60 mA cm^{-2} to 120 mA cm^{-2} an increase of the average size of tungsten grains, the thickness and the hardness of tungsten coatings occurs. The average size of tungsten grains can reach 7.13 μm, the thickness of tungsten coating was in the range of 28.8–51 μm, and the hardness of coating was higher than 400 HV. No cracks or voids were observed between tungsten coating and graphite substrate. The oxygen content of tungsten coating is about 0.022 wt%.

1. Introduction

It has been discovered that tungsten has a good compatibility with fusion plasma [1] due to its high melting point, good thermal properties and low erosion rate. However, it is counteracted by a number of critical issues, such as poor processing, poor welding performance, high cost and heavy weight. Carbon materials, like graphite and C/C fiber composite (CFC) have been used for plasma facing materials (PFMs), because of their high thermal shock resistance, light weight and high strength. However, one critical issue is high erosion rates of graphite or CFC at elevated temperature. A solution to overcome this issue is tungsten coating of carbon materials like graphite or CFC. This solution has been successfully applied in ASDEX Upgrade [2,3] for many years and recently in the ITER-like Wall at JET [4]. Nowadays the techniques such as plasma spray (PS), physical vapor deposition (PVD) and chemical vapor deposition (CVD), combined magnetron sputtering and ion implantation (CMSII) techniques have been applied in tungsten coating of graphite as PFMs [5–9]. However, there are still some disadvantages, for example, detrimental phases is prone to being introduced; the cost is high; the procedures are complicate, etc.

Electro-deposition is a viable and promising method for preparing tungsten coatings due to the simple operation and low cost. Furthermore, uniform and dense tungsten coatings could be easily prepared on each face of the sample. Recently, Liu et al. obtained pure tungsten coatings on Al_2O_3–Cu substrates from the Na_2WO_4–WO_3 melt [10,11]. It is a great prospect of application, because this molten salt system is not sensitive to oxygen or water, it also has the advantages of simple operation, low cost, low equipment requirement, etc. This stimulated us to study the possibility of fabrication tungsten coatings on graphite. By using this method, tungsten coatings on graphite substrates with high purity, low oxygen content are expected to be prepared.

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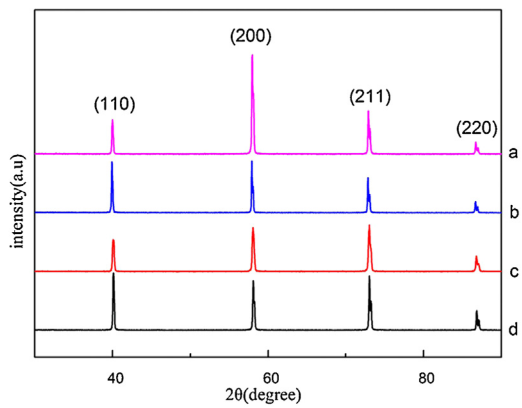

Fig. 1. XRD patterns of tungsten coatings obtained at current density of (a) 60 mA cm^{-2}, (b) 80 mA cm^{-2}, (c) 100 mA cm^{-2}, and (d) 120 mA cm^{-2}.

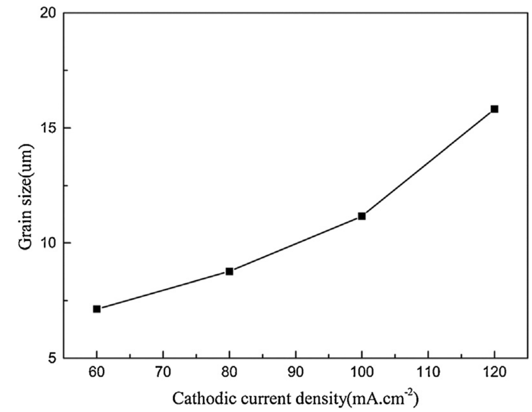

Fig. 3. Average grain size of tungsten coatings obtained at current density of (a) 60 mA cm^{-2}, (b) 80 mA cm^{-2}, (c) 100 mA cm^{-2}, and (d) 120 mA cm^{-2}.

In this paper, tungsten coatings on graphite substrates were electro-deposited from the Na_2WO_4–WO_3 melt at 1173 K. In order to obtain well defined structures for tungsten coatings, different pulsed current densities were investigated. The study of pulsed current density on tungsten nucleation and its performance were conducted.

2. Experimental procedure

Graphite (IG-430) substrate of 15 mm × 10 mm × 5 mm was coated with tungsten by electro-deposition, using anhydrous Na_2WO_4 and WO_3 (Na_2WO_4:WO_3 = 3:1, by mole ratio) molten salts. The working electrodes (graphite substrates) were polished by mechanical and chemical methods to obtain high quality surface. And counter electrode was a tungsten plate (15 mm × 10 mm × 5 mm). Molten salts were mixed into the eutectic composition in an alumina crucible. Then the crucible was put in an electric furnace and was heated at a rate of 5 °C/min. Electro-deposition was performed at 1173 K using a pulse power supply (HPMCC-5) holding for one hour, the duty cycle and period were 0.8 and 2 ms, respectively. After the deposition, the sample was fetched out and cooled in the air. Subsequently, the samples were ultrasonic-cleaned in a 10 M NaOH solution and deionized water to remove adherent salts. The composition, structure, thickness of the tungsten coating were analyzed by Energy Dispersive X-ray Fluorescence (EDXRF, EDX-720) and X-ray diffraction (XRD, Rigaku Industrial Co., Ltd., D/MAX-RB) with a scanning electron microscope (SEM, JSM 6480LV). Micro-hardness of the coatings was performed by a MH-6 micro-hardness instrument with a loading force of 50 g and loading time of 15 s. The adhesion was detected by nano scratch tests. The oxygen content was measured by the Nitrogen/Oxygen Analyzer (TC600, LECO, USA).

3. Results and discussion

The coatings were electro-deposited in the Na_2WO_4–WO_3 melt at 1173 K for 1 h with different pulse current densities (60 mA cm^{-2}, 80 mA cm^{-2}, 100 mA cm^{-2} and 120 mA cm^{-2}). The duty cycle was 0.8 for a period of 2 ms. Then the W coatings were investigated in terms of composition, morphology, oxygen content, adhesion and hardness.

3.1. Composition

The intrinsic structure of tungsten coating was analyzed by XRD. Fig. 1(a)–(d) is the XRD patterns refer to current density of 60 mA cm^{-2}, 80 mA cm^{-2}, 100 mA cm^{-2}, and 120 mA cm^{-2}, respectively. As can be seen, the crystal planes of (1 1 0), (2 0 0), (2 1 1)

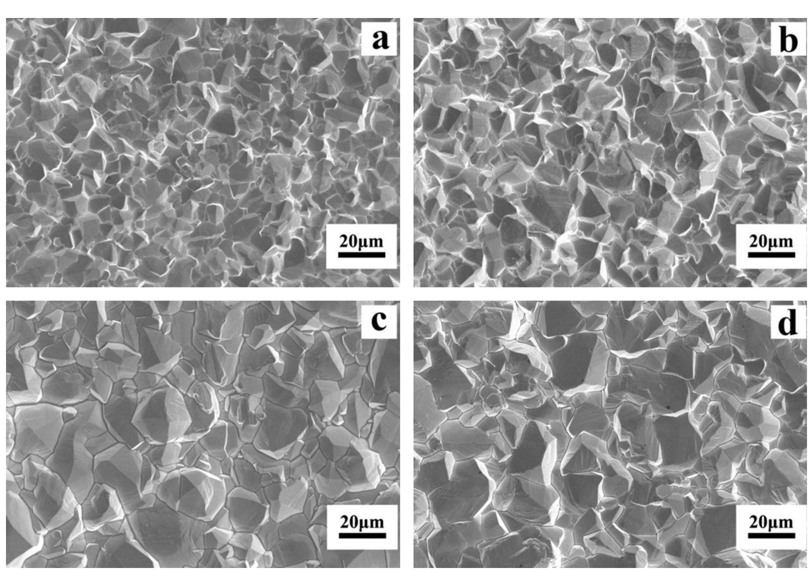

Fig. 2. The surface SEM images of electro-deposition tungsten coatings obtained at duty cycle of 0.8 and current density of (a) 60 mA cm^{-2}, (b) 80 mA cm^{-2}, (c) 100 mA cm^{-2}, and (d) 120 mA cm^{-2}.