Fusion Engineering and Design 112 (2016) 136–142

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

Electrodeposition of tungsten coatings on molybdenum substrates and deuterium irradiation effect

Ziwei Lian [a], Xianqin Fang [b], Wenjia Han [a], Jiangang Yu [a], Zhanlei Wang [a], Ying Zhang [a], Kaigui Zhu [a,c,*]

[a] Department of Physics, Beihang University, 37 Xueyuan Road, Haidian District, Beijing 100191, China
[b] Key Laboratory of Micro-Nano Measurement-Manipulation and Physics, Ministry of Education, 37 Xueyuan Road, Haidian District, Beijing 100191, China
[c] Beijing Key Laboratory of Advanced Nuclear Energy Materials and Physics, Beihang University, Beijing 100191, China

HIGHLIGHTS

- Tungsten coatings were successfully electroplated on molybdenum substrates.
- The current density affected the performance of tungsten coatings.
- Deuterium irradiation property of tungsten coatings was investigated.
- Deuterium retention in the tungsten coating was less than that in the bulk tungsten.

ARTICLE INFO

Article history:
Received 22 March 2016
Received in revised form 4 August 2016
Accepted 5 August 2016

Keywords:
Tungsten coating
Electrodeposition
Current density
Deuterium irradiation
Thermal desorption spectroscopy

ABSTRACT

Tungsten coatings were prepared using pulse electrodeposition on the molybdenum substrates. Effects of variations in current density on surface morphology, thickness distribution and crystal orientation of the coatings were investigated. The results indicate that with the current density increasing, the grain size of tungsten coatings first decreases, then increases; while the deposited thickness increases all the time. And all of tungsten coatings exhibit the preferred orientation of (200) plane. Moreover, the polished tungsten coating and bulk tungsten were exposed to low energy (80 eV) and high flux (7.2×10^{20} D/m^2/s) deuterium plasma in a linear plasma device (Simulator of Tokamak Edge Plasma, STEP). Deuterium (D) retention was measured by thermal desorption spectroscopy (TDS). It is found that blisters on the tungsten coating are much fewer than that on the bulk tungsten. TDS spectroscopy of the tungsten coating reveals one D_2 release peak at 740 K, while the bulk tungsten has two D_2 release peaks at 500 K and 660 K. The amount of deuterium retention in the tungsten coating is lower.

1. Introduction

As a high-Z material, tungsten(W) is an excellent candidate plasma facing material (PFM) for Experimental Advanced Superconducting Tokamak (EAST) [1] and International Thermonuclear Experimental Reactor (ITER) [2] because of its high threshold for physical sputtering energy ($E_{th}\sim$ = 200 eV for deuterium), high melting point (T_m = 3695 K), low vapor pressure ($P_v = 1.3 \times 10^{-7}$ Pa at *T*m), high thermal conductivity and good thermal shock resistance [3]. However, the limiting factors of tungsten are its poor workability, low fracture toughness (FT) and high ductile-to-brittle-transition temperature (DBTT) [4,5]. Intensive effort on material processing has been carried out trying to improve tungsten performance. Many grades of tungsten and tungsten alloy were investigated, like sintered W, rolled W, metal injection molded (MIM)-W made by hot isostatic pressing, nano-structure W, oxide dispersion strengthened W, W–Si–Cr et al. [6]. Many previous researches have studied the basic characteristic and irradiation effect on these W materials [7–10]. Besides on bulk tungsten, varies of tungsten coating techniques, including magnetron sputtering, vacuum plasma spray(VPS), physical vapor deposition (PVD) and chemical vapor deposition (CVD) have been investigated over recent decades [11]. However, another technique, pulse electrodepositing tungsten, looks particularly attractive one owing to its simplicity and low cost [12]. Thus, one possible solution for the utilization of tungsten as plasma facing is to coat the structure or heat sink material with a tungsten layer by electrodeposition. Further-

* Corresponding author.
E-mail address: kgzhu@buaa.edu.cn (K. Zhu).

http://dx.doi.org/10.1016/j.fusengdes.2016.08.003
0920-3796/

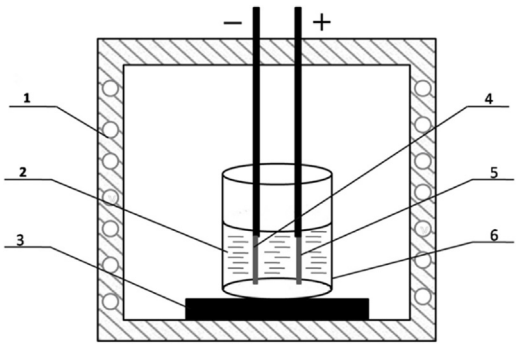

Fig 1. Schematic diagram of the coating device: (1) vertical electric resistance furnace; (2) molten salt of Na_2WO_4 and WO_3; (3) firebrick; (4) cathode electrode (molybdenum); (5) anode electrode (tungsten); (6) aluminum crucible.

more, the study on the deuterium irradiation of tungsten coatings by electrodeposition has been not so much for these years.

As early as in 1960s, Senderoff and Mellors [13] had successfully obtained compact and smooth tungsten film by electroplating technique in a LiF-KF-NaF molten salt. And Baraboshkin et al. [14] electrodeposited tungsten coating from Na_2WO_4–WO_3 system at the temperatures of 923–1223 K. V.A. Pavlovskii [15] electrodeposited tungsten coatings on molybdenum substrates from chloride-fluoride-WO_3 melt at 1113–1193 K. Zhang et al. focused on the research of work electrode in Na_2WO_4–WO_3 system and successfully obtained thin tungsten coating on the substrate of Al_2O_3—Cu alloy [16], low activation steel [17], V-4Cr-4Ti alloy [18] and graphite [19], respectively. Electrodeposition of tungsten coatings on molybdenum from Na_2WO_4–WO_3 molten salt has not been reported in past literature and molybdenum is chosen as a substrate material for tungsten coating because of the match of crystal structure and physics properties, such as the same bcc structure and high melting point [20].

In this work, the dense tungsten coatings were prepared by pulse electrodeposition on pure molybdenum from Na_2WO_4–WO_3 molten salt at 1173 K in atmosphere. Effects of variations in current densities on surface and cross-sectional morphology and crystal orientation of tungsten coating were investigated. In addition, blistering and deuterium retention in the electrodeposited tungsten coating and the bulk tungsten exposed to high fluence (up to 10^{24} D/m^2) of high flux (10^{20} D/m^2/s) and low-energy (<100 eV) deuterium plasma was examined.

2. Experimental

All the chemicals were anhydrous reagent grade. $Na_2WO_4{\cdot}2H_2O$ was dried at 773 K for 12 h and WO_3 was dried at 573 K for more than 8 h in furnace, respectively. The anhydrous reagents were thoroughly mixed into an eutectic composition (Na_2WO_4: WO_3 = 0.6: 0.2, in mole ratio) in an alumina crucible, and then melted in a vertical electric resistance furnace at 1173 K. Fig. 1 shows a schematic diagram of the device. The two electrodes fixed to graphite rod were immersed in the molten salt. The working electrode was a pure molybdenum plate (99.9%, 20 mm × 10 mm × 5 mm). A polycrystalline bulk tungsten plate (99.9%, 20 mm × 10 mm × 5 mm) was used as the counter electrode. The vertical resistance furnace was used to heat the salt. Prior to electrodeposited, the surface of electrodes were polished by mechanical method, and then ultrasonically cleaned in acetone and distilled water in sequence and finally dried by nitrogen puffing. Tungsten coatings were prepared on the molybdenum substrates at current densities of 10, 30, 50, 70 mA/cm^2 using a pulse power supply (MS-MCC) holding for 2 h, with the duty cycle of 0.3 and period of 1 ms. The obtained samples were immediately immersed in NaOH aqueous solution (5 mol/L) to remove adherent salts, and then ultrasonically cleaned in distilled water.

The surface morphologies of the coatings were examined by scanning electron microscopy (SEM, Quanta 250 FEG), After mechanical polishing, the morphology and composition of the coatings were then characterized by scanning electron microscopy (SEM, S4800) and energy dispersive spectrometer (EDS, IE 300 X) from their cross section. In addition, the phase composition and crystal orientation of deposits were determined by X-ray diffraction (XRD, XRD-6000). File test was applied to evaluate the adherence of tungsten coating. The oxygen content of the tungsten coating was measured by the Nitrogen/Oxygen Analyzer (TC600, LECO,USA). Archimedes method was applied to measure the density of tungsten coating.

In this work, the electrodeposited tungsten coating and polycrystalline bulk tungsten were mechanically polished to a mirror-like finish, and then electro-polished in 5 wt.% NaOH solution. These two specimens were exposed to a low energy (80 eV), high fluence (3.9×10^{24} D/m^2) of high-flux (7.2×10^{20} D/m^2/s) deuterium (D) plasmas at 463 K in a recently built linear plasma device (Simulator of Tokamak Edge Plasma, STEP) in Beihang University [21]. The generator consists of vacuum chamber, plasma source, target station and magnetic coils, with peripheral system of vacuum pumping station, power supplies and gas and water cooling circulating units. Plasma was ignited by an electron-emitting LaB_6 cathode. A single Langmuir probe is equipped to obtain basic plasma parameters. The temperature of the sample was measured using a K-type thermocouple tightly pressing the rear of the sample. After the plasma exposures, the morphologies of irradiated samples were observed by scanning electron microscopy (SEM, S4800). The desorption behavior of D was analyzed by thermal desorption spectroscopy (TDS). The samples were heated from 300 K to 1273 K at a

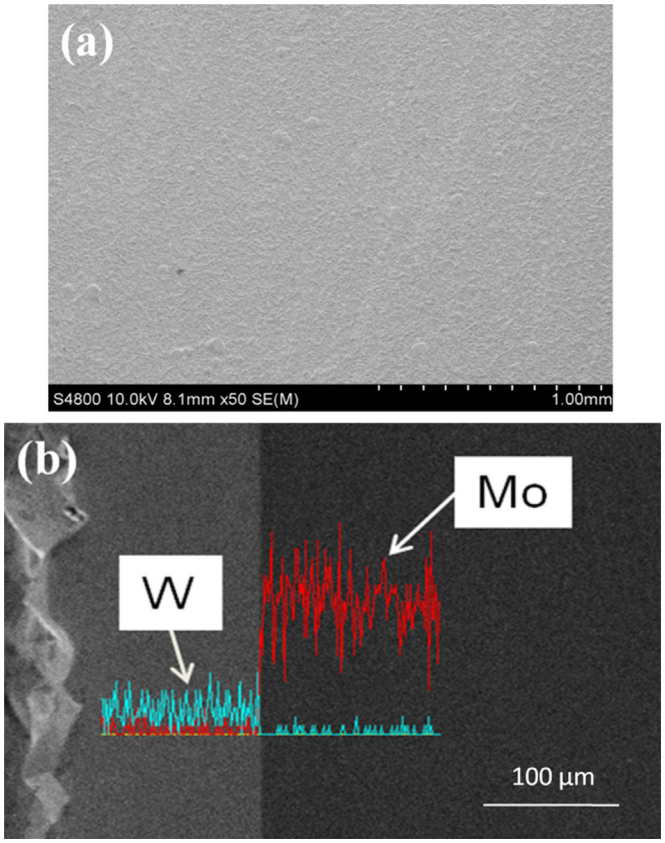

Fig. 2. (a) Surface morphology, (b) cross-sectional morphology and line scanning analysis of EDS.