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Effect of cathodic current density on performance of tungsten coatings on molybdenum prepared by electrodeposition in molten salt

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ABSTRACT

Smooth tungsten coatings were prepared at current density below 70 mA cm^{-2} by electrodeposition on molybdenum substrate from Na_2WO_4-WO_3 -melt at 1173 K in air atmosphere. As the current density reached up to 90 mA cm^{-2}, many significant nodules were observed on the surface of the coating. Surface characterization, microstructure and mechanical properties were performed on the tungsten coatings. As the increasing of current density, the preferred orientation of the coatings changed to (200). All coatings exhibited columnar-grained-crystalline. There was about a 2 μm thick diffusion layer between tungsten coating and molybdenum substrate. The bending test revealed the tungsten coating had –good bonding strength with the molybdenum substrate. There is a down trend of the grain size of the coating on molybdenum as the current density increased from 30 mA cm^{-2} to 50 mA cm^{-2}. The coating obtained at 50 mA cm^{-2} had a minimum grain size of 4.57 μm, while the microhardness of this coating reached to a maximum value of 495 HV.

1. Introduction

Owing to its excellent strength at high temperature, high thermal conductivity and elastic modulus, low thermal-expansion coefficient, sound corrosion resistance, molybdenum has been widely utilized in the aerospace, metallurgy, nuclear and energy industries, including high performance electronics, lighting technology, high temperature furnace construction, and sputtering targets [1–5]. However, molybdenum is vaporized in vacuum and reacts easily with shielding materials in the high-temperature vacuum condition [6].

As we know, tungsten exhibits high melting point among the metals, possesses high physical sputtering threshold energy, good thermal properties under high temperature condition [7–9]. Therefore, applying tungsten coating opens up a new way to increase the working ability of molybdenum (Mo). Nowadays, as technologies able to realize tungsten coatings on molybdenum substrates, chemical vapor deposition (CVD) [8] and plasma spray (PS) [5] have been proposed and studied. However, each method has its own disadvantages. PS presents high porosity and micro-cracks [9] and comparatively low strength between coating and substrate. CVD method also has many shortcomings, such as complicated procedures and poisonous.

As compared with above technologies, electroplating from molten salts is a promising and ideal surface finishing process for fabricating tungsten layer over coat heat sinks because of its simplicity, low cost and easy implementation [10]. V. A. Pavlovskii successfully electrodeposited metallic tungsten coatings on molybdenum bars from chloride-fluoride-WO_3 melts [11]. The corrosion-resistant tungsten coatings could be deposited under the follow conditions: cathodic current density 0.01–0.05 A cm^{-2}, temperature 920 ± 20 °C. However, the effect of cathodic current density on the crystal structure, surface roughness and mircohardness of the coating need to be sufficiently studied.

The Na_2WO_4-WO_3 system is nonvolatile, chemically stable, easily prepared, and reduces the complexity of the facility for electroplating in molten salt [12]. In our previous work [13], a compact and smooth tungsten coating was prepared on molybdenum substrate in an open bath in the air atmosphere by electro deposition from this system. As we know, the nucleation energy and crystallization kinetics depends on the cathodic overpotential and the overpotential increases with increasing current density. In this study, the aim is to study the effect of the cathodic current density on the surface morphology and microstructure of tungsten coating.

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2. Experimental

Tungsten coatings were electrodeposited over molybdenum substrates by electrodepositing from molten salt at 1173 K. All electroplating - were performed using a pulse power supply (HPMCC-5). The experimental procedure was referred to the Ref. [13]. All the chemicals used in the experiments were anhydrous reagent grade. Na_2WO_4 and WO_3 were dried in a furnace at 773K for 24 h. The chemicals after drying treatment were - mixed into a eutectic composition (Na_2WO_4:WO_3 = 3:1, by mole ratio [14]) and then melt in an electric resistance furnace under air atmosphere at 1173K. The working electrode was a molybdenum plate (purity: 99.95%, 10 mm × 10 mm × 5 mm). A tungsten plate (purity: 99.95%, 15 mm × 15 mm × 5 mm) was used as a counter electrode. Prior to deposition, the electrodes' surfaces were mechanically polished and then ultrasonically cleaned in acetone and distilled water. The cathodic current density was varied from 10 to 90 mA cm^{-2}. The operating conditions for electroplating were summarized in Table 1. After the electroplating, samples were immediately immersed in a 5 M NaOH solution in order to remove adherent salts.

The obtained samples were examined by X-ray diffraction (XRD, Rigaku Industrial Co., Ltd., D/MAX-BB) using Cu K_α radiation from at a scan rate of 5°/min from 10° to 100° of 2θ for phase identification. Scanning electron microscopy (SEM, JSM 6480LV) was employed to characterize the surface morphologies of the deposits. The cross section morphologies were observed by scanning electron microscopy (SEM, JSM 6480LV) with line analysis. Surface roughness was tested by laser scanning confocal microscope (LSCM, LEXT OLS4000 3D) for four times and the average value was selected. The current efficiency (η) was calculated by the following formula:

$$\eta = \frac{m}{CIt} \times 100\% \quad (2\text{-}1)$$

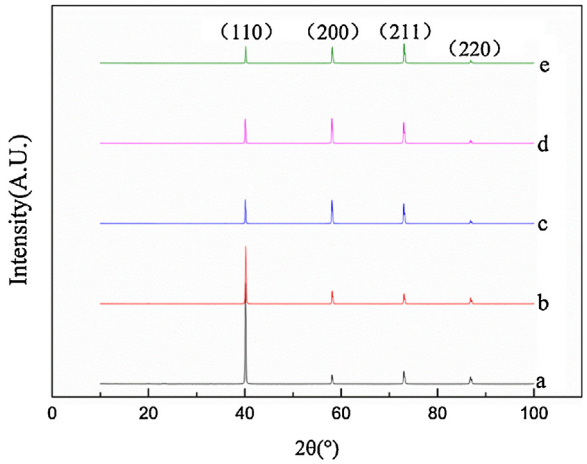

Fig. 1. XRD patterns of tungsten obtained at different current densities: (a) 10 mA cm^{-2} (b) 30 mA cm^{-2} (c) 50 mA cm^{-2} (d) 70 mA cm^{-2} (e) 90 mA cm^{-2}.

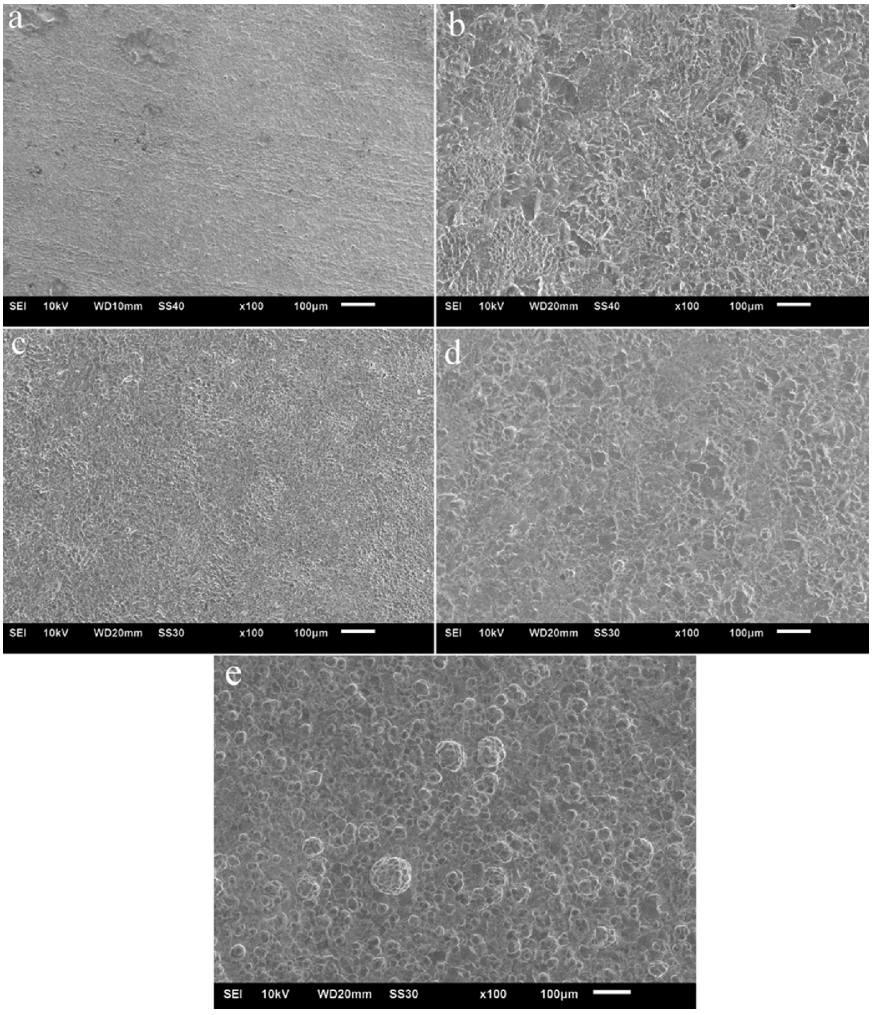

Fig. 2. Surface SEM images of tungsten coatings obtained at different current densities: (a) 10 mA cm^{-2} (b) 30 mA cm^{-2} (c) 50 mA cm^{-2} (d) 70 mA cm^{-2} (e) 90 mA cm^{-2}.