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Tungsten coating prepared on molybdenum substrate by electrodeposition from molten salt in air atmosphere

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ABSTRACT

Compact and smooth tungsten coating on molybdenum substrate was obtained by electrodeposition from Na_2WO_4–WO_3 molten salt at 1173 K in atmosphere. Microstructure, morphology and properties were performed on the tungsten coating. The tungsten coating had columnar structure with the preferential growth orientation of (2 0 0). There was about 2 μm thick diffusion layer of tungsten in the molybdenum substrate. The bending test and thermal shock test showed the tungsten coating had good adhesion with the molybdenum substrate. The microhardness of the coating was about 492 HV and the oxygen content of the coating was 0.032 wt%. The high-temperature could enhance the high-temperature oxidation resistance and bond strength of the tungsten coating.

1. Introduction

Characterized by excellent strength at high temperature, sound corrosion resistance, high thermal conductivity, high elastic modulus, and low thermal-expansion coefficient, molybdenum plays an important role in a range of applications in the metallurgy, aerospace, nuclear and energy industries, including high temperature furnace construction, lighting technology, high performance electronics, and sputtering targets [1–4]. But, the molybdenum is vaporized in vacuum and reacts chemically with shielding material in a high-temperature vacuum condition [5]. In addition, molybdenum degrades under prolonged exposure to thermal radiation and decreases its high temperature mechanical properties due to the embrittlement of large recrystallized grains [6,7].

Tungsten is the metal with the highest melting point among the metals, high physical sputtering threshold energy, and low erosion under plasma loading [8,9]. As a result, the deposition of tungsten coatings on molybdenum substrate is one possible method to improve the working ability of molybdenum. As coating technologies, plasma spray (PS) [5] and chemical vapor deposition (CVD) [10] have been proposed in providing tungsten armor on molybdenum. However, tungsten coatings prepared by PS always display high porosity and micro-cracks [11] and comparatively low strength of adhesion to the substrate. CVD technology also has some disadvantages, such as high cost and complicated procedures. Comparative analysis of the considered of obtaining tungsten coatings shows that electro-deposition is rather promising and attractive. This method can be used for preparing pore-free coatings on various materials with complex extended surfaces. The process also requires no costly and complex equipments [12]. However, it has been observed that tungsten cannot be electrodeposited individually from aqueous electrolyte [13]. Research on electroplating tungsten coatings from molten salt developed since 1960s [14], and had obtained some considerable achievements so far. Pavlovskii electrodeposited metallic tungsten on molybdenum bar from chloride–fluoride–WO_3 melts at 1113–1193 K [15]. Nitta et al. successfully obtained smooth tungsten film from a Li_2WO_4–Na_2WO_4–K_2WO_4–LiCl–NaCl–KCl melt by addition of KF at 873 K [16]. It was also noteworthy that all the electroplating processes mentioned above were conducted in inert atmosphere which would increase the complexity of the electroplating equipment.

The aim of the present paper was to study the tungsten coating on the molybdenum substrate obtained by electrodeposition from –Na_2WO_4–WO_3 system. Compared to other salt systems, the Na_2WO_4–WO_3 melt is nonvolatile, no-hygroscopic, chemically stable and easily prepared. The characteristics and the adhesion of the tungsten coating were investigated. The interface between tungsten coating and molybdenum substrate was observed by scanning electron microscopy (SEM). Thermal shock test was conducted to elucidate the durability of the tungsten coating, and the

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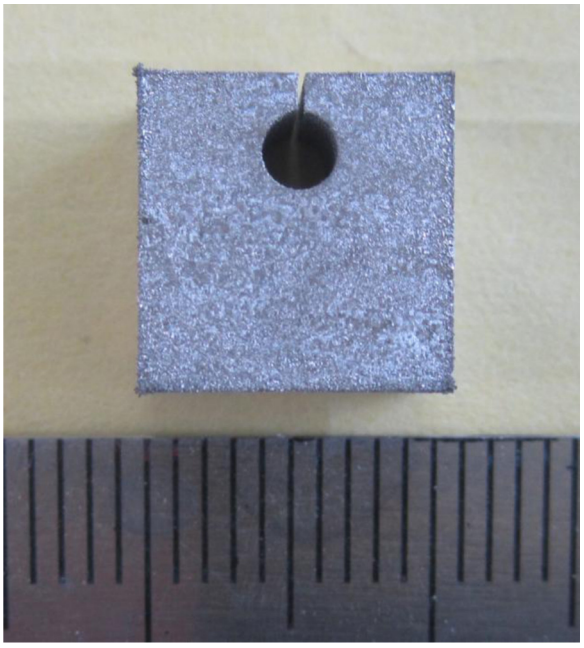

Fig. 1. Appearance of the tungsten coating obtained at 40 mA cm^{-2} for 2 h.

vacuum heating treatment was preformed to investigate the high-temperature diffusibility of tungsten coating on molybdenum.

2. Experiment

All the chemicals used in our experiments were anhydrous reagent grade. Na_2WO_4 and WO_3 (99.5%, Tianjin Fu Chen Chemical Reagents Factory) were dried in a furnace at 773 K for 24 h. The dried chemicals were well mixed into a eutectic composition (Na_2WO_4:WO_3 = 0.6:0.2, by mole ratio [17]) in an alumina crucible (99.9%), and then melt in an electric resistance furnace at 1173 K. The working electrode was a molybdenum plate (purity: 99.95%, 10 mm × 10 mm × 5 mm, TLWM Co., Ltd). A tungsten plate (purity: 99.95%, 15 mm × 15 mm × 5 mm, TLWM Co., Ltd) was employed as a counter electrode. Prior to electrodeposition, the electrodes' surfaces were mechanically polished to obtain high quality surfaces and then cleaned in acetone and distilled water by ultrasonic cleaning. Tungsten coating was electrodeposited on molybdenum substrate from the molten salt in an open bath at the temperature of 1173 K. The electro deposition was carried out at a constant current density of 40 mA cm^{-2} and the deposition duration was 2 h.

The phase and crystal orientation identification of tungsten coating were examined by X-ray diffraction (XRD, Rigaku Industrial Co., Ltd., D/MAX-BB). The surface morphologies of the deposits were characterized by scanning electron microscopy (SEM, JSM 6480LV). The cross section morphologies were observed by scanning electron microscopy (SEM, JSM 6480LV) with line analysis. The strength between the coating and substrate was tested by bending test. The high-temperature diffusibility of tungsten coating on molybdenum was studied by heating under vacuum at 2073 K for 2 h. Archimedes method was applied to measure the density of tungsten coating. Surface roughness of coatings was tested by laser scanning confocal microscope (LSCM, LEXT OLS4000 3D) for four times and the average value was selected. The microhardness of the tungsten coatings was measured according to Vickers microhardness test procedure using microhardness tester (MH-6) with a load of 200 g for 15 s and average of 10 indentations was evaluated. The oxygen content of this tungsten coating was measured by the Nitrogen/Oxygen Analyzer (TC600, LECO, USA).

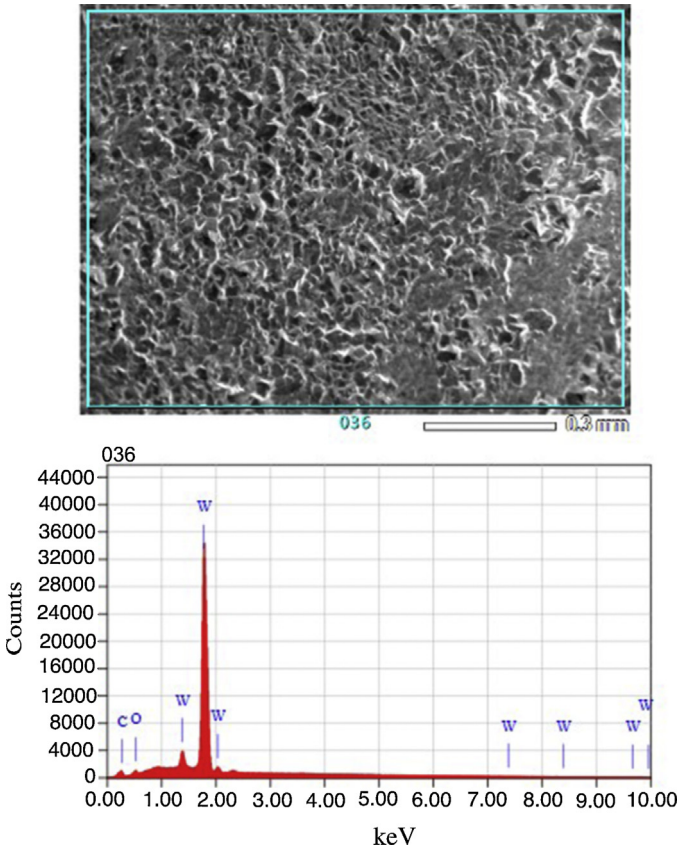

Fig. 2. Surface SEM image with EDS analysis of tungsten coating.

3. Results and discussion

3.1. Crystal structure and microstructure

Figs. 1 and 2 show the appearance and SEM micrographs with EDS analysis of tungsten coating on molybdenum substrate. The silver gray coating is compact and dense without any cracks and voids. The electroplating is an electrochemical reduction and the tungsten coating has been always purified during the electro deposition process. Thus, it can be obtained the tungsten coating with high purity. Fig. 3 shows the electron microprobe scanning curve in the tungsten/molybdenum diffusion layer. It can be inferred that the bonding between the tungsten coating and molybdenum

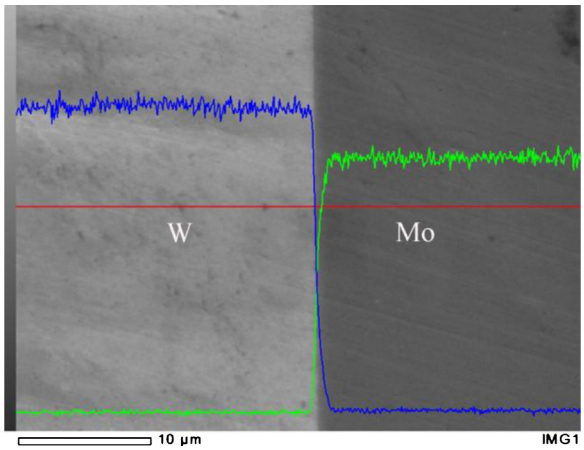

Fig. 3. SEM image with line scanning analysis of the cross section of tungsten coating/molybdenum substrate interface.