



Effects of cathodic current density and temperature on morphology and microstructure of iridium coating prepared by electrodeposition in molten salt under the air atmosphere

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ABSTRACT

Iridium coating was prepared on a Re/C substrate (graphite substrate coated with rhenium coating by CVD) using electrodeposition in molten salt of chlorides in the air atmosphere. The effects of cathodic current density and temperature on both the morphology and microstructure of the coating were studied, respectively. The morphology and orientation of the coating were determined by scanning electron microscope and X-ray diffraction, respectively. The results showed that the cathodic current density and temperature both affected the surface morphology, roughness, grain size and compactness of the iridium coating. With increasing of current density from 5 to 50 mA/cm², the surface roughness of the coating increased from Ra 0.98 to Ra 6.29 μm, while the grain size decreased. With temperature increasing from 520 to 640 °C, the surface roughness of the coating decreased from Ra 4.41 to Ra 1.16 μm, while the grain size increased. The preferred orientations of the coatings changed in the row <111>–<220>–<311> with current density increasing. With the increase of temperature, the preferred orientation of the coating changed from <111> to <220>, and again to <111>. The coating with <111> orientation was smoother and denser than that with <220> orientation regardless of the grain size.

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1. Introduction

The noble metal iridium (Ir), with a high melting point, good chemical stability, low oxygen permeability and vapor pressure, is a promising protective coating for refractory materials in high-temperature oxidizing environment [1,2]. The most successful application of Ir as a coating is the Ultramet's flagship product iridium/rhenium (Ir/Re) combustion chamber [3], which has demonstrated operation at high temperatures (up to 2200 °C) and for long lifetimes (hours). Moreover, Ir is not reactive with carbon below 2280 °C, which makes it a candidate material as a barrier coating for carbonaceous substrates in oxidizing atmospheres at high temperatures. Therefore, the preparation of Ir coating on refractory material substrates has received significant attention in recent years [4–12].

Electrodeposition in molten salt is an ideal one for fabricating uniform Ir coating with high quality and productivity [13–16]. The most favorite electrolyte for the preparation of Ir coating is the nontoxic ternary eutectic molten salt of NaCl–KCl–CsCl. However, almost all of these electrodeposition processes were performed either in inert atmosphere (argon or helium) or in chlorine, which

increased the complexity of the facility for electrodeposition in molten salt. In our previous work [17], a compact and coherent Ir coating was prepared on rhenium (Re) substrate in an open bath in the air by electrodeposition from molten salt of chlorides.

In general, the properties of the coating depend on its microstructure and morphology. For example, the thermal stability of the coating is related to its grain type and size, and the orientation of the coating affects its oxygen diffusion resistance [18]. Thus, the effects of the deposition conditions on the coating microstructure and morphology attract much attention all the time. The research of Saltykova et al. [19] in the above molten salt under inert or chlorine atmosphere showed that Ir deposits were obtained in the form of compact and coherent layers at 500–650 °C, while in the form of dendrites at higher temperatures. The change of the atmosphere above the molten salt from the inert gas to chlorine resulted in the decreasing of both the grain size and surface roughness, moreover, the preferred orientation of the coating transferred from <111> to <110>. The increase of the cathodic current density increased the surface roughness but decreased the grain size.

A great progress in the investigation of electrodeposition conditions of Ir coating in molten salt under inert and chloride atmosphere has been made. Whereas there have been few reports on the relevant research in the atmosphere of air. In the present paper, the effects of the deposition conditions including temperature and cathodic current density on the surface morphology

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Table 1
Electrodeposition parameters of Ir coating.^a

Experimental serial number	1	2	3	4	5	6	7
Current density (mA/cm ²)	5	20	50	100	20	20	20
Temperature (°C)	580	580	580	580	520	640	700

^a Deposition time was 1 h for all experiments.

and microstructure of Ir coating prepared by electrodeposition in molten salt in the air were investigated.

2. Experimental

2.1. Preparation of substrate

A graphite planchet ($\Phi 21 \text{ mm} \times 3 \text{ mm}$) coated with Re produced by chemical vapor deposition (CVD) was used as the substrate (so-called Re/C). Prior to the CVD process, the graphite planchet was polished using metallographic abrasive paper, cleaned in acetone by ultrasonic cleaning and then dried in an oven for 20 min. The Re coating was prepared by thermal decomposition of ReCl_5 produced by in situ chlorination of Re powder (purity: 99.999%, ZHU ZHOU KETE INDUSTRIES CO., LTD). The detailed description of the home-made CVD device was reported in the previous investigation [20]. The CVD conditions were as follows: chlorination temperature 730 °C; deposition temperature 1130 °C; flow rate of Chlorine (purity: 99.999%) 90 mL/min; flow rate of Argon (purity: 99.999%) 500 mL/min; total pressure 20 kPa and deposition time 1 h.

2.2. Preparation of Ir coating

The Re/C planchet and a graphite plate ($50 \text{ mm} \times 40 \text{ mm} \times 5 \text{ mm}$) were used as the cathode and anode, respectively. The cathode sample, which was connected to the carbon fiber, was overall immersed in the molten salt, while the anode plate, tied to iron chrome alloy silk, was partly dipped in the electrolyte with the designed dipping area of about 25 cm^2 . During the electrodeposition, the cathode sample was rotated 180° periodically (about every 10 min) to make the electric field uniformly distributed on both side of the cathode surface as far as possible. The electrolyte salts were dried in a vacuum drying chamber at 150 °C for 5 h before being mixed.

Ir coating was electrodeposited on the Re/C substrate in a ternary eutectic molten salt of NaCl–KCl–CsCl (29.8, 29.8 and 40.4 mol.%, respectively) containing 1.9 mol.% Ir ion at 520–700 °C in the air atmosphere in an open bath. The cathodic current density was varied from 5 to 100 mA/cm². The Ir ions were introduced by adding iridium trichloride (purity: 99.99%, SHAANXI KAIDA CHEMICAL ENGINEERING CO., LTD) directly into the ternary molten salt

solvent at room temperature. The detailed parameters of the experiment are listed in Table 1.

2.3. Characterization

The surface and fracture cross section morphologies of Re and Ir coatings were observed by Hitachi S-4800 scanning electron microscope (SEM). The phase identification and growth direction of the coatings were determined by X-ray diffraction (XRD, Rigaku D/Max 2550VB⁺) using Ni-filtered $\text{Cu K}\alpha$ radiation at a scanning rate of 5°/min. The surface roughness measurement of the coatings was conducted using a Form Talysurf PGI 1240 type profilometer (Taylor Hobson Ltd.) with stylus radius of 2 μm . The result of surface roughness R_a was the mean value of 9 measurements performed along 3 random directions on the coating surface. The cathodic current efficiencies under different deposition conditions were calculated according to the following equation:

$$\eta_e = \frac{m_{mea}}{m_{cal}} 100\% \quad (1)$$

where η_e is the cathodic current efficiency, m_{mea} is the measured Ir coating weight (change of the substrate weight before and after the experiment) and m_{cal} is the calculated Ir coating weight, which can be calculated by the following equation:

$$m_{cal} = \frac{jtsm_{Ir}}{ze_0} \quad (2)$$

where j is the cathodic current density, t is the electrodeposition time, s is the cathode surface area, m_{Ir} is the Ir atomic weight, z is the valency of the deposited ion and e_0 is the elementary electric charge.

3. Results and discussion

3.1. CVD Re coating

The SEM micrographs of the surface and fracture cross section of the Re coating prepared by CVD are shown in Fig. 1. The Re coating was composed of columnar grains with the average diameter of about 20 μm throughout the coating thickness (about 30 μm), and had a flat topped tower-like surface, which is the type topography of Re coatings obtained at low deposition pressures [20]. It could be found in the XRD pattern of Re coating (Fig. 2) that the coating

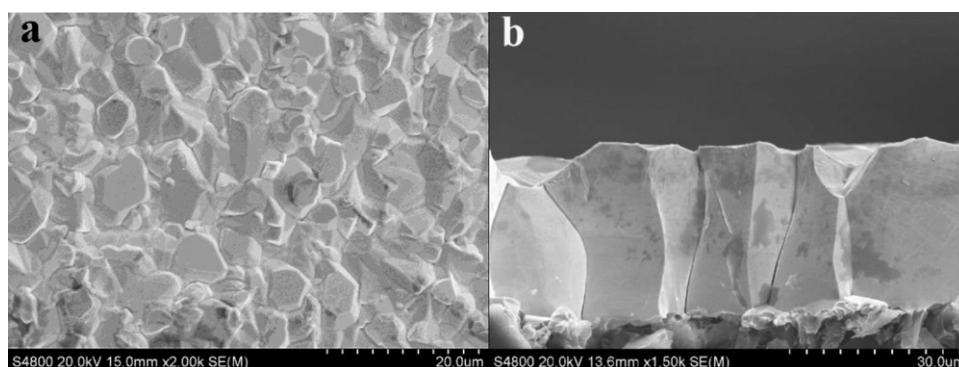


Fig. 1. SEM micrographs of the surface and fracture cross section of the CVD Re coating.

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