

Long-term high-temperature oxidation of iridium coated rhenium by electrical resistance heating method



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ARTICLE INFO

Article history:

Received 3 November 2013

Accepted 24 January 2014

Available online 31 January 2014

Keywords:

Iridium

Rhenium

Oxidation

Resistance heating

High temperature

ABSTRACT

A continuous and compact iridium (Ir) coating with a thickness of ~100 μm was electrodeposited on a rhenium (Re) rod in molten salt at 580 °C for 4 h. The oxidation resistance and failure mechanism of the Ir coated Re (Ir/Re) material were investigated by resistance heating method at 2000 °C in air till the Ir coating failed. The results showed that the lifetime of the Ir/Re rod oxidized at 2000 °C in air was 183 min. After high-temperature oxidation, except for the failure position, the Ir coating in most of the heated regions kept dense and exhibited excellent adhesion on the substrate, with smooth surface and large grain size. The preferred orientation of the Ir coating changed from <220> to <111> after oxidation test. From the end to the center of the as-oxidized Ir/Re sample, the Ir coating became thinner, and the diffusion layer between Ir and Re got thicker. Meanwhile, the preferential oxidation of grain boundaries of Ir coating was more and more severe. It was found that the lifetime of the Ir/Re material in high-temperature oxidizing environment is closely related to the consumption rates of Ir coating by both the direct oxidation of Ir and the diffusion of Re into Ir coating. Based on the diffusion and oxidation kinetics of Re and Ir, the lifetime of the Ir/Re sample in the present study was calculated to be 242 min. The difference between the calculated and real lifetimes can be attributed to the ignored fact that Re diffuses rapidly along the grain boundaries of Ir coating in the calculation.

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1. Introduction

Refractory metal rhenium (Re) has attracted considerable attention as a high-temperature structural material because it exhibits excellent ductility, superior high-temperature tensile strength and creep rupture strength (up to 2000 °C) [1]. However, the poor resistance to oxygen of Re at elevated temperatures restricts its practical application seriously. Iridium (Ir) is considered as a promising protective coating material on Re, due to its high melting point (2440 °C), low vapor pressure and oxygen permeability at high temperatures, and good physical and chemical compatibility with Re [2]. Actually, an Ir coated Re (Ir/Re) thruster chamber has been successfully used in an on-orbit communication satellite (Hughes 601HP), which demonstrates the excellent compatibility and reliability of the Ir/Re material system in high-temperature oxidizing environment [3,4].

Various methods have been used to prepare Ir coatings on refractory materials, such as metal organic chemical vapor deposition (MOCVD) [5–8], magnetron sputtering (MS) [9–12], pulsed laser deposition (PLD) [13–15], double glow plasma (DGP) [16,17] and electrodeposition in molten chloride salt (ED) [18–20]. Among these methods, ED is an ideal one for fabricating uniform and dense Ir coatings on the complex-shaped components with high quality and productivity

[21,22]. Furthermore, it has been reported that the manufacturing cost of the Ir/Re thruster chamber was reduced by 30% using ED process compared with the traditional CVD method, without compromise of the Ir coating quality. Hence, the National Aeronautics and Space Administration (NASA) has selected ED instead of the mature CVD to fabricate the Ir/Re thruster chamber for a new developed rocket engine [23], which demonstrates that ED is a very promising method for preparing the Ir coating.

Previous researchers have investigated the effects of various parameters of ED process on the morphology and microstructure of Ir coating, and determined some properties of the Ir coating, such as density, purity and hardness [22,24–27]. However, the studies related to the high-temperature oxidation resistance of ED-Ir coating on Re substrate are very limited. Smirnov [28] conducted the research on high-temperature (1950 °C) mutual diffusion in the couple of ED-Ir and ED-Re in vacuum, and obtained the interdiffusion coefficient of Ir–Re couple at 1950 °C. Bai et al. [29] studied the diffusion behavior in the couple of ED-Ir and CVD-Re at 1800–2200 °C in vacuum, and estimated the lifetime of ED-Ir coating on Re substrate at 2200 °C only considering the coating degradation resulting from the diffusion of Re into Ir. Etenko [18] performed the oxidation test of ED-Ir coating, which was peeled from the substrate, at 1300 °C and 1400 °C in stagnant air and obtained the oxidation recession rates of the Ir coating at corresponding temperatures. However, the Ir/Re thruster chambers commonly operate at temperatures of 1800–2200 °C. The oxidation resistance and failure mechanism of ED-Ir coated Re should

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be investigated at a temperature close to its service temperatures. Therefore, in the present work, an Ir coating was electrodeposited on a Re rod and heated up to 2000 °C by electrical resistance heating method in stagnant air. The oxidation test was carried out continuously until the Ir coating failed, so as to figure out the failure mechanism of the ED-Ir coating on Re substrate in high-temperature oxidizing environment.

2. Experimental

2.1. Preparation of Ir coating

The apparatus for electrodeposition of Ir coating under inert atmosphere protection has been previously described [30]. A Re rod (Φ 5 mm \times 70 mm, relative density > 99%) fabricated by hot isostatic pressing (HIP) of high-purity Re powder (99.99%) was used as the cathode. Before electrodeposition, the Re rod was polished using metallographic abrasive paper, cleaned in acetone by ultrasonic cleaning and then dried in an oven for 20 min. The Re rod, connected to a carbon fiber, was completely immersed into the molten salt during electrodeposition. A cylinder graphite crucible (Φ 70 mm \times 130 mm) storing the electrolyte salts was used as the anode, which was degassed at 1500 °C for 1 h in a vacuum furnace before used. The salts used for making electrolyte bath were dried at 150 °C in a vacuum chamber for 5 h before being mixed and melted. Ir coating was electrodeposited on the Re rod 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 580 °C under the argon atmosphere. The Ir ions were introduced by adding solid iridium trichloride (purity: 99.99%, Shaanxi Kaida Chemical Engineering Co., Ltd) directly into the ternary molten salt bath. The Ir coating was deposited at current density of 25 mA/cm² for 4 h.

2.2. Oxidation test

The schematic diagram in Fig. 1 shows the set-up of the oxidation test. The Ir/Re rod was firmly connected to the two water-cooled copper electrodes and resistance heated when the current flow through the rod. The clamping length on both sides of the sample was \sim 8 mm. The temperature at the sample center was monitored by a two-color infrared pyrometer (Raytek MR1SCCF), which had been calibrated using a high-temperature vacuum furnace equipped with a W–Re thermocouple. The measured temperature was fed back to an automatic

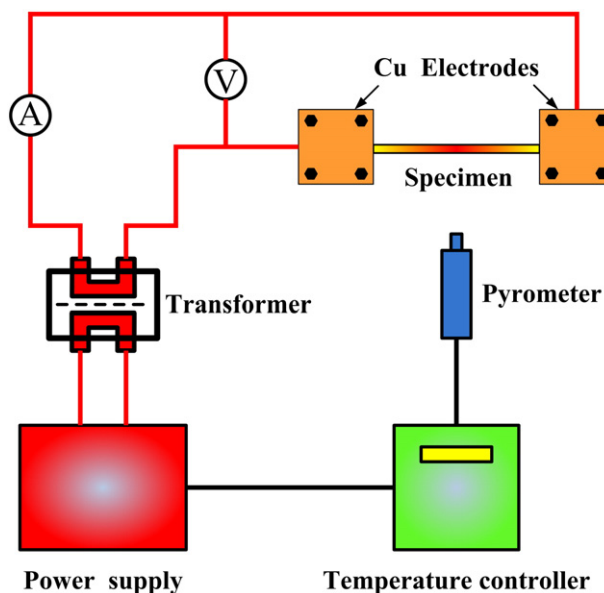


Fig. 1. Schematic diagram of the oxidation testing set-up.

temperature controller, which was used to adjust the current output and control the temperature. The isothermal oxidation of the sample was carried out in stagnant air at 2000 ± 5 °C, and stopped as soon as the failure of the Ir coating was observed.

2.3. Characterization

The morphologies of the Ir coating before and after oxidation were observed by a FEI Quanta 200F environmental scanning electron microscope (ESEM). The compositions of the oxidation products of Ir coating and Re substrate were qualitatively examined by an X-ray energy dispersive spectrometer (EDS) equipped to the ESEM. The phase identifications and growth directions of the Ir coating before and after oxidation were examined by X-ray diffraction (XRD, Rigaku D/Max 2550VB⁺) using Ni-filtered Cu K α radiation at a scanning rate of 5°/min and 2 θ angle from 30° to 85°.

3. Results and discussion

The oxidation test of the Ir/Re rod at 2000 °C lasted 183 min before the Ir coating failed. Fig. 2 shows the macrographs of the Re rod, as-prepared Ir/Re rod and as-oxidized Ir/Re rod. The as-deposited Ir coating was silver-white, continuous and compact in appearance, with an average thickness of 98.5 μ m, which was derived from the weight gain of the sample after the deposition process. After high-temperature oxidation, the Ir coating in heated region of the sample, with a length of \sim 50 mm, was much smoother, shining like a mirror (see Fig. 2(c)). The failure of Ir coating occurred in the very central region (\sim 10 mm wide) of the sample, where there was the maximum temperature during the oxidation test. A narrow dark “ring” was observed at the boundary between heated and unheated regions of the sample, which was presumably composed of the oxides of Ir. The as-oxidized sample was divided into 4 regions (A to D) from the end to the center as shown in Fig. 2(c) for study purpose. The morphologies of as-oxidized Ir coating were observed at midpoints of regions A, B and C. In the region D, besides the failure position, the observation was also conducted at area closely adjacent to the failure position in order to determine the interdiffusion phenomenon between Ir and Re.

The temperature distribution along the Ir/Re rod from the center ($X = 0$) to the end ($X = 35$ mm) during the oxidation test is shown in Fig. 3. It was found that the temperature gradient along the Ir/Re rod was quite high. The temperature decreased rapidly from \sim 2000 °C at the midpoint ($X = 0$) to \sim 1200 °C in the area near the A/B boundary in region B ($X = 23$ mm). The temperature in the area close to the A/B boundary in region A ($X = 26$ mm) was so low that the pyrometer could not measure it. As the temperature measurement range of the pyrometer is 1100–2300 °C, the temperature in this area was lower than 1100 °C. The temperature of the rod end ($X = 35$ mm) was below 50 °C during the whole test. According to this temperature profile, the temperatures at the observation points, i.e., the midpoints of regions B ($X = 20$ mm), C ($X = 10$ mm) and D ($X = 0$), were 1585 °C, 1898 °C and 2002 °C, respectively.

The SEM images of the heated Ir coating adjacent to the boundary between region A and region B are shown in Fig. 4. The as-oxidized Ir coating adjacent to the boundary had a very flat surface with many “hillocks” uniformly distributed on it. These “hillocks” with an average diameter of \sim 5 μ m contained mainly Ir and O elements, which indicate that solid oxides of Ir were steadily formed in this area. According to the temperature profile along the rod in Fig. 3, the temperature in this area was below 1200 °C. It is known that Ir starts to oxidize and form solid iridium dioxide (IrO₂(s)) in air when the temperature is higher than 400 °C [31]. When the temperature is above 1100 °C, IrO₂(s) decomposes into Ir and oxygen again. The oxidation products of Ir are mainly gaseous IrO₃ (IrO₃(g)) with small amount of IrO₂(g) in the temperature range of 1100–1500 °C, and at even higher temperatures, the amount of IrO₂(g) increases obviously [32,33]. Therefore, the dark “ring” at the

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