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Oxidation of iridium coating on rhenium coated graphite at elevated temperature in stagnated air

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ABSTRACT

Continuous and dense iridium coatings were prepared on the rhenium coated graphite specimens by electrodeposition. The iridium/rhenium coated graphite (Ir/Re/C) specimens were oxidized at elevated temperatures in stagnated air for 3600 s. The purification of the as-prepared Ir coating was higher than about 99.98% with the main impurity elements Si, Al, Fe and Ru. After oxidation, the Ir/Re/C specimens kept integrity without significant failures and the average oxidation rate was about 0.219 mg/(cm^2 min). Pores were found at the grain boundaries and concentrated to penetrating holes with the growth of Ir grains, which resulted in disastrous failures of the Ir coating.

1. Introduction

Carbon structural materials, such as carbon/carbon composites and graphite, have superior characteristics of low density, high thermal conductivity, good thermal shock resistance and high temperature strength [1], which meet requirements of severe operational environment applications for high temperature about 2000 °C. However, the rapid oxidation of carbon structural materials is unavoidable when the temperature is higher than 400 °C in the oxidized environment, which severely restricts its high-temperature applications [2].

Iridium (Ir) is considered as promising candidate for oxidation resistant materials at elevated temperature due to its high melting point (2430 °C), good chemical stability, low oxygen permeability, good chemical compatibility and low carbon solubility below the eutectic temperature of 2100–2300 °C [3,4]. Contemporary engineering design requirements for ultra-high temperature structural materials have stimulated interest in Ir coating for carbon structural materials [5]. Good adherence, dense and homogeneous Ir coating on carbon materials will result in elimination of most of the problems such as erosion and oxidation at high temperature. Ir coated C/C composite combustion chambers taking advantage of its high melting point, low density, high mechanical properties and good oxidation resistance at elevated temperature have been used for liquid rocket engines, leading edges and noses of hypersonic aircrafts [6,7].

Ir coatings have been prepared by many kinds of methods, such as metal organic chemical vapor deposition (MOCVD), sputtering deposition (SP), double-glow plasma deposition (DPD) and electrodeposition (ED) [8–15]. Among these methods, ED is considered as one of the most efficient and high-quality methods for fabricating continuous and dense Ir coating on complex-shaped components, because of its low cost, high deposition rate and high purity of iridium coating [16,17]. Much attention has been paid to the effects of various parameters of ED process on the morphology and microstructure of iridium coating, and determined some properties of Ir coating, such as density, purity and hardness. Knowledge about oxidation resistance at high temperature of Ir coating is crucially important for applications of this coating. Etenko [18] performed the oxidation test of ED-Ir coating at 1300 °C and 1400 °C in stagnant air and the Ir coating was peeled from the substrate. Zhu et al. [19] studied that the life time of the ED-Ir coating with a thickness of ~100 μm on rhenium rod oxidized at 2000 °C in air was about 183 min. It was found that the life time of the Ir/Re material was closely related to the consumption rates of Ir coating by both the direct oxidation of Ir and the diffusion of Re into Ir coating. Bao et al. [20] studied Ir–Al alloys exhibited good oxidation resistance at 1100 °C in stagnated air. Zhu et al. [21] investigated that the Ir–Al coating prepared by pack cementation exhibited superior oxidation resistance oxidized at 1900 °C in stagnated air. However,

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researches about oxidation resistance at elevated temperature and its failure mechanism of ED-Ir coating on carbon materials were very limited.

In the present work, Ir coatings were prepared on the Re coated graphite (Re/C) by ED. The Ir/Re/C specimens were heat treated (in argon atmosphere) and oxidized (in stagnated air) at elevated temperature for 3600 s by electromagnetic induction heating method, respectively. Morphologies, structure and composition of the as-prepared, heat treated and as-oxidized Ir coating were investigated. The oxidation recession rate and failure mechanism of the Ir coating was estimated and discussed.

2. Experimental

2.1. Preparation of Ir coating

The apparatus for electrodeposition of Ir coating under inert atmosphere protection has been previously described [22]. Re coated graphite specimens (8710, SGL Carbon Company, Germany) were used as substrates. The Re coating on graphite specimen was prepared by chemical vapor deposition (CVD). The details of the CVD process were reported in our previous work [23,24]. Before electrodeposition, to remove entrapped gases and impurity from the substrates, the Re/C specimens were carefully ultrasonically cleaned in acetone and heat-treated at 1300 °C for 10 min in argon atmosphere by an induction heater. The Re/C specimens used as the cathode and were immersed into the molten salt during electrodeposition. A cylinder graphite crucible (Φ90mm × 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. A ternary eutectic molten salt of NaCl–KCl–CsCl (29.8, 29.8 and 40.4 mol%, respectively) used for making electrolyte bath were dried at 150 °C in a vacuum chamber for 5 h before being mixed and melted. Ir ions were introduced by adding $IrCl_3$ (purity: 99.99%, Shaanxi Kaida Chemical Engineering Co., Ltd.) directly into the melting salts. The electrodeposition parameters are listed in Table 1.

2.2. Heat treatment

The Ir/Re/C specimens were annealed at 1800 °C in a quartz tube at argon atmosphere for 3600 s by induction heater.

Table 1
Electrodeposition parameters of iridium coating.

Deposition temperature	500–700 °C
Content of Ir^{3+}	1.9 at.%
Atmosphere	Air
Cathodic current density	25 mA/cm^2
Growth rate	~25 μm/h
Deposition time	1–2 h

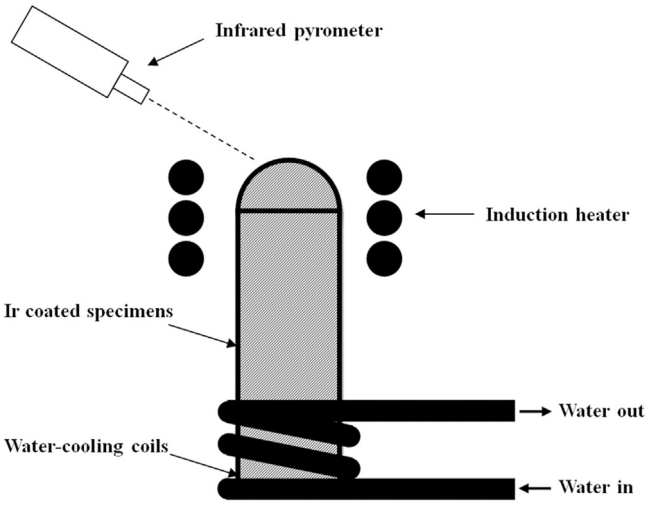

Fig. 1. Devices of oxidation test.

2.3. Oxidation

The schematic diagram of the oxidation test used in this work is shown in Fig. 1. The bottom of the Ir/Re/C specimen was closely intertwined by water-cooling copper coils. The specimen was firmly clamped to brandreth table. The induction heater (XG-25, Zheng Zhou Ke Chuang electronics Co., Ltd.) with a four-circle induction coil was used to heat the specimen during oxidation test. Temperature distribution of the specimen during oxidation test was monitored by a two-color infrared pyrometer (Raytek MR1SCCF, USA), which had been calibrated using a high-temperature vacuum furnace equipped with a W–Re thermocouple. The oxidation temperature was manually controlled by

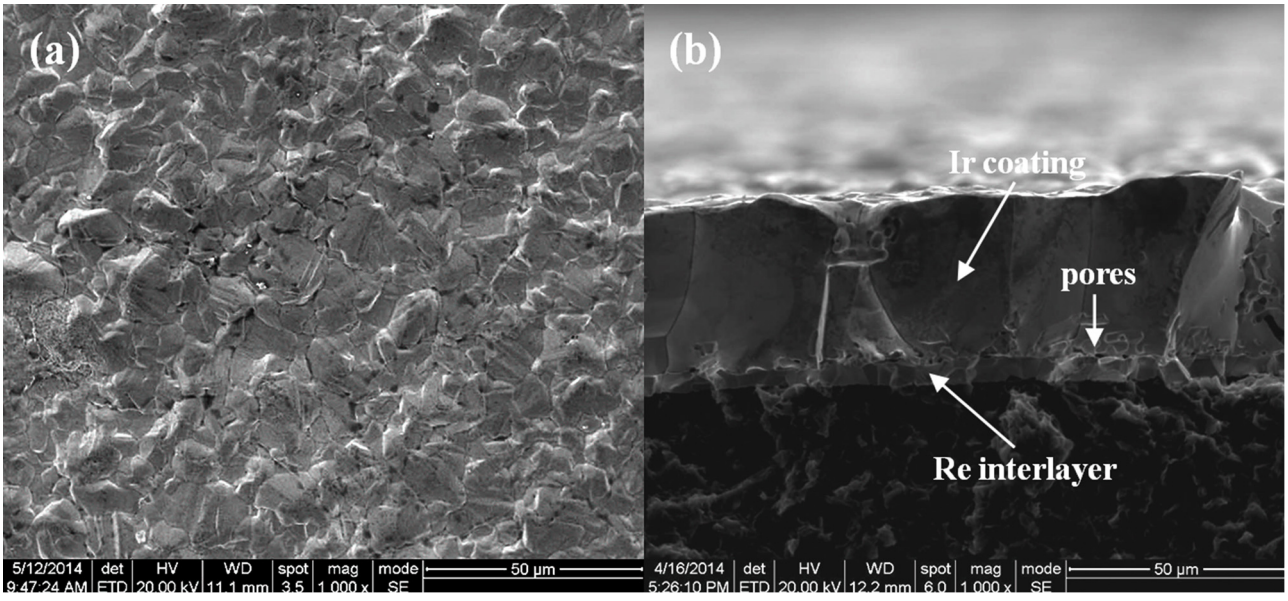

Fig. 2. Morphologies of the as-prepared Ir coating: (a) surface and (b) fracture face.