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Microstructure and evolution of iridium coating on the C/C composites ablated by oxyacetylene torch

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ABSTRACT

Dense and adhesive iridium coating was obtained on the carbon/carbon composites by double glow plasma. The coating was ablated by an oxyacetylene torch to determine the stability of the coating at 2000 °C. Although some pores and the rumpling appeared on the surface after oxyacetylene flame, the integrality and adhesion of the coating kept up. The rumpling could be attributed to plastic deformation of iridium coating which resulted from expansion or contraction mismatch of the coating and the substrate. The ablation results indicated that iridium coating by double glow plasma could afford the high temperature up to 2000 °C and protect the carbon/carbon composites from quick oxidizing.

1. Introduction

Carbon/carbon (C/C) composites have received much attention due to their excellent high-temperature strength, high thermal conductivity, low coefficient of thermal expansion (CTE), good thermal shock resistance and good ablation resistance [1,2]. However, the poor oxidation resistance of the C/C, which readily oxidizes at 500 °C, restricts its high temperature applications. The method, accepted as the most feasible way to protect C/C composites from oxidation, involves a coating of the surface of the C/C with appropriate refractory materials in order to prevent oxygen attacking the substrate. Iridium (Ir) is a candidate material as a barrier coating for carbonaceous substrates due to its high melting point, low oxygen permeability, excellent chemical stability, non-reactive with carbon up to 2280 °C, effective barrier to carbon diffusion and chemical compatibility with carbon [3–5]. For the expensive price and important role, the preparation method of Ir coating has received great concern. Magnetron sputtering, electro-chemical deposition, metalorganic chemical vapor deposition (MOCVD), laser-induced chemical decomposition (LICD) and double glow plasma (DGP) processes have prepared Ir coating [1,6–11]. Ultramet's 100% dense and ductile Ir coatings by CVD could provide tens of hours of useful operation in high stress environment such as liquid rocket motor [12]. Although MOCVD Ir coating was applied to graphite and carbon composite, the coating became porous when exposed to air at high temperature due to carbon content in the coating arising from decomposition of the organic ligand [13]. Not only was the precursor of MOCVD Ir coating very expensive, but also the yield of MOCVD Ir coating was very low. Magnetron sputtering could produce the impurity-free coating which was too thin to afford the long-term high temperature. LICD could produce a thick Ir coating, but the coating was not uniform. After oxidation test of LICD Ir coating in air at 650 °C, cracks and pores presented on the surface of the coating [1]. The electrodepositing by the fused cyanide always caused the rupture of Ir coating at subsequent heating because of the cyanide entrapment during deposition. Ir coating by electro-chemical deposition on the graphite was oxidized at ~2000 °C in air, the result indicated that the amount of

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CO_2 generated during oxidation was proportional to the porosity within the coating [10]. DGP technology could apply almost all solid metallic elements to realize surface alloying of substrates [14]. The advantages of DGP technology include high deposition rate, good coating uniformity, controllability of coating thickness and strong adhesion to the complex shaped substrate. However, few of the literature discussed the ablation property of Ir coating prepared by DGP on the C/C composites. The purpose of the present paper is to investigate the morphology and microstructure of the ablated Ir coating on the C/C composites.

2. Experimental procedure

2.1. Preparation of the coating

A detailed description of the preparation of the coating and schematic diagram of DGP system were reported elsewhere [15,16]. An Ir plate (purity: 99.95%, ∅ 50 mm×3.5 mm) was used as the target material. Some C/C composites (16 mm×16 mm×4 mm) were used as the substrates. Argon (purity: 99.99%) was used as the working gas. The C/C specimens were re-polished by metallographic abrasive paper (5–20 μm) and then using the 1 μm diamond media. At the beginning of the deposition, the substrate cathode power was turned on, a glow was discharged to heat and clean the surface of C/C substrates. After cleaning, the target cathode power was turned on, the coating began to deposit. The deposition conditions were: base pressure 4×10^{-4} Pa, substrate temperature 750–850 °C (altered), Ir target bias voltage −800 V, the C/C substrate bias voltage −300 V, working pressure 30 Pa, target-substrate spacing 15 mm, deposition time 2 h.

2.2. Ablation experiment

The as-coated specimen was exposed to air for 40 s in flowing oxyacetylene torch environment. The specimen was laid on the surface of the alumina firebrick. The oxygen volume flow and acetylene volume flow were 0.24 and 0.18 L/s, respectively. Oxygen pressure and acetylene pressure were 0.4 and 0.095 MPa, respectively. The distance between the oxyacetylene torch and specimen was ∼20 mm. The nozzle tip diameter of the oxyacetylene torch was 2 mm. The surface temperature of the specimen was about 2000 °C, which was monitored with an optical pyrometer.

2.3. Specimens characterization

The morphology and microstructure of the coating were examined by a scanning electron microscopy (SEM, FEI CO., Quanta200). Phase identification and crystallization evaluation were determined with X-ray diffraction (XRD, D8Advance) using Ni-filtered Cu Ka radiation at a scanning rate of 0.2°/s and 0.5°/s, and scanning from 20° to 100° of 2θ.

3. Results and discussions

Fig. 1 shows the SEM micrograph of the surface of the C/C substrate. It was found that the carbon filaments were surrounded by the laminative pyrolytic carbon. Some microcracks and microgaps presented on the polished C/C composites. Despite very careful polishing, it was difficult to produce a smooth surface on C/C composites for the brittleness. Because the random structure is sensitive to the microstructure and physical property of the deposited coating, Ir coating on C/C composites must have a good surface coverage.

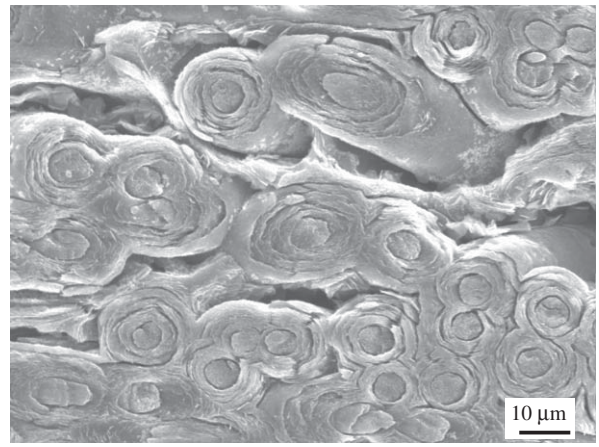

Fig. 1. SEM micrograph of the surface of the C/C substrate.

Fig. 2 shows the macroscopical photographs of the as-coated specimen and the as-ablated specimen. It was found that the successive Ir coatings were obtained on the C/C composites by DGP. Some voids on the surface of the coating were attributed to the intrinsic microstructure of the C/C composites (see Fig. 1). In spite of the defects, all of the voids and grooves were synchronously deposited. The conformal and integrative coatings could result from the continuous bombardment of the plasma and the counter-sputtering of the surface of the substrate during depositing process.

Fig. 3 shows the SEM micrographs of the surface of the as-deposited coating. No microcrack was seen in Fig. 3(a). The coating was composed of fine Ir grains, small particles about 0.2–0.5 μm in size and some aggregates about 1–2 μm in size. Small particles could be proved by Fig. 3(b). Smaller particles about nano-sized could be proved by Fig. 3(c). In general, Ir target was located above C/C substrates in the chamber. Both the sputtered Ir atoms and the sputtered clusters dropped on the surface of the C/C substrate to form the coating during deposition. It indicated that difference of the particles size resulted from the intrinsic characterization of the experimental process.

Fig. 4 shows the XRD patterns of Ir coating before and after ablation. Compared with the standard *d*-values taken from JCPDS Card (no: 43-0144), the as-deposited coating has a polycrystalline structure. The diffraction peak of the as-ablated coating was thinner than the as-deposited coating, which indicated that the as-ablated Ir grains were bigger than the as-deposited Ir grains. The XRD results were consistent with the results of SEM micrograph. Fig. 5 shows the SEM micrograph and EDS spectrum of the cross-section of Ir coating. The coating exhibits good adherence to the C/C substrate. The SEM micrograph in the corner of Fig. 5 is