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Study on graphite surface modification by non-electrode plasma electrolysis

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ABSTRACT

A novel non-electrode plasma electrolysis technique was developed in the paper in order to rapidly prepare a coating on graphite. The coating was mainly composed of SiO_2 with the thickness of ~ 7 μm. A small amount of SiC was also detected in the coating. The coating exhibited superior oxidation resistance at 1000 °C in air, evidenced by the visible polishing marks, smooth surface and the much decreased roughness from ~ 120 μm to ~ 50 μm. The simulation of thermal effects was conducted in order to establish the formation mechanism of the coating on graphite. We believe that the novel non-electrode plasma electrolysis technique will find a wide range of applications in preparing high-performance graphite and its composites.

1. Introduction

The development of industrial techniques has harsher requirements on materials. It requires that the materials have to behave a certain performance. It is imperative to improve the properties of materials. As a kind of high temperature structural material, graphite has been fast developing these years [1–4]. It has excellent performances so the productivity and efficiency are rapidly increased these years [5]. Graphite performances of light weight, high modulus, high strength, high temperature stability and thermal shock resistance make it a good candidate for a high-temperature structural and engineering material [6]. It has been used as refractories, conductive material, thermal shock resistant material at this stage. In the future, the demand for graphite products will continue to grow faster and faster with the rapid development of metallurgical, chemical, mechanical, medical, nuclear, automotive, aerospace and other industries.

However, all these excellent performances can only be kept in the inert atmosphere. Oxidation can easily occur above 643 K and the rate of oxidation can be rapidly increased when the temperature reaches to 843 K, which has greatly limited the extensive application of graphite in oxidizing environments [7]. In addition, graphite is a regular hexagonal structure consisting of carbon atoms with a multi-layer structure. There is only van Edward force between the adjacent layers. The interlayer binding force is pretty weak. Therefore, the layer can be peeled off easily when a shear stress is put on graphite; thus, graphite has poor wear resistance. The abrasion increases the costs of the production. So much attention has been paid to the development of new graphites for the oxidation protection and the improvement of wear resistance these years.

In order to overcome the shortcomings in the practical application for graphite, several countermeasures have been put forward. The Coating technology has become one of the best choices to solve the problem [8–10].

Recently in order to protect graphite, a variety of coatings have been studied about the surface modification, including SiC, SiO_2, SiC/Si/$MoSi_2$. Shirani et al. studied ZrB_2-SiC-WC coating by spark plasma sintering process [11]. This is an expensive and time-consuming coating method. But the binding force between the coating and the graphite substrate is good because of the existence of mesophase. Jiang et al. adopted a new technology named slurry dipping and vapor silicon infiltration to prepare oxidation protective $MoSi_2$-SiC-Si coating [12]. The coating prepared by the method has excellent protective capability when the temperature varies from room temperature to 1875 K in air, and the coating can protect the substrate for up to 353 h at 1773 K. He et al. did the study to get SiC coating by impregnation and pyrolysis of polycarbosilane. In this way a high-purity coating can be got at a low treat temperature [13]. Chen et al. used two-step pack cementation to get SiC/Si/$MoSi_2$ multilayer coatings [1]. Wang et al. made an attempt to get a composite coating to solve the problem [6]. Buffer layer ZrB_2 was prepared by pack cementation while outer layer SiC was prepared by chemical vapor deposition (CVD). The abrasion and oxidation resistant multilayer coatings improved the properties of graphite a lot in this way. However, the method is time-consuming. The thermal stress is easily caused by the difference of thermal expansion coefficient between coating and substrate. Liao et al. prepared SiC coating on the expanded graphite surface by the introduction of anti-oxidation additives [14]. Lee et al. did an experiment on the graphite surface for Korean HCCR TBM by the chemical vapor reaction (CVR) [15]. Kurzeja

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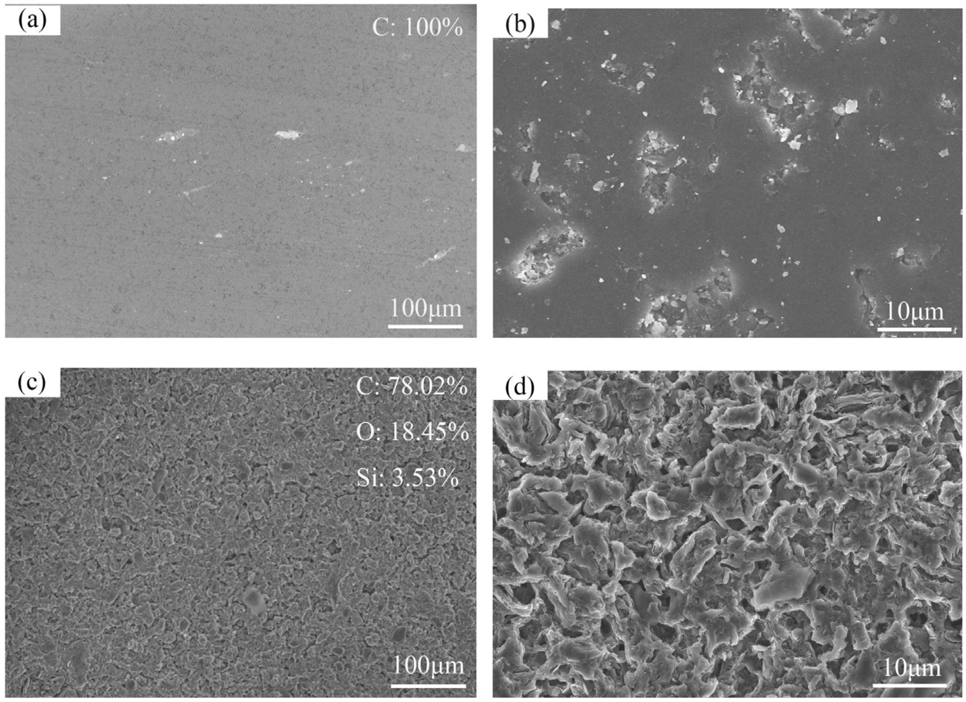

Fig. 1. Surface morphologies of the graphites: (a) (b) bare sample, (c) (d) coating sample. The EDS results were shown in the inset of (a) and (c).

patented a method to fabricate a coating consisting of 15–40% colloidal silica for graphite electrodes to protect the graphite from wearing [16]. Chen et al. adopted CVD to get Al_2O_3-SiO_2 composite coatings on graphite surface [17], the coating was dense, uniform and adherent.

SiO_2 coating is popular these years [18]. The raw material is cheap and the process is simple. Sol-Gel, thermal spraying method, slurry method, chemical liquid deposition and vacuum pressure infiltration are all the technologies preferred by factory in the industrial application field to prepare anti-oxidation coating on the graphite [19–21]. Chen et al. studied SiO_2 coatings on the graphite surface by vacuum pressure infiltration [22]. The method helped to shorten production cycle but the equipment cost is expensive and the operation is complex. Baumgartner et al. tried to manufacture thick SiO_2 coating on carbon fibers using self-assembly process of polyamine mediation [23]. Song et al. studied performance of silica anti-coking coating. They found the coating had the excellent performance while treated with steam at 1000 °C, but that property may be sharply decreased when the temperature rose to 1200 °C.

In this study, a novel non-electrode plasma electrolysis technique was put forward to manufacture SiO_2 coating on the graphite. The technique is a new method that combines liquid chemical deposition with electric discharge machining. Compared with conventional technologies like sol-gel, liquid-phase chemical vapor deposition [24], the new technology has many advantages including simple equipment, time saving, high efficiency and low cost of technology. The microstructure and properties were systematically investigated. The formation mechanism of the SiO_2 coating was also discussed. We believe that the novel non-electrode plasma electrolysis technique will lay a solid foundation for the widely application of graphite and other carbon-based materials.

2. Experimental

2.1. Preparation of coating

The commercialized (11 mm × 3 mm × 3 mm) graphite was polished by different mesh sandpapers of #80, #200, #800 and #1000. Then polished cloth was used to polish the samples with the Al_2O_3 polishing solution to fill the scratches caused by grinding of sandpapers so that the patterns can be flat. So the spacing from the workpiece to electrode could be guaranteed. Then the specimens were cleaned by deionized water to remove the impurities attached to the graphite.

In the non-electrode plasma electrolysis process, the anode and cathode were made of high-purity graphite and copper tube, respectively. Both electrodes were put in Na_2SiO_3 aqueous solution with the concentration of 50 g/L. When a relatively high voltage was applied between the anode and the cathode, a plasma was formed on the surface of the cathode copper tube. The graphite substrate was put in the plasma with the distance of 20 mm near to the cathode copper tube. The applied voltage was ~ 150 V with the processing time of 3 min. After the processing, the graphite substrate was pulled out from the aqueous solution, and then washed using the running water for several times. Finally the graphite substrate was dried in an oven for 10 h at 80 °C.

2.2. Characterization of coating

The surface morphologies of the bare sample and coating samples were observed by a scanning electron microscope (SEM). Then the energy dispersive spectroscopy (EDS) analysis was conducted to obtain the element composition in the coating. The cross-sectional morphology was characterized by SEM and an optical microscope. X-ray photoelectron spectroscopy (XPS) was conducted to obtain the composition of the samples to be measured. The Fourier transform infrared spectroscopy (FT-IR) analysis was carried out to figure out the components in the SiO_2 coating. The three dimensional surface morphology was characterized by a hyperfield 3D optical microscope. The temperature change during non-electrode plasma electrolysis was simulated using the Ansys 14.5 software.

2.3. Oxidation test

The oxidation resistance of the bare and coating graphites was measured in a furnace at 1000 °C for 1 h in air. Firstly, both graphites were put in a furnace, and then heated to 1000 °C at a rate of 10 °C/min. Both graphites were kept at 1000 °C for 1 h, and then cooled down to room temperature at a rate of 15 °C/min.

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