



Carbon foam/hydroxyapatite coating for carbon/carbon composites: Microstructure and biocompatibility



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ABSTRACT

To improve the surface biocompatibility of carbon/carbon composites, a carbon foam/hydroxyapatite coating was applied using a combination method of slurry procedure and ultrasound-assisted electrochemical deposition procedure. The morphology, microstructure and chemical composition of the coating were investigated by scanning electron microscopy, energy dispersive X-ray spectroscopy, Fourier transform infrared spectroscopy, Raman spectroscopy and X-ray diffraction. The biocompatibility of the carbon foam/hydroxyapatite coating was investigated by osteoblast-like MG63 cell culture tests. The results showed that the carbon foam could provide a large number of pores on the surface of carbon/carbon composites. The hydroxyapatite crystals could infiltrate into the pores and form the carbon foam/hydroxyapatite coating. The coating covered the carbon/carbon composites fully and uniformly with slice morphology. The cell response tests showed that the MG63 cells on carbon foam/hydroxyapatite coating had a better cell adhesion and cell proliferation than those on uncoated carbon/carbon composites. The carbon foam/hydroxyapatite coatings were cytocompatible and were beneficial to improve the biocompatibility. The approach presented here may be exploited for fabrication of carbon/carbon composite implant surfaces.

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1. Introduction

The degenerative and inflammatory problems of bone affect millions of people worldwide. These diseases need to be solved by using orthopedic implants to substitute natural bone. Various biomaterials have been used as orthopedic implants such as polymers, metals, ceramics and composites [1,2]. Carbon/carbon (C/C) composites are considered the most promising artificial materials for orthopedic implants owing to their unique properties, such as light weight, high toughness, high specific strength and corrosion resistance. In particular, C/C composites have a more similar elastic modulus to that of natural bone in comparison with metallic implants, which may help to avoid stress shielding and sequential bone absorption [3–5]. However, the surface of C/C composites is biologically inert and fails to form a chemical bond with the host bone.

Applying bioactive coating for C/C composites is a logical choice to improve the bioactivity so as to improve osseointegration with bone tissues. Hydroxyapatite (HA) is the major inorganic component of natural bone. It possesses excellent biocompatibility and

ability to form a chemical bonding with the bone tissue [6–8]. Thus HA has been identified as suitable bioactive coating for orthopedic implants. Many coating techniques are being used to prepare HA coatings, such as plasma spraying [9,10], electrochemical deposition (ECD) [11], sol–gel [12,13], electrophoretic deposition [14] and biomimetic method [15,16]. Among these methods, ECD is simple and inexpensive technique, without needing complex vacuum apparatus. In addition, the ECD procedure can be performed at room temperature. However, the HA coating prepared by the ECD method is prone to form a loose structure, which is mainly caused by the fact that the H₂ gas formed during ECD could adhere itself to the cathode and inhibit the compact deposition of HA coating [17–19]. In our previous work, the ultrasound is introduced into the ECD method to get rid of the adhered H₂ gas, forming a method of ultrasound-assisted electrochemical deposition (UECD). This method offers various advantages over conventional ECD methods, such as improvement in coating hardness and uniformity. However, poor bonding strength between the HA coatings prepared by UECD and C/C composites is still a problem. Zhai et al. reported that the HA coating on C/C composites prepared by UECD has a bonding strength less than 4 MPa [4]. Several researchers have introduced reinforcing phases including collagen [20], SiC nanowire [21] and carbon nanotube [22] into the HA coating to improve the bonding strength. The reinforcing phases could not

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only induce the formation of HA coating with compact structure but also improve the cohesion strength of the coating, thus promoting the bonding strength between the HA coating and C/C composites.

In this work, a novel approach to improve the bonding strength of HA coating is proposed. Carbon foam (CF) which possesses favorable physicochemical compatibility to C/C composites is applied on the surface of C/C composites, which could provide a large number of pores for the deposition of HA crystal. The HA crystals could infiltrate into the pores of CF and form CF/HA coatings on C/C composites. The morphology and microstructure of the CF/HA coating is studied. Moreover, the biocompatibility of the CF/HA coating is assessed by testing the cell morphology, adhesion and proliferation on the coating.

2. Materials and methods

Small specimens (8 mm × 8 mm × 2 mm) were cut from the C/C composite bulk with a density of 1.78 g/cm³. C/C composites used in this work were prepared using the following procedure. Two-dimensional needle-punched carbon fiber felt with apparent density of 0.45 g/cm³ was used as preform, whose architecture was made of continuous long carbon fiber layers laminated alternately with short carbon fiber layers. The long carbon fiber layers were interlaced with a special orientation of 0°/90°/0°/90°. The carbon fibers were polyacrylonitrile (PAN)-based fibers produced by Toray Corporation in Japan. The fibers had a density of 1.72 g/cm³ and a mean diameter of 7 μm. Densification of preforms was carried out in a thermal gradient chemical vapor infiltration (TCVI) furnace. The schematic of the TCVI furnace was shown in Fig. 1. The furnace was vacuumed before deposition procedure. The deposition was performed at 1273 K using natural gas as the precursor gas with a flow rate of 5 m³/h. The moving speed of the thermocouple was controlled around 0.25 mm/h. The specimens were hand-polished with 400 grit SiC paper, ultracleaned in turn by acetone, ethanol and distilled water, and dried at 373 K for 10 h.

The preparation process of the CF/HA coating on C/C composites was shown in Fig. 2. Firstly, a slurry procedure was employed for applying precursor of CF on C/C composites. The precursor was composed of phenolic hollow microspheres and home-made phenolic resins. The phenolic hollow microsphere (BJO-0930) had a density of 0.25 g/cm³ and an average particle size of 51.50 μm. The wall thickness of the microsphere was 1.84 μm. The procedure of preparing the home-made phenolic resins was as follows. Aqueous formaldehyde (37 wt%) and phenol with a molar ratio of 1.3 were placed into a flask equipped with a stirrer. Sodium hydroxide aqueous solution (0.1 mol/L) used as a catalyst was added to the solution with a separating funnel to adjust the pH to 11. The reaction was maintained at 373 K for 3 h with a stirring speed of 800 rpm to obtain the phenolic resins. The phenolic hollow microspheres were mixed to the home-made phenolic resins with the volume fraction of 50%. Using ethanol as dispersant, the phenolic hollow

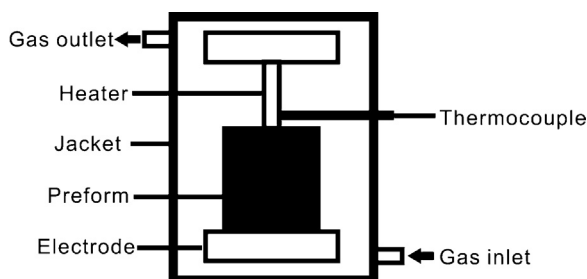


Fig. 1. Schematic of the TCVI furnace.

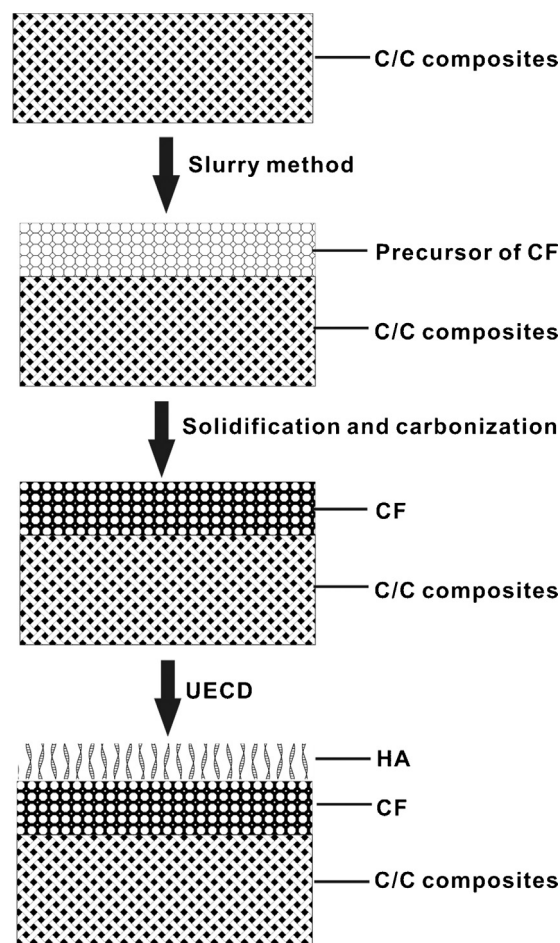


Fig. 2. The preparation process of the CF/HA coating on C/C composites.

microspheres and the phenolic resins were mixed by a magnetic stirring apparatus for 3 h, leading to the formation of slurry. Then the slurry was brushed directly to the surface of C/C composites with the thickness being controlled by brush times. Secondly, a solidification and carbonization process is carried out to obtain the CF. The as-prepared samples were heated at 323 K for 10 h, at 353 K for 3 h and then at 383 K for 10 h. This is followed by a temperature increasing at 20 K/h to 1173 K and held for 3 h. Then the temperature was decreased at 30 K/h to room temperature to obtain the CF on C/C composites. Thirdly, the UECD was carried out on CF modified C/C composites using a two-electrode electrochemistry system to prepare CF/HA coating. The CF modified C/C composites were served as cathode while graphite was acted as anode. The electrolytes were prepared by dissolving $\text{NH}_4\text{H}_2\text{PO}_4$ and $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in distilled water. The concentration of the $\text{NH}_4\text{H}_2\text{PO}_4$ and $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ were 10.0 mmol/L and 16.7 mmol/L, respectively. The pH value was adjusted to 4.5 using dilute HNO_3 and $\text{NH}_3 \cdot \text{H}_2\text{O}$ solution. A constant current of 10 mA was applied at 333 ± 1 K. The deposition times were 1 min and 10 min. Ultrasound was introduced by an emitting device with a frequency of 45 kHz and power of 100 W. After deposition, CF/HA coated C/C composites were washed thoroughly with distilled water. For comparison, the UECD was also performed on C/C composites without CF using same parameters.

The crystalline structure of CF and CF/HA coating was measured with X-ray diffraction (XRD, X'Pert PRO, PANalytical). The morphology and element of CF and CF/HA were analyzed by a scanning electron microscopy (SEM, SUPRA55), equipped with energy

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