Research Article

Improved Surface Wettability of Water by Applying SiC/Ti6Al4V Coatings on Carbon/Carbon Composites

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SiC/Ti6Al4V coatings were applied on carbon/carbon composites to improve the surface wettability of water. SiC interlayers were preprepared by pack cementation to bond both the carbon/carbon composites and the Ti6Al4V, and then the Ti6Al4V coatings were applied by magnetron sputtering technique. The morphology and crystalline of the SiC/Ti6Al4V coatings were analyzed by scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction. The surface wettability of the coatings was tested by video-based contact angle measuring device. The results showed that SiC can serve as an interlayer between the carbon/carbon composites and Ti6Al4V. The SiC/Ti6Al4V coatings covered the carbon/carbon composites uniformly with spherical morphology. The coatings improved the surface wettability of carbon/carbon composites with the contact angle of water decreasing from $85.7 \pm 4.1^\circ$ to $26.5 \pm 0.1^\circ$.

1. Introduction

Carbon/carbon (C/C) composites play an important role in biomedical application because they possess excellent biocompatibility and mechanical properties [1, 2]. Their elastic modulus is close to that of human bones, thus avoiding the bone resorption caused by the effect of stress shielding [3, 4]. The cell culture experiment confirms that the C/C composites are supportive for the cell adhesion and growth [5]. However, poor surface wettability of the C/C composites reduces protein absorption and cell attachment thus reducing the osteoconductivity and reducing bone regeneration. Many studies have been made to improve the surface wettability of C/C composites. Zhao et al. have reported the ultraassisted anodic oxidation treatment for C/C composites to improve the surface wettability. They found that some hydrophilic groups were grafted on the surface of C/C composites and the surface wettability was improved [6]. Xiong et al. have made surface modification to C/C composites by hydrothermal treatment in an autoclave containing ammonium persulfate solution. They found that the percentage of oxygen atoms on the surface of C/C composites was significantly increased [7].

The above-mentioned methods to improve the surface wettability are mainly surface oxidation. In this work, SiC/Ti6Al4V coatings were applied on C/C composites to improve the surface wettability of water. The SiC interlayer was used to bond both the C/C composites and the Ti6Al4V, and the Ti6Al4V was used to improve surface wettability of the C/C composites. The Ti6Al4V has been widely applied to both dental implantology and orthopedic replacement. This is an inert biomaterial with excellent biocompatibility, bone-implant integration, and unique resistance to corrosion.

2. Experimental

Small specimens (8 mm × 8 mm × 2 mm) were cut from bulk two dimensional C/C composites with a density of 1.82 g/cm$^3$ and a porosity of 8.10%. The preform of the C/C composites was needle-punched carbon fiber felt. According to the different characterizations under polarizing microscope, the pyrolytic carbon was classified into three structures, including smooth laminar structure, rough laminar structure, and isotropic structure [8, 9]. In this work, the pyrolytic carbon
shows smooth laminar structures. The specimens were hand-abraded with 400 grit SiC paper, ultracleaned in turn by acetone, ethanol, and distilled water, and dried at 353 K for 5 h. The SEM micrographs of the C/C composites were shown in Figure 1. The surface roughness of the C/C composites was $R_a = 1.6 \pm 0.1 \mu m$.

SiC interlayers were prepared by pack cementation technology. Pack cementation technology is carried out at high temperatures by embedding the substrate in powders composed of depositing material and inert filler. Using this method, the coating can be applied on the substrate through vapor transport and diffusion. The powders for preparing SiC were 60–80 wt% Si, 10–25 wt% graphite, and 5–15 wt% $Al_2O_3$. In this work, alumina is used as sintering aids in the preparation of SiC. The alumina not only can improve density of the SiC but also can decrease the sintering temperature. The specimens and pack powders were put in a graphite crucible and heat-treated at 2173 K for 2 h in an argon protective atmosphere to form the SiC interlayers.

After the preparation of SiC interlayers, Ti6Al4V coatings were prepared on SiC coated C/C composite specimens using an ultrahigh vacuum magnetron sputtering machine. Before deposition, the surface of SiC coated C/C composites was cleaned with Ar+ for 1 h to remove the impurities. The target used in this work was purchased from Baoji Titanium Industry Co., Ltd., China. The parameters of the magnetron sputtering process were listed in Table 1.

<table>
<thead>
<tr>
<th>Distance between the sample and target (mm)</th>
<th>Sputtering power (W)</th>
<th>Ar pressure (Pa)</th>
<th>Sputtering time (hour)</th>
</tr>
</thead>
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<tr>
<td>60</td>
<td>500</td>
<td>3</td>
<td>5</td>
</tr>
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</table>

The surface morphology and crystalline of the coatings were analyzed by a SUPRA55 scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) and a Philips X’pert PRO X-ray diffraction (XRD).

The surface roughness of the SiC interlayer was tested by a Lasertec CI30 laser confocal scanning microscope. The two dimensional surface profiles were recorded. To obtain the average surface roughness of $R_a$, measurements were performed on five samples and five positions were tested for each sample.

Surface wettability of C/C composite specimens and the SiC/Ti6Al4V coatings were analyzed using a video-based contact angle measuring device (Dataphysics OCA20) by testing the contact angle of distilled water. Six measurements were taken for each specimen and an average contact angle was calculated. The tested data were presented as the mean and standard deviation. The Student’s $t$-test was applied for analytical statistics [10, 11]. Differences between each sample were considered statistically significant at a $P$ value of $<0.05$.

### 3. Results and Discussion

Figure 2 shows XRD pattern of interlayer prepared by pack cementation process. It shows the diffraction peaks of $\alpha$-SiC, $\beta$-SiC, and Si. The strong peaks for $\alpha$-SiC are observed, suggesting that the interlayers are mainly composed of $\alpha$-SiC phase.

Figure 3 shows the SEM micrograph and surface roughness curve of the SiC interlayers. The SiC interlayers cover the surface of C/C composites entirely with a dense morphology without visible cracks. The surface of the SiC interlayers has rough morphology. The surface roughness curve demonstrates that the surface profile of the SiC interlayers fluctuates around the datum line with large fluctuation amplitude. The average surface roughness value of the SiC interlayer is $R_a = 2.6 \pm 0.2 \mu m$, which is higher than the surface roughness of uncoated C/C composites ($R_a = 1.6 \pm 0.1 \mu m$) [12]. The rough surface of the SiC interlayers may favor the bonding to the following Ti6Al4V coatings.

Figure 4 shows the XRD pattern, EDS result, and SEM micrograph of Ti6Al4V coatings prepared by magnetron sputtering.
sputtering. The XRD pattern shows that the coatings are composed of hexagonal closed-packed (hcp) \( \alpha \)-Ti phase. The EDS result shows that the coatings are composed of Ti element, Al element, and V element. It also confirms the successful formation of the Ti6Al4V coatings. The SEM micrograph shows that the Ti6Al4V coatings fully cover the
SiC interlayer coated C/C composites. The deposition of the Ti6Al4V coatings is primarily following the contours of the SiC interlayers.

Figure 5 shows the cross-section micrograph and EDS result of the SiC/Ti6Al4V coatings. The cross-section micrograph shows that the thickness of the coatings is about 130 μm. The SiC interlayers may be formed by the reaction of Si with the graphite powder or Si with the C on the surface of C/C composites. The SiC interlayer may bond to the C/C composites by chemical reaction of Si with the C on the surface of the C/C composites. The EDS result confirms the infiltration of SiC into the inside of C/C composites. In addition, the SiC interlayer also forms a bonding to the Ti6Al4V coatings tightly due to its rough surface morphology. No obvious gap between the SiC interlayer and Ti6Al4V coatings is observed.

Figure 6 shows the photos and values of contact angle toward distilled water for uncoated C/C composites and the SiC/Ti6Al4V coatings. The extension of the distilled water on SiC/Ti6Al4V coatings is better than that on uncoated C/C composites. The contact angles of water are 26.5 ± 0.1° for SiC/Ti6Al4V coatings and 85.7 ± 4.1° for uncoated C/C composites. The significant decrease of contact angles is found between the uncoated C/C composites and SiC/Ti6Al4V coatings ($P < 0.001$), suggesting a remarkably improvement of surface wettability of water. The water contact angle is the angle where a water/vapor interface meets a solid surface. It quantifies the wettability of a solid surface by water. As the tendency of a water drop to spread out over a solid surface increases, the contact angle decreases. Thus, the contact angle provides an inverse measure of wettability. In the field of biomaterial researches, an improvement in surface wettability of biomaterials can promote cell survival and attachment. Thus, a decreased water contact angle indicated the improved cell response and favorable osteoconducive behavior.

4. Conclusions

SiC/Ti6Al4V coatings were applied on C/C composites by combination of pack cementation and magnetron sputtering.
technique. The SiC can serve as an interlayer between the C/C composites and Ti6Al4V. The SiC/Ti6Al4V coatings significantly improve the surface wettability of C/C composites with the contact angle of water decreasing from 85.7 ± 4.1° to 26.5 ± 0.1°.

Conflict of Interests
All the authors of this paper do not have a direct financial relation with the commercial identity mentioned in this paper that might lead to a conflict of interests for any of the authors.

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