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Addition of Si₃N₄ Strengthens SiC Coatings via Heat Treatment with Nitrogen Gas onto Carbon/Carbon Composites

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Abstract: We report the synthesis of SiC/Si₃N₄ coatings on carbon/carbon composites via pack cementation and heat treatment with nitrogen gas, the latter of which improves the coating wear resistance. X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS) and X-ray photoelectron spectroscopy (XPS) are used to analyse the microstructure, surface morphology, chemical states and elemental distribution, respectively, of SiC and SiC/Si₃N₄ coatings. In addition, we analyse the tribological behaviour of the SiC and SiC/Si₃N₄ coatings and the related microstructure and wear mechanisms. The results show that SiC/Si₃N₄ coatings are compact and contain the three phases: Si₃N₄, SiC and C. Additionally, specimens with the SiC/Si₃N₄ coating have smaller, more stable friction coefficients and less weight loss than specimens with only the SiC coating. Adhesive wear and abrasive wear are the main wear mechanisms contributing to the higher friction coefficient of the SiC coating. Furthermore, with the SiC/Si₃N₄ coating, adhesive wear is the main wear mechanism causing a high friction coefficient at the initial stage of frictional wear. In the subsequent stages of frictional wear, the graphite in the SiC/Si₃N₄ coating generates a thin lubricating film that decreases the friction coefficient.

Keywords: carbon/carbon composites; SiC/Si $_3N_4$ coating; microstructure; tribological behaviour; wear mechanism

1. Introduction

Mechanical seals are extensively used in industry to prevent losses of lubricant or gases and to prevent the entry of contaminants into hydraulic or lubrication circuits. A mechanical seal comprises two parts: a static seal ring and a rotating ring. Many situations employing seals require frequent seal replacement because of wear and surface damage caused by external contaminants in the system. The replacement of seals requires stopping of rotating equipment, which can incur losses to productivity. To address these concerns and to improve the life and energy efficiency of seals, many approaches have been explored worldwide [1]. Carbon/carbon composite (C/C composites) have drawn great interest owing to their excellent mechanical properties such as light weight, high module, high specific strength, low thermal expansion, high temperature resistance, outstanding corrosion resistance and low friction coefficient [2–5]. However, for conventional C/C composite-based sealing materials, the stability of the friction coefficient is relatively low and the wear resistance is poor when applied as mechanical seals. Therefore, there is an urgent need to modify conventional C/C composites to improve their tribological properties.



To address this issue, conventional C/C composites may be coated with wear-resistant materials to improve their tribological properties. Silicon carbide (SiC) exhibits several excellent properties such as a high melting point, high stiffness, good chemical resistance, good electrical conductivity and remarkable wear resistance [6–8]. Studies have also shown that the SiC coating on C/C composites improves their tribological properties [9–13].

Several methods are used to prepare SiC coatings. Pack cementation is cheap, simple, and it has no sample shape requirements [14–18]. Through the use of this technique, all layers of a surface can be coated using a single process. However, SiC coatings prepared through pack cementation exhibit poor performance. Some researchers have attempted to address this shortcoming. Pourasad reported that SiC/ZrO₂ coatings can be prepared in two steps. The first step involves the preparation of a functionally graded SiC layer via through the pack cementation process. The second step involves the formation of SiC/ZrO₂ coating through pack cementation at 1873 K [19]. Huang stated that SiC coatings were strengthened when doped with Al synthesised on C/C composites through pack cementation [20]. Liu reported that the SiC and ZrSi₂ coatings were prepared in two steps: pack cementation and supersonic atmospheric plasma spraying, respectively. In the first step, the SiC coating was prepared via the pack cementation process. In the second step, the outer ZrSi₂ coating was deposited via supersonic atmospheric plasma spraying [21].

Silicon nitride (Si₃N₄), a typical non-oxide ceramic material, has been used in applications requiring high tribological performance owing to its low chemical activity, high wear resistance and satisfactory mechanical properties [22,23]. In a previous study [24], SiC coatings have been prepared through the use of a low-temperature embedding method to remove any loosely embedded material. However, SiC coatings suffer from defects that reduce the adhesion between the SiC coating and substrate. To address these problems, we applied nitriding and low-temperature packing cementation to form SiC/Si₃N₄ recombination coatings on C/C composites. The resulting Si₃N₄ strengthens the SiC coating.

2. Experiment

2.1. Sample Preparation

Bulk two-dimensional C/C composites with a density of $1.2-1.4 \text{ g/cm}^3$ were produced through chemical vapour deposition. Substrate specimens (7 mm × 7 mm × 30 mm) were cut from the bulk material and hand-polished using 600-grit SiC paper. Prior to pack cementation, the C/C substrates were cleaned ultrasonically in acetone, then rinsed with distilled water and dried in a drying oven at 100 °C for 2 h, followed by immersion in peroxide for 1 h and drying again in a drying oven at 100 °C for 2 h.

The packing powder materials were composed of 300 mesh Si powder, 325 mesh graphite powder and 300 mesh MgO powder. The MgO served to increase the diffusing reaction rate at high temperature. All powders were of analytical grade and were cleaned ultrasonically in ethanol for 30 min, dried at 70 °C for 24 h and then mixed in a powder-mixing machine for 12 h.

After treatment, the C/C composite specimens were packed with the as-prepared powder mixture and then placed in a crucible. The entire crucible was placed in a tube furnace, and SiC was formed upon heating the impregnated specimens under argon gas at 1100 °C for 8 h. The specimens were then allowed to cool passively in the furnace.

Following pack cementation, all specimens were removed from the crucible, and the embedded materials were cleaned off with a brush. The specimens were again placed in the crucible, heated under nitrogen to 1300 °C for 2 h and allowed to cool passively in the furnace. The specimens were then taken out and cleaned ultrasonically in distilled water for 5 min and finally dried at 100 °C for 2 h. Figure 1 shows the preparation process of the specimens.



Figure 1. Flow chart describing the specimen preparation process.

2.2. Friction and Wear Tests

The wear-resistance properties were investigated via ring-block contact wear tests (see Figure 2) with a sliding-wear tester serving for the friction and wear tests (Model MM-P2 Screen display wear tester, Jinan Times Gold Assay Testing Machine Co., Ltd., Jinan, China). The test ring was 23.38 mm in diameter and made of #45 steel. For wear testing, the test load was 100 N, the test ring speed was a constant 200 rpm and the testing time was 30 min. The experimental results were digitised and recorded by the integrated computer system. Before each test, the samples and test ring were carefully hand-polished using 3000 grit SiC paper, followed by ultrasonic cleaning in anhydrous ethanol and acetone and drying at 100 °C for 2 h. These tests were carried out under a relative humidity level of 50–70% and room temperature. Prior to each test, the test ring was hand-polished with 3000 grit SiC paper. For each sample type, four samples were tested, with each individual sample tested four times. The final mass loss was the average of these 16 measurements. The mass loss *W* of the samples was calculated by using:

$$W = [(m_0 - m)/m_0] \times 100\%$$
(1)

where m_0 and m are the mass of the samples before and after wear testing, respectively.



Figure 2. Schematic describing friction and wear tests.

2.3. Specimen Characterisation

The crystalline structure of the coatings was analysed by X-ray diffractometry (XRD, SmartLab Studio ll, Rigaku, Tokyo, Japan). The XRD patterns were collected by using a Bruker D8 diffractometer with Cu K α irradiation over a 2 θ range of 10° to 80°. The chemical states of the samples were investigated by X-ray photoelectron spectroscopy (XPS; Thermo Fisher—VG Scientific, ESCALAB

250Xi, Waltham, MA, USA) with an Al K α source gun operating at 1486.6 eV. The chamber pressure was fixed at 1×10^{-7} mbar, and the beam power was 200 W. The surface morphology of both the initial surface and of typical worn surfaces were analysed by scanning electron microscopy (SEM, Hitachi S-3400 and Hitachi S-4800, Hitachi, Ltd., Tokyo, Japan).

3. Results and Discussion

3.1. Characterisation of SiC and SiC/Si₃N₄ Coatings

Figure 3 shows the XRD patterns of the SiC and SiC/Si₃N₄ coatings, which reveal that the SiC, Si₃N₄ and carbon phases are all present in the SiC/Si₃N₄ coating on the C/C composite surface and that the SiC, Si and carbon phases are all present in the SiC coating. SiC forms mainly during the process of embedding. At temperatures below 1400 °C, the following solid-state reaction occurs:

Si (s) + C (s)
$$\rightarrow$$
 SiC (s) (2)

Figure 3. X-ray diffractometry (XRD) patterns of coated specimens.

The reaction between the two solid phases proceeds at a lower rate than that between solid and liquid phases. Therefore, some reactions do not complete at temperatures below 1400 °C, as demonstrated in our previous work [24]. Thus, at temperatures below 1400 °C, Si phases remain in the SiC coating after embedding, which degrades the friction and wear properties of the coatings. To remove this residual Si phase, the specimens were heated to 1300 °C under nitrogen after pack cementation. Upon injecting nitrogen, the residual Si reacts with nitrogen gas as follows:

$$3Si(s) + 2N_2(g) \rightarrow Si_3N_4(s)$$
 (3)

The nitrogen converts all residual Si to Si_3N_4 in the SiC/Si₃N₄ coatings, as indicated in Figure 3. The carbon phase includes the matrix phase and residual graphite.

An XPS survey shows the chemical states of Si, N, C and O on the surface of the SiC/Si₃N₄ coatings (Figure 4a). Figure 4b shows the deconvolution of the Si 2*p* spectrum into sub-peaks that indicate binding energies of 101.8 and 102.9 eV, which are attributed to Si-C and Si-N, respectively. This finding is consistent with the coexistence of SiC and Si₃N₄ evinced by XRD (Figure 3) [25,26]. The peaks at 284.7 and 285.7 eV in the high-resolution spectra of Figure 4c are assigned to C 1*s*, and the peak at 285.7 eV confirms the presence of SiC. The other deconvolved sub-peak at 284.7 eV corresponds to C=C, which is attributed to graphite in the coatings [25–27]. In Figure 4d, the peak at 397.3 eV confirms the presence of Si₃N₄ [26,28].





Figure 4. X-ray photoelectron spectroscopy (XPS) patterns of SiC/Si₃N₄-coated specimens. (**a**) spectrum and high resolution spectra of (**b**) Si2p, (**c**) C1s, and (**d**) N1s.

Figure 5 shows the surface morphology of C/C composites, revealing numerous large holes in the matrix. Some fibres appear in Figure 5, and the specimen surface is uneven. During the preparation of the coating, SiC and Si₃N₄ penetrate through the pores in the C/C composite, which improves the bonding strength between the coating and the matrix. Thus, the C/C composite microstructure facilitates the bonding between coating and matrix.



Figure 5. Scanning electron microscopy (SEM) image showing surface morphology of carbon/carbon composites.

Figure 6 shows the surface morphology of the SiC and SiC/Si₃N₄ coatings on C/C composites. Figure 6a reveals various defects, such as cracks and loose structures, in the SiC coating, which affect the friction and wear characteristics of the coating. Conversely, the specimen coated by SiC/Si₃N₄ has fewer defects, as shown in Figure 6b. The cracks in the SiC/Si_3N_4 coating can be attributed to the coefficients of thermal expansion. The lower defect density in the SiC/Si_3N_4 coating reduces the friction coefficient and the wear of the coating.



Figure 6. SEM images showing surface morphology of (a) SiC and (b) SiC/Si_3N_4 coatings on carbon/carbon composites.

Figure 7 shows the cross-section of a SiC/Si₃N₄ coating and its energy dispersive X-ray spectrum (EDS). The coating is determined to be 8.25 μ m thick. Si atoms that diffuse into the C/C composite react with the matrix, which increases the adhesion between these materials. In addition, the coating penetrates into the C/C composite, which also increases the adhesion between the materials. The EDS analysis reveals the presence in the SiC/Si₃N₄ coating of elemental Si, C and N, but no MgO.



Figure 7. The cross-section morphology and energy dispersive X-ray spectrum (EDS) of SiC/Si_3N_4 coating.

3.2. Tribological Performance

Figure 8 shows the friction coefficient μ as a function of time from the onset of wear testing for C/C composites with different coatings. For the specimens with the SiC coating, the friction coefficient increases at the outset, and then decreases. In a follow-up experiment (i.e., with the same specimen), the friction coefficient likewise decreases after a small increase. Finally, the friction coefficient for SiC coatings stabilises around 0.2 after ca. 10^3 s.



Figure 8. Friction coefficients for C/C composites with different coatings as a function of time from the onset of wear testing.

For specimens with the SiC/Si₃N₄ coating, the friction coefficient (μ) decreases immediately from the outset to rapidly (ca. 10² s) approach a constant value of less than 0.1, which is less than the stable value of the friction coefficient of the SiC-coated specimens. Microstructural analysis of the SiC/Si₃N₄ coating indicates that the Si and graphite powder do not react completely so that a significant quantity of graphite powder remains after processing at 1100 °C. This remaining graphite powder is consumed as the residual Si proceeds with the reaction, decreasing the friction coefficient. In addition, as shown in Figure 7, the defect density in the SiC/Si₃N₄ coating is much less than in the SiC coating. Thus, the SiC/Si₃N₄-coated specimens yield a relatively small friction coefficient (less than 0.1; see Figure 8). The rather large (about 0.2–0.4) and unstable friction coefficient for SiC-coated specimens is attributed to the loose structure and the type of wear in the SiC coatings.

Figure 9 shows the average weight loss of several specimens, which is directly related to the friction coefficient. As shown in Figure 8, the friction coefficient of SiC coatings is unstable, so the weight loss is significant. The opposite is true for the SiC/Si₃N₄-coated specimens: the friction coefficient is lower and more stable, resulting in much less weight loss. Furthermore, the Si₃N₄ in the SiC/Si₃N₄ coatings is more resistant to wear than Si in the SiC coating, so the SiC/Si₃N₄ coating loses less mass than the SiC coating, which also contributes to the more stable friction coefficient of the SiC/Si₃N₄ coating.



Figure 9. Average weight loss of different coatings.

3.3. Analysis of Worn Surface

Figure 10a,b shows SEM images that reveal the morphology of the worn surface of specimens with the SiC coating and with the SiC/Si₃N₄ coating, respectively. These images show different levels of deterioration of the worn surfaces of the two specimens. Moreover, the typical debris and the characteristics of the worn surface are closely linked with the wear process, which helps to understand the wear mechanisms.



Figure 10. SEM images showing surface morphology of worn surfaces of (a) SiC and (b) SiC/Si $_3N_4$ coatings on carbon/carbon composites.

Figure 10a shows the surface of a SiC coating worn at room temperature. The wear track is very deep, and debris is apparent along with some degree of exfoliation. However, a smooth, worn surface is also apparent. Additionally, several furrows appear on the worn surface. These results imply that the worn surface contributes to abrasive and adhesive wear. Initially, adhesive wear proceeds easily because of the effect of polishing of the specimens. Moreover, because of the brittle fracture of SiC grains, some debris forms easily during wear [29,30], which means that adhesive wear is accompanied by abrasive wear. In the first stage of the dynamics of the friction coefficient of SiC coatings (see Figure 8), the large friction coefficient may contribute to adhesive and abrasive wear. Along with the development of friction and wear, a thin film forms on the coating surface, which decreases the friction coefficient of the coating. In turn, this decreases the friction coefficient in the subsequent stage of the dynamics of the friction coefficient of SiC coatings.

Figure 10b shows the SEM image of the surface of a SiC/Si₃N₄ coating worn at room temperature. The image reveals a shallow wear track, some smooth worn regions and several non-worn regions. The deformation and fracture of the SiC/Si₃N₄ coating are mainly due to the friction process. Because the coating was polished beforehand, the surface of the coating is smooth and clean. Adhesive wear is facilitated on such smooth and clean surfaces, especially if the surfaces are dense and resistant to moisture or other hydrated contaminations [12,31]. Thus, we deduce that the smooth and clean worn surface contributes to the adhesive wear. Meanwhile, a small quantity of debris appears on the worn surface, which leads to mild abrasive wear. Adhesive wear combined with mild abrasive wear could lead to a large friction coefficient [30], which can explain the large initial friction coefficient in the first stage of the dynamics of the friction coefficient (Figure 8). In addition to the development of friction and wear, a layer of thin film forms on the coating surface, which decreases in the latter stage of the coating. As a result, the friction coefficient of SiC/Si₃N₄ coatings decreases in the latter stage of the friction coefficient dynamics (Figure 8). Compared with SiC coatings, SiC/Si₃N₄ coatings has a lower friction coefficient forms of wear.

4. Conclusions

To improve wear resistance, SiC and SiC/Si $_3N_4$ coatings were synthesised on C/C composites via pack cementation and, for the SiC/Si $_3N_4$ coatings, heat treatment with nitrogen gas. XRD, XPS, EDS and

SEM analyses reveal differences between the SiC and SiC/Si₃N₄ coatings in terms of microstructure, surface morphology and tribological behaviour. The SiC coatings synthesised through pack cementation at 1100 °C are loose and contain large pores, whereas the SiC/Si₃N₄ coatings similarly synthesised but with a subsequent nitrogen-gas heat treatment are more compact. The SiC coating has a relatively large friction coefficient, which is caused mainly by adhesive and abrasive wear. The SiC/Si₃N₄ coating yields a smaller, more stable friction coefficient and less weight loss than the SiC coating. At the onset of frictional wear of the SiC/Si₃N₄ coating, the friction coefficient is relatively large (although not as large as that of the SiC coating) and is due mainly to adhesive wear. Graphite in both the SiC/Si₃N₄ and the SiC coatings produces a thin lubricating film that subsequently decreases the friction coefficient after certain duration (10^2 to 10^3 s, respectively) of frictional wear. Thus, compared with SiC coating, SiC/Si₃N₄ coating offers a lower friction coefficient and better wear performance.

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