Influence of nitrogen ion implantation energies on surface chemical bonding structure and mechanical properties of nitrogen-implanted silicon carbide ceramics

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A B S T R A C T

Nitrogen ions were implanted into silicon carbide ceramics (N+-implanted SiC) at different ions energies. The surface chemical bonding structure of N+-implanted SiC ceramics were investigated by using X-ray photoelectron spectroscopy (XPS). The hardness of N+-implanted SiC ceramics was measured using nano-indenteter, and the friction and wear properties of the N+-implanted SiC/SiC tribopairs were studied using ball-on-disk type tribo-meter in water lubrication. The wear tracks were observed using non-contact surface profilometer and scanning electron microscope (SEM). The results showed that the surface roughness of N+-implanted SiC ceramic was higher than that of SiC ceramic, and some chemical bonds such as Si–N, C–C, C–N and C–N bonds were formed in N+-implanted layer besides Si–C bonds. In comparison of SiC ceramic’s hardness, the hardness of N+-implanted SiC ceramics at 30 and 50 keV was higher while that at 65 keV was lower. Under water lubrication, the friction coefficient and the specific wear rates for the N+-implanted SiC/SiC tribopairs were all lower than those of the SiC/SiC tribopairs, and displayed the lowest values at 50 keV. According to XPS analysis, it was concluded that the high wear resistance and low friction coefficient for the N+-implanted SiC/SiC tribopairs were attributed to the formation of carbon rich composite on the surface of N+-implanted SiC ceramics.

1. Introduction

Due to excellent mechanical and chemical properties, SiC ceramics are increasingly used in tribological applications such as mechanical face seal rings and journal bearings etc. [1]. Until now, most researches about the tribological properties of SiC ceramics in water lubrication have focused on their low friction coefficient and related mechanism [2–4]. Although SiC ceramics possess high corrosion resistance or chemical inertness under static conditions, they will undergo tribochemical reactions and suffer severe wear during sliding in water lubrication [4–6]. It is imperative to enhance the wear resistance of SiC ceramics in water lubrication. Actually, the friction and wear properties of ceramics are very surface-sensitive. When the amorphous carbon nitride (a-CN x) coatings were deposited on the SiC disks and slid against SiC ball in water, the running-in distance of the a-CN x/SiC tribopairs was shorter than that of the SiC/SiC tribopairs, and the low friction coefficient of 0.01–0.02 was obtained. Furthermore, the SiC ball’s wear resistance for the a-CN x/SiC tribopairs was better than that of the SiC/SiC tribopair in water [6–10]. This indicates that the a-CN x/SiC tribopairs are the promising candidates for the sliding parts in water lubrication systems. However, the delamination of the a-CN x coatings at lower sliding speed and higher normal load was not useful to improve the tribological of SiC ceramics in water lubrication [10].

In comparison to surface coatings deposition, ion implantation is a surface engineering technique whereby the mechanical and physical properties of a thin (typically 0.1–0.5 μm) near-surface layer can be altered via implanting high energy (typically ranging from 25 keV up to several MeV) metallic or non-metallic ions. It has been shown that the ion implantation of ceramics with various ions can improve their tribological properties in dry air by reducing adhesion [11–14]. But up to now, the research on the tribological properties of N+-implanted SiC ceramics in water is still limited [15]. The objective of this study was to clarify the influence of N+ ions energies on surface chemical bonding structure, hardness and tribological properties of N+-implanted SiC ceramics in water lubrication.

2. Experimental procedures

2.1. Preparation of N+-implanted SiC ceramics

The reactive sintered polycrystalline SiC ceramic disks (Ø30 mm × t4 mm) were supplied by Shanghai Ceramic Institute,
Chinese Academy of Science. These SiC disks were mechanically polished and ultrasonically cleaned in acetone for about 10 min prior to ion implantation. Nitrogen ion implantation was performed on the custom multi-functional ion implanter schematically depicted in Fig. 1. The instrument was equipped with a DC and RF magnetron sputtering source. The SiC disks were placed into chamber, and the working vacuum pressure in the chamber was 1.0 × 10⁻² Pa. The diameter for implanted area was 100 mm. Nitrogen ions were implanted into the SiC ceramics at fluence of 5.0 × 10¹⁷ ions/cm² and the ions implantation energy in the range of 30–65 keV. During ion implantation, the beam current was 1.7 mA, and substrate temperature was 300 °C.

2.2. Surface roughness and chemical bonding structure of N⁺-implanted SiC ceramics

The surface roughness of N⁺-implanted SiC disks was measured using a MicroXAM™ non-contact optical profilometer (ADE Phase-Shift, USA). To obtain the surface roughness accurately, at least five measurement tests were carried out for each specimen. The chemical bonding structure of N⁺-implanted SiC ceramics were analyzed by Thermo ESCALAB 250 X-ray photoelectron spectroscopy (XPS, America). The XPS analysis was carried out using a 150 W Al Kα (hv = 1486.6 eV) X-ray source with a 500 μm beam diameter.

2.3. N⁺-implanted SiC ceramics’ hardness and their tribological behaviors in water lubrication

The hardness of SiC and N⁺-implanted SiC ceramics was determined using a SA2 nano-indentation hardness tester (MTS, USA) with a Berkovich-typed diamond indenter. The maximum load was 20 mN. The time for loading and unloading was fixed to 30 s, and the loading/unloading rate was 0.67 mN/s. The holding time at the maximum load was 1 s. To obtain the accurate hardness and elastic modulus, at least nine indentation tests were made for each disk. The projected contact area A_c was determined from tip-shaped polynomial function of the contact depth at maximum indentation load. The reduced elastic modulus is given by $1/E_r = (1 - v_i^2)/E_i + (1 - v_s^2)/E_s$, where $E_i$, $E_s$ and $v_i$, $v_s$ are the elastic modulus and Poisson’s ratio for the indenter and the substrate materials, respectively. For a diamond indenter material, $E_i$ is 1050 GPa and $v_i$ is 0.1. The Oliver–Pharr method [16] is used to calculate the hardness and reduced modulus values from loading–unloading curves. The reduced elastic modulus was calculated from the force–indentation depth curve by the relationship $E_r = S/2(\pi A_c)^{1/2}$ where $S$ is the stiffness, defined as the slop of the unloading curve at the initial unloading $(dp/dh)$. The stiffness is obtained by the first fitting the unloading curve to the power law relation, $P = A(h - h_f)^m$, where $A$, $h_f$ and $m$ are arbitrary fitting parameters. The stiffness can be calculated from the derivative of the preceding equation: $S = dP/dh(h_{max}) = mA(h_{max} - h_f)^{m-1}$. The hardness is defined as the ratio between the maximum load and the projected contact area, $H = P_{max}/A$. The contact area is defined from a tip calibration function $A(h_c)$ where $h_c$, the contact depth, is found by using the following equation: $h_c = h_{max} - \varepsilon P_{max}/S$, where $\varepsilon$ is 0.75.

The friction and wear tests were performed on a ball-on-disk type apparatus which consisted of rotating disk sliding against a stationary ball at 0.15 m/s and 1.8 N (Fig. 2). Prior to each sliding test, The SiC balls with the diameter of 8 mm and disks were ultrasonically cleaned in acetone and ethanol for 15 min. The rubbing
surface was submerged in distilled water at room temperature. The total sliding distance was 10,000 m. The friction force was detected by a LMA-A-10 N load cell (Kyowa Co. LTD, Japan). The load cell voltage was measured using a DPM-700B strain amplifier (Kyowa Co. LTD, Japan) and recorded by NR-110/150 data collection system (Keyence Co. LTD, Japan) with a compatible personal computer. The diameter of SiC ball’s wear scar was measured using an optical microscope, and the cross-sectional area of wear track on disk was determined using non-contact optical profilometer (ADE Phase-Shift, USA), then the specific wear rates for balls and disks were calculated. To obtain the mean stable friction coefficient and specific wear rate accurately, the friction tests were performed reproducibility for five times. Moreover, the wear tracks on disks were observed using JEOL-JSM-7001F FE-SEM.

3. Results and discussion

3.1. Surface roughness and chemical bonding structure of N⁺-implanted SiC ceramics

Fig. 3 shows the surface roughness of SiC ceramics before and after N⁺ ions implantation. It is clear that the arithmetic mean roughness Ra of N⁺-implanted SiC ceramics was higher than that of SiC ceramics, and increased from 44 to 65 nm at N⁺ ion energy at 30 keV. If N⁺ ions energy was higher than 50 keV, the surface roughness fluctuated slightly in the range of 53.6–59.3 nm. This indicated that the increasing roughness could be easily attributed to cracks caused by stress induced by implantation. Figs. 4–6 illustrate wide-scan, Si2p, C1s and N1s peaks for N⁺-implanted SiC ceramics at different ion energies, respectively. The wide-scan peaks in Figs. 4(a), 5(a) and 6(a) showed the presence of C, N, Si, and a small amount of O in the implantation layer. Because the oxygen peak centered at 533 eV was very week and almost disappeared after sputtering with Ar⁺ ions. Thus, the effect of oxygen

Fig. 3. Influence of nitrogen ion energies on surface roughness of the N⁺-implanted SiC ceramics.

Fig. 4. XPS spectra of wide-scan peak (a), Si2p photoelectron peaks (b), C1s photoelectron peaks (c) and N1s photoelectron peaks (d) for SiC ceramics implanted with N⁺ ions at 30 keV.
was neglected. To tell from the chemical bonding configurations of N+-implanted SiC ceramics, the individual Si2p, C1s and N1s spectra all were deconvoluted into Gaussian line shapes. When N+ ions were implanted into SiC ceramics at 30 keV, the deconvoluted Si2p spectrum in Fig. 4(b) exhibited three peaks at the binding energies of 101.2, 102.2 and 103.2 eV. Besling et al. [17] reported that the binding energy of Si–C bond, Si–N bond in Si3N4 and Si–O bond varied in the range of 99.8–100.8, 101.5–102.3 eV and 102–103 eV, respectively. It is evident that the binding energy of Si–C bond in Si–C–N system was higher than that in SiC ceramic, while the binding energy of Si–N bond in Si–C–N system was identical to that in Si3N4 ceramic. This indicated that the elements electronegativity played a crucial role in the chemical shift of corresponding bonding state in XPS spectra. Because the electronegativity of N was 3.04, higher than that of C (2.55), and the partial replacement of C in Si–C bonding by N was occurred during nitrogen ion implantation, so the chemical binding energy for Si–C bond shifted to higher position. Therefore, the peaks at 101.2, 102.2 and 103.2 eV in Fig. 4(b) were assigned to Si–C, Si–N and Si–O bonds, respectively. As seen in Fig. 4(c) and (d), the deconvoluted C1s spectra exhibited four peaks at the binding energies of 283.4, 285.0, 287.0 and 288.8 eV, while the deconvoluted N1s spectra displayed three peaks at the binding energies of 398.1, 400.4 and 403.1 eV. It is known that the binding energy of C–Si and N–Si bonds varied in the range of 283.2–283.6 eV and 397.1–397.8 eV, respectively, Scharf et al. [18] indicated that the peaks at binding energies of 284.5, 285.2, 286.5 and 288.6 eV for the deconvoluted C1s spectra were attributed to C–C, C–N–C, C–N and C–O bonds, respectively, while the peaks at 398.6, 400.1 and 402.3 eV for the N1s line were assigned to C–N, C=N and N–O bonds, respectively.

In comparison to the data of [18,19], the peaks at 283.4, 285.0, 287.0 and 288.7 eV in Fig. 4(c) were assigned to C–Si, C–C=C=N, C–N and C–O bonds, respectively, while the peaks at 398.1, 400.4, 403.1 eV in Fig. 4(d) were marked as N–Si, C=N and N–O bonds, respectively. If N+ ions were implanted into SiC ceramics at 50 keV, the Si2p spectrum could be fitted to three peaks at 101.3, 102.2 and 103.3 eV, which were attributed to Si–C, Si–N and Si–O bonds, respectively (Fig. 5(b)). For the C1s core level line in Fig. 5(c), there were four peaks at 283.3, 285.2, 286.6 and 288.3 eV. It is obvious from [20] that the peak at 283.3 eV was assigned to Si–C, whereas the peaks at 285.2, 286.6 and 288.3 eV were related to C–C=C=N, C–N and C–O bonds, respectively. The deconvoluted N1s spectrum in Fig. 5(d) displayed three peaks at 398, 400.7 and 402.9 eV, which were assigned to C–N, C=N and N–O bonds, respectively. The appearance of Si–O, C–O and N–O bonds revealed that the nitrogen ion implantation surfaces were contaminated by oxygen from air.

To clarify the influence of N+ ions energies on the surface chemical bond of N+-implanted SiC ceramics, the variation of
the relative percentage of chemical bonds with N+ ions energy was tabulated in Tables 1–3. It is evident that, when N+ ions energy was 50 keV, the relative percentage of chemical bonds such as Si–C, Si–N, Si–O and N–O bonds became the lowest value, while that of C–C/C=N and C–N bonds displayed the highest value. This indicated that the surface structure of N+-implanted SiC ceramics was changed from SiC to SiCN–SiC composite structure, and the rich carbon composite compounds were formed on the surface of N+-implanted SiC ceramics, which would influence the tribological properties of N+-implanted SiC ceramics in water environment.

### 3.2. Mechanical properties of N+-implanted SiC ceramics

Fig. 7 shows the influence of nitrogen ion implantation energies on hardness of the SiC ceramics before and after N+ ions implantation. It is obvious that the hardness for SiC ceramics was 17.7 GPa, while that of N+-implanted SiC ceramics was 22 GPa at 30 keV. The results from XPS analyses showed that the formation of Si–N bonds or Si–C–N bonds after N+ ions implanted into SiC ceramics could

<table>
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<th>Specimens</th>
<th>/notification energy, ions/cm²</th>
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<tr>
<td>30 keV, 5 × 10¹⁷</td>
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<td>9.81</td>
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<tr>
<td>50 keV, 5 × 10¹⁷</td>
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<td>65 keV, 5 × 10¹⁷</td>
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<td>12.18</td>
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<th>C–O</th>
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<td>65 keV, 5 × 10¹⁷</td>
<td>17.64</td>
<td>5.55</td>
<td>0.9</td>
<td>24.1</td>
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Fig. 6. XPS spectra of wide-scan peak (a), Si2p photoelectron peaks (b), C1s photoelectron peaks (c) and N1s photoelectron peaks (d) for SiC ceramics implanted with N+ ions at 65 keV.
enhance the surface hardness of SiC ceramics. If nitrogen ions energy was higher than 50 keV, the hardness of N⁺-implanted SiC ceramics decreased. From [20] reported that the hardness of N⁺-implanted SiC ceramics was related to the formation of bubbles below the Si–C–N compound. This pointed out that the probability of bubble formation was the biggest at 30 keV, then decreased with increasing ion energy.

Fig. 8 displays the variation of friction coefficients with sliding distance for the N⁺-implanted SiC/SiC tribopairs in water. It is evident that the initial friction coefficient for the N⁺-implanted SiC/SiC tribopairs was 0.22 or so, slightly lower than that of SiC/SiC tribopair (0.26). For the SiC/SiC tribopair, the friction coefficient first decreased gradually from a higher initial value (0.26) to 0.04 as the running-in period was shorter than 4260 m. With further sliding, the friction coefficient fluctuated in the range of 0.038–0.041. When N⁺ ions were implanted into SiC ceramic at 30 keV, the friction coefficient of N⁺-implanted SiC/SiC tribopair first decreased rapidly from 0.23 to 0.036 as the sliding distance shorter than 2460 m, and then varied around 0.036 with further sliding. At N⁺ ions energy of 50 keV, the friction coefficient first decreased suddenly from 0.23 to 0.039 as the running-in period shorter than 1520 m, and then decreased slightly from 0.04 to 0.018 with increasing sliding distance. As N⁺ ions implantation energy was 65 keV, the friction coefficient of N⁺-implanted SiC/SiC tribopair first decreased gradually from 0.22 to 0.04 as the sliding distance shorter than 4740 m, and then fluctuated in the range of 0.027–0.035 with further sliding. This indicated that the N⁺-implanted SiC ceramics at 50 keV exhibited the lowest friction coefficient and the shortest running-in distance.

The influence of N⁺ ions energy on the mean stable friction coefficient (μ̄) after running-in and the specific wear rates for tribomaterials is illustrated in Fig. 9. As seen in Fig. 9(a), the mean steady-state friction coefficients decreased markedly from 0.044 to 0.022 when N⁺ ions energy was lower than 50 keV. It is obvious from Fig. 9(b) that the specific wear rates of SiC balls were approximately ten times lower than those of SiC disks, and the specific wear rates of disk and ball for N⁺-implanted SiC/SiC tribopair were lower than those for SiC/SiC tribopair. With an increase in N⁺ ions energy, the specific wear rates of disk and ball first decreased to minimum values at 50 keV, and then increased slightly. This showed that N⁺ ions implanted into SiC ceramics at 50 keV had the best tribological properties in water among the studied samples. The difference of tribological behavior between SiC/SiC and N⁺-implanted SiC/SiC tribopairs indicated that nitrogen ion implantation had positive influences on the friction and wear properties of SiC ceramics in water.

3.3. Observation of worn surface on disk at different ion implantation energies

Fig. 10 shows the sectional morphologies of wear track on SiC and N⁺-implanted SiC ceramics at various ion energies. As the nitrogen ion energy was lower than 50 keV, the depth and width of wear track were higher. If the nitrogen ion energy was higher than 50 keV, the depth and width of the wear track were lower. This indicated that the wear resistance of SiC disk was enhanced by N⁺ ions implantation at high ion energy. To know the influence of ion energy on the wear track surfaces, the wear track surfaces were observed by SEM. As seen in Fig. 11, the wear track surfaces became smooth and flat besides many original voids and black carbons. For SiC disk, there were some wavelike films on the smooth surface along sliding direction (as seen red cycle in Fig. 11(a)). But for N⁺-implanted SiC ceramics at 30 keV, there were many black parts and some pits on the smooth surface (Fig. 11(b)). When the ion energy was 50 keV, the black parts became gray parts, and there were thin wavelike films on the smooth wear track surface (as seen red cycle in Fig. 11(c)). When N⁺ ions were implanted into SiC ceramics at 65 keV, the wear track surface was covered with thick wavelike and larger size voids (as seen red cycle in Fig. 11(d)). This indicated the wear mechanism was mix wear between mechanical wear and tribochemical wear.
3.4. Discussion

In the viewpoint of friction, the friction extent between two sliding surfaces is largely governed by the physical condition of the contacting interface and the chemical interactions between sliding interfaces and the environment. Physically, rough surfaces can create higher friction coefficient. As seen in Figs. 1 and 7, the surface roughness of N+-implanted SiC ceramic was higher than that of SiC disk. However, the initial friction coefficient of N+-implanted SiC/SiC tribopair was lower than that of SiC/SiC tribopair. This indicated that nitrogen ion implantation would change the SiC ceramics’ surface characteristics. Furthermore, the chemical interactions between two sliding surfaces and water can play more important role in the friction and wear behavior of N+-implanted SiC ceramics in water. The large difference in friction and wear behaviors between N+-implanted SiC/SiC and SiC/SiC tribo-system suggested that their lubrication and wear mechanism be different. It is clear that the difference of running-in distance between SiC and N+-implanted SiC could be attributed to their energy gap difference [3]. The smaller the energy gap between both reagents, i.e. the higher the reaction rate is. As seen in Figs. 4–6, it is obvious that silicon atoms may maintain carbon-to-nitrogen bonds in sp³ configurations instead of sp² hybrids [20]. Although the energy gap of SiC and Si(C)₃N₄ is low compared with water, the difference of energy gap between Si(C)₃N₄ and water is 3.0 or 1.25 eV but that between SiC and water is 5.2, so the reaction rate between Si(C)₃N₄ and water is higher than that between SiC and water [21]. This indicated that nitrogen ion implantation would have more acceleration effects on the tribochemical reaction during sliding in water. Thus, the running-in distance of the N+-implanted SiC/SiC tribopair was shorter than that of the SiC/SiC tribopair. As seen in Tables 1–3, the surface structure for N+-implanted SiC ceramics at 50 keV became the SiC/SiCN composite carbon rich layers, and the atomic concentration of C–C/C≡N and C–N bonds displayed the highest value. Our previous studies [8] have showed that the sp³ C≡N and sp² C≡N bonds were the major components in the a-CNₓ coatings. When the SiC balls slid against the a-CNₓ coated SiC and SiC disk in water, respectively, the running-in distance of the a-CNₓ/SiC tribopair was shorter than that of the SiC/SiC tribopair, and the wear resistance of SiC ball for the a-CNₓ/SiC tribopair was better than that for the SiC/SiC tribopair. It is clear from Figs. 8 and 9 that the N+-implanted SiC ceramic at 50 keV possesses the best tribological properties in water. Generally, the amorphization of the near-surface region and the formation of lubricating film on the wear track have been proposed to play a role in wear reduction of the implanted ceramics [19]. This indicated that nitrogen ion implantation would change the surface elements concentration and chemical bonds of SiC ceramics, which would influence the friction and wear behavior of SiC ceramic in water. When the surface structure of SiC ceramics was changed into carbon rich or carbon nitride composite layer, the N+-implanted SiC ceramic possessed the low friction coefficient and the low specific wear rate as sliding against SiC balls in water.

4. Conclusions

The influences of nitrogen ions energy on the surface chemical bonding structure and mechanical properties of N+-implanted SiC...
ceramics have been investigated in detail. The main results could be summarized as:

1. The surface roughness of N⁺-implanted SiC ceramic was higher than that of SiC ceramic. Some chemical bonds such as Si–N, C–C, C≡N, C–N bonds and the carbon rich composite layer were formed on the surface of SiC ceramics as N⁺ ions implanted into SiC at 50 keV.

2. With an increase in nitrogen ion energy, the hardness of N⁺-implanted SiC ceramic first increased, and then decreased. The highest hardness of 22 GPa was obtained as N⁺ ions implanted into SiC ceramics at 30 keV.

3. The friction coefficients and the specific wear rates of tribomaterials for N⁺-implanted SiC ceramic/SiC tribopairs first decreased to minimum value, and then increased a little with increasing nitrogen ion energy. The N⁺-implanted SiC ceramic at 50 keV possesses the excellent tribological properties in water lubrication.

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