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Stable and super-low friction of amorphous carbon nitride coatings in nitrogen gas by using two-step ball-on-disk friction test

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ABSTRACT

Effect of running-in process on friction behaviour of carbon nitride (CNx) coating in N_2 gas stream was investigated with a newly introduced two-step ball-on-disk friction test, where the rubbed Si_3N_4 ball in the pre-sliding (step 1) was replaced by a new CNx-coated Si_3N_4 ball in the subsequent sliding stage under N_2 gas (step 2). The two-step friction test is clarified to be a simple but effective technique for obtaining contact material combination of self-mated CNx coatings and for achieving stable and low frictions of CNx coatings. Friction coefficients of CNx/CNx in N_2 gas stream decrease greatly from 0.07 without pre-sliding to less than 0.025 in two-step friction tests. The minimum friction coefficient of 0.004 was obtained by introducing 500 cycles of pre-sliding in ambient air. These stable and low frictions are attributed to the generation of self-mated CNx coatings and the formation of a lubricious layer on the disk surface.

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KEY WORDS: carbon nitride; nitrogen gas; friction coefficient; running-in; pre-sliding

INTRODUCTION

Amorphous carbon nitride (CNx) coating has been expected as a promising solid lubrication coating in demanding industrial applications because of its excellent tribological performances, such as low friction coefficient and high wear resistance. The friction behaviour of CNx coatings is highly related to surrounding gas environments. Typically, friction coefficients of higher than 0.1 are observed in air and oxygen gas, whereas friction coefficients in the order of 0.01 or even 0.001 are recorded in N_2 gas environment.[1–5] However, the low friction mechanism of CNx coatings in N_2 gas environment is still not yet clearly understood.

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The beneficial effect of N_2 gas on reducing frictions of CNx coatings is improved by running-in process, which is pre-sliding before introducing N_2 gas to the sliding interface.[6–8] Specifically, a quick reduction of the steady-state friction coefficient from 0.1 to 0.02 is observed in the sliding contact of Si_3N_4 ball/CNx-coated Si_3N_4 disk by blowing N_2 gas at the 1000th cycle after pre-sliding in air. Such effect is much more pronounced when the pre-sliding is conducted in O_2 gas for the initial 50 cycles, the steady-state friction coefficient in the subsequent N_2 gas environment decreases drastically to 0.005 in the sliding contact of CNx-coated Si_3N_4 ball/CNx-coated Si_3N_4 disk. It is concluded that sliding history in air or O_2 gas before N_2 gas supply strongly affects the subsequent friction behaviour of CNx coatings in N_2 gas environment; stable and low frictions of CNx coatings in N_2 gas can be obtained by giving proper initial sliding history.

To elucidate the low friction mechanisms of CNx coatings in N_2 gas environment after running-in process, much effort has been devoted to the composition and structural changes of the mating ball surface in the ball-on-disk tribosystem, as the formation of a uniform and homogenous carbon-rich tribofilm on the ball surface is generally considered as a major point for achieving stable and low frictions of CNx coatings in N_2 gas environment.[6–8] On the other hand, few studies have focused on the composition and structural changes of the disk surface after running-in process.[9] Moreover, graphitisation can also be observed on the top layer of the disk surface in the low friction condition of CNx coatings in N_2 gas environment.[4] Therefore, to clarify the role of composition and structural changes of disk surface in the low friction mechanisms of CNx coatings in N_2 gas environment, a new testing method in relation to the control of disk surface, two-step ball-on-disk friction test, is introduced in this study to investigate the effect of running-in process on the friction behaviour of CNx coatings in N_2 gas environment. The two-step ball-on-disk friction test includes pre-sliding and ball exchange, where the rubbed Si_3N_4 ball in the pre-sliding (step 1) is replaced by a new CNx-coated Si_3N_4 ball in the subsequent sliding stage under N_2 gas environment (step 2). The possibility of obtaining further stable and low friction coefficients of CNx coatings in N_2 gas environment with suitable actively controlled sliding history is clarified. Moreover, the mechanism for the low frictions of CN_x coatings in N_2 gas environment is discussed from the viewpoint of the composition and structural changes of the disk surface.

EXPERIMENTAL

CNx coating preparation

Carbon nitride coatings were prepared in an ion beam-assisted deposition system at room temperature and schematic illustration of the system can be found elsewhere.[3,10] The CNx coatings were grown on the substrates by the deposition of carbon from a carbon target together with the bombardment and mixing of carbon with the nitrogen ions generated simultaneously from an ion beam gun. The substrate materials were Si_3N_4 disks with diameter of 30 mm and thickness of 4 mm and Si_3N_4 balls with diameter of 8 mm. These substrates were sequentially cleaned in an ultrasonic bath with acetone, ethanol and deionized water for 20 min each before loading into the deposition chamber. Prior to deposition, they were further sputter-cleaned by 5 min bombardment with nitrogen ions in order to remove the native oxides and other adsorbed species from their surfaces. The deposition rate of carbon was about 2.0 nm s^{-1}. The total coating thickness on the substrates was about 400 nm. The details of the deposition procedures and the deposition parameters for the CNx coatings are described elsewhere.[10]

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Ball-on-disk tribometer

The sliding friction tests were conducted by using a customised ball-on-disk tribometer with a gas supply unit, as schematically shown in Figure 1. The gas environment around the contact interface was controlled by directly blowing the gas into the contact point using a gas nozzle. The gas nozzle with inner diameter of 4.5 mm was placed perpendicular to the ball holder, with a vertical angle of 15° and a distance of 10 mm from the contact point between the ball and disk. The gas flow rate was fixed at 2.0 L min^{-1}. Two types of high-purity commercial industrial gases, such as nitrogen (>99.9995 vol.%) and oxygen (>99.9 vol.%) were employed for friction tests. The friction tests were also conducted in dry air (relative humidity of around 10% RH), ambient air (relative humidity of around 30% RH), and humid air (relative humidity of around 50% RH) for comparison.

A two-step ball-on-disk friction test, as schematically shown in Figure 2, was introduced to study the effect of running-in process on the friction behaviour of CNx coatings. The first step, 'step 1', was also called pre-sliding, separated from the subsequent 'step 2'. The rubbed Si_3N_4 ball was replaced by a new CNx-coated Si_3N_4 ball between the two steps. The atmosphere in step 1 was N_2, O_2 or none gas blow in ambient air (thereafter referred to as ambient air). The number of cycles in pre-sliding ranged from 0 to 1000 cycles (0, 50, 100, 250, 500 and 1000). The effect of pre-sliding given in various conditions such as number of cycles and gas atmospheres was evaluated by steady-state friction coefficient in step 2.

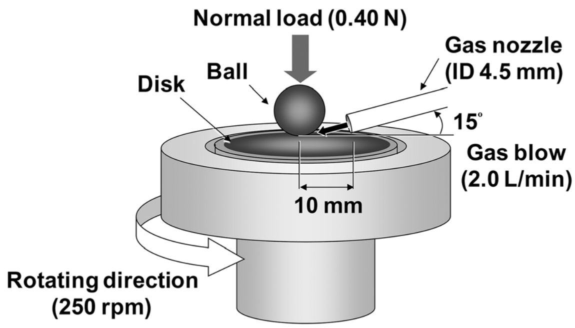

Figure 1. Schematic illustration of the ball-on-disk tribometer with gas nozzle.

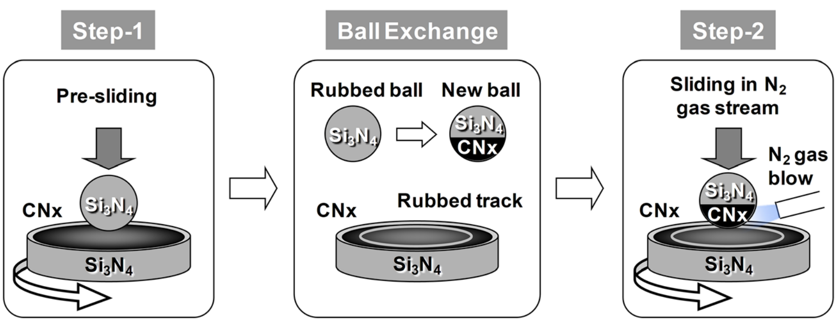

Figure 2. Schematic illustration of the two-step ball-on-disk friction test.

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Carbon nitride-coated Si_3N_4 disks were driven to rub against Si_3N_4 balls (denoted as Si_3N_4/CNx) or CNx-coated Si_3N_4 balls (denoted as CNx/CNx) at a room temperature ranging from 18 to 23°C. The normal load applied on the ball holder was 0.40 N, creating a maximum Hertzian contact pressure of 517 MPa for the contact material combination of CNx/CNx. The wear track diameter on the disk varied between 16 and 24 mm, which corresponded to 0.21–0.31 $m\,s^{-1}$ sliding velocity at a fixed rotation speed of 250 rpm. The friction test in step 2 ran for a sliding duration of 40 min (10 000 cycles).

To clarify the role of disk surface roughness in the pre-sliding, the CNx-coated Si_3N_4 disk was polished by using a lapping machine (Doctor-Lap, Maruto Instrument Corporation, Japan). The surface roughness, such as roughness average (R_a) and maximum height of the profile (R_z), of the disk decreased from 34 nm and 1.027 μm to 3 nm and 0.025 μm, respectively.

Characterization of worn surface

The worn surfaces on the CNx-coated Si_3N_4 balls after friction tests were observed by an optical microscope (ECLIPSE LV150 and Digital Slight DS-L1, Nikon Corporation, Japan). The chemical compositions of the initial and worn surfaces on the CNx-coated Si_3N_4 disks were analysed by X-ray photoelectron spectroscopy (Theta Probe, Thermo Fisher Scientific Corporation, USA). The monochromated Al-Kα (hν = 1486.68 eV) radiation was used as the excitation source. The structural changes of the initial and worn surfaces on the CNx-coated Si_3N_4 disks were characterised by transmission electron microscopy (TEM, HF-2000, Hitachi Corporation, Japan) and Raman spectroscopy (NRS-3100, JASCO Corporation, Japan).

RESULTS AND DISCUSSION

Effect of pre-sliding on friction of CNx/CNx in N_2 gas stream

Representative effect of pre-sliding on the friction behaviour of CNx/CNx in N_2 gas stream is shown in Figure 3. The corresponding optical images of worn surfaces on the CNx-coated Si_3N_4 balls are shown in Figure 4. Without pre-sliding, the friction coefficient under N_2 gas stream decreased gradually from higher than 0.5 to less than 0.1 after 1000 cycles, which followed by a large fluctuation and spikes. The corresponding optical image of worn surface on the CNx-coated Si_3N_4 ball, as shown in Figure 4a, obviously indicated that the CNx coating worn out on the ball surface after friction test. The contact material combination changed from CNx/CNx to Si_3N_4/CNx after friction test. However, friction coefficients of CNx coatings in N_2 gas stream were greatly decreased with the introduction of pre-sliding. Friction coefficient of CNx/CNx was reduced down to a value of less than 0.05 in the steady stage when N_2 gas was blown to the sliding interface after 1000 cycles of pre-sliding in ambient air.[6] In case of the two-step friction test, when 1000 cycles of pre-sliding was given in step 1 using a Si_3N_4 ball, the friction coefficient of CNx/CNx in step 2 decreased shortly to a steady stage together with a stable and low value of less than 0.01. Furthermore, CNx coating still covered all the wear scar of the ball surface after friction test, as clearly shown in Figure 4b. Few wear particles or transfer films were observed on the wear scar. It was found that the contact material combination of self-mated CNx coating sustains in the friction test with the introduction of two-step ball-on-disk friction test.

The steady-state friction coefficient (μ_s) and corresponding standard deviation of friction coefficient (σ_s) of CNx/CNx in N_2 gas stream with and without pre-sliding are shown in Figure 5. The steady-state friction coefficients were calculated by averaging the measured values of friction coefficients from

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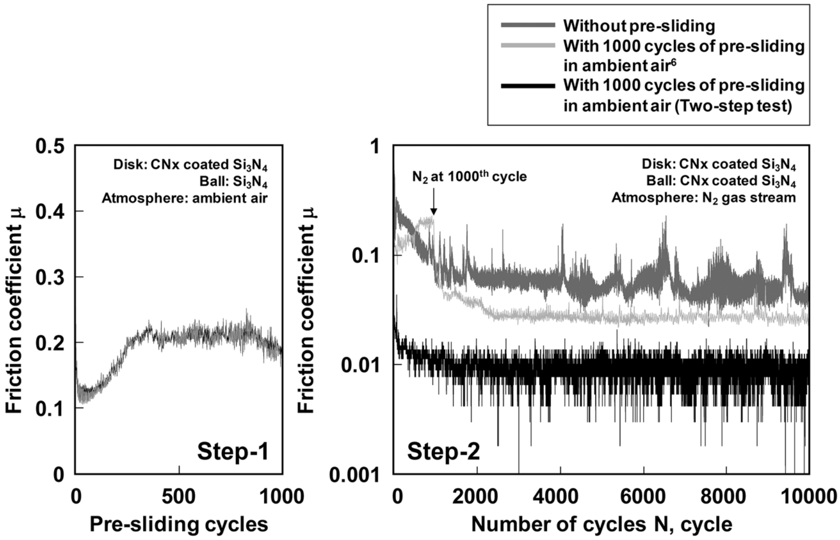

Figure 3. Representative effect of pre-sliding (step 1) on friction behaviour of CNx/CNx in N_2 gas stream (step 2). Friction curves of CNx/CNx in N_2 gas stream without pre-sliding and with pre-sliding in ambient air for 1000 cycles are also shown for reference.

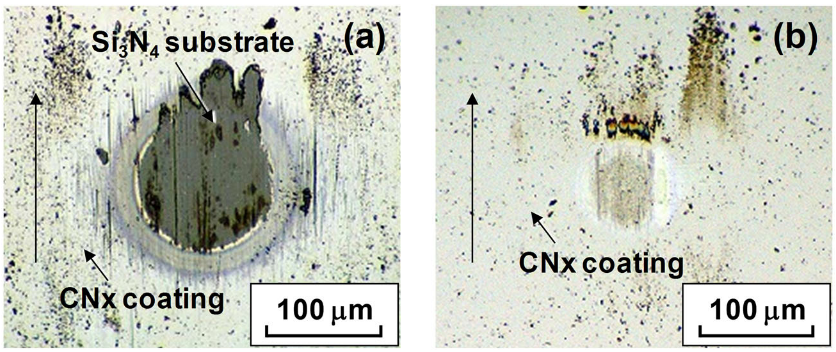

Figure 4. Optical images of wear scars on the CNx-coated Si_3N_4 ball (a) without pre-sliding and (b) with pre-sliding in two-step friction test. The black arrows indicate the sliding direction of the ball.

5000 to 10 000 cycles in each test. The steady-state friction coefficient decreased significantly from 0.07 without pre-sliding to one tenth, 0.007, after 1000 cycles of pre-sliding in ambient air by a Si_3N_4 ball in the two-step friction test. The standard deviation of friction coefficient also decreased greatly to one tenth after pre-sliding, from 0.02 to 0.002. On the other hand, the steady-state friction coefficient of CNx/CNx with 1000 cycles of pre-sliding in ambient air only decreased to 0.043 together with a low standard deviation of 0.003. It was clarified that the two-step ball-on-disk friction test is a simple but effective method for obtaining further stable and low friction coefficients of CNx coatings in N_2 gas stream.

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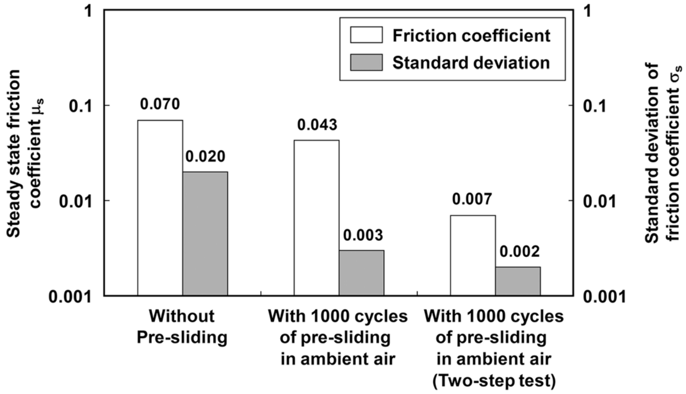

Figure 5. Effect of pre-sliding on steady-state friction coefficient and corresponding standard deviation of friction coefficient of CNx/CNx in N_2 gas stream.

Effects of number of cycles and gas atmospheres for pre-sliding

Two-step friction test has been clarified to be a promising technique for obtaining stable and low frictions of CNx coatings in N_2 gas stream. According to the previous research, the steady-state friction coefficients under N_2 gas stream are greatly affected by the pre-sliding process, especially the pre-sliding cycles and gas atmospheres.[6–8,11] Hence, the effects of pre-sliding cycles (0–1000) and gas atmospheres (N_2, O_2 and ambient air) in step 1 on the friction behaviour of CNx/CNx in N_2 gas stream (step 2) were systematically investigated, and typical results are presented in Figures 6 and 7, respectively. Effect of pre-sliding under various conditions in step 1 on the steady-state friction coefficient in step 2 is summarised in Figure 8. It is

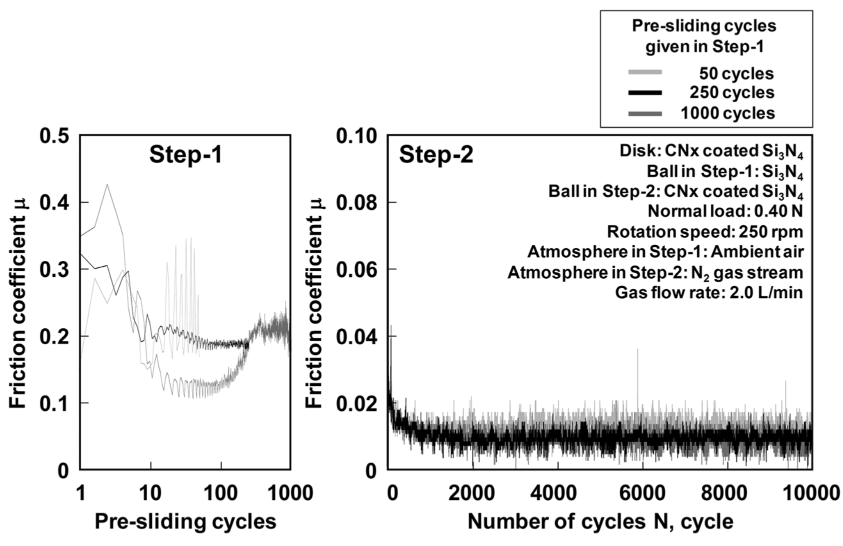

Figure 6. Effect of pre-sliding cycles in ambient air (step 1) on friction behaviour of CNx/CNx in N_2 gas stream (step 2).

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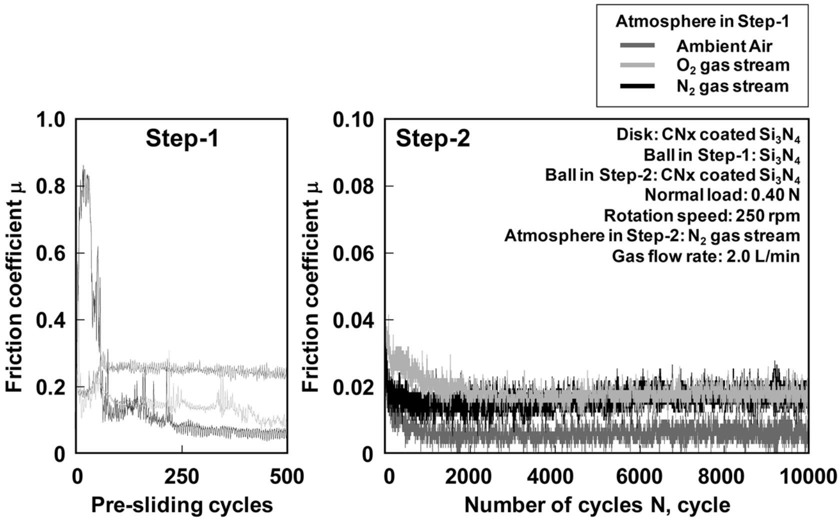

Figure 7. Effect of gas atmosphere in pre-sliding (step 1) on friction behaviour of CNx/CNx in N_2 gas stream (step 2).

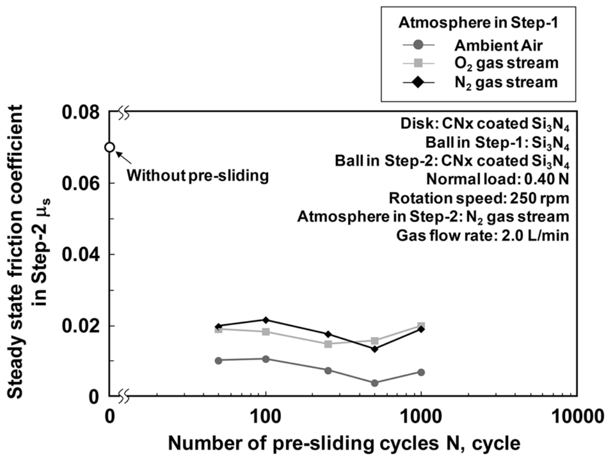

Figure 8. Effects of pre-sliding cycles and gas atmospheres in step 1 on steady-state friction coefficient of CNx/CNx in N_2 gas stream (step 2).

clearly shown that friction coefficients observed in step 2 are much affected by both pre-sliding cycles and gas atmospheres in Step-1. Steady-state friction coefficients decreased greatly from 0.07 without pre-sliding to lower than 0.025 with the introduction of two-step friction test. The condition of pre-sliding in ambient air exhibited lower friction coefficients than those in N_2 and O_2 gas stream, although the friction coefficients varied slightly with different pre-sliding cycles in each pre-sliding gas condition. Particularly, when 500 cycles of pre-sliding in ambient air was given in step 1, minimum friction coefficient

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of 0.004 was observed in step 2. Therefore, it was definitely confirmed that the two-step friction test is a simple but effective technique for achieving stable and low friction coefficients of CNx coatings in N_2 gas stream. Moreover, the optical images of worn surface on the CNx-coated Si_3N_4 ball (not shown here) after all the friction tests are similar to that shown in Figure 4b, namely, CNx coating covered all the wear scar of the ball surface and wear particles can scarcely be observed on the wear scar. It was further clarified that the contact material combination of self-mated CNx coatings persists in all the two-step friction tests.

The finding of self-mated CNx coatings in the two-step friction test can be understood from the following two aspects. On the one hand, the CNx coating commonly wears out in the initial friction process (e.g. 10 cycles) for the sliding contact of CNx/CNx, the contact material combination of self-mated CNx coatings cannot be preserved in the whole sliding period in the ball-on-disk tribosystem.[7] Two-step friction test is proved to be an effective technique for obtaining the contact material combination of self-mated CNx coatings, and it provides a new pathway for studying the potential outstanding tribological properties of self-mated CNx coatings in N_2 gas environment. On the other hand, although the formation of a uniform tribofilm on the wear scar of the ball surface is beneficial for obtaining super-low friction of CNx coatings in N_2 gas environment, the tribofilm is unstable, and it has been found that a random lost or detachment of tribofilm on the ball surface occasionally leading to high and unstable friction coefficients ($\mu > 0.3$).[12] On the contrary, high and unstable friction coefficients were not observed in the current work when the contact material combination of self-mated CNx coatings was generated. Therefore, the stable and low friction behaviour of CNx/CNx in N_2 gas stream can be attributed to the formation of self-mated CNx coatings.

The promising effect of running-in for achieving stable and low frictions of CNx coatings has usually been attributed to several factors, such as removal of oxidised top layer, smoothen of microasperities on the surface and structural changes of the contact interface (including build-up of a homogeneous transfer film on the mating counterface).[2,11,13,14] The existence of oxidised layer can strongly determine the running-in process and the following steady stage.[15,16] However, the removal of oxidised layer is a general phenomenon during the running-in process, and thus, it will not be further discussed in this paper. In order to elucidate the roles of other two factors in the pre-sliding, the effects of polishing of disk surface and relative humidity of ambient air in step 1 on the friction behaviour of CNx/CNx in N_2 gas stream were examined, and the results will be presented in the following section.

Effects of polishing of disk surface and relative humidity of air in step 1

During pre-sliding, microasperities on the initial coating surface are removed, and a flat contact interface is generated, leading to a low interlocking force at the contact interface and therefore is beneficial for the low friction.[11] To verify the role of microasperities, the CNx-coated Si_3N_4 disk was polished and tested without pre-sliding. The polished disk successfully prevented unstable friction phenomenon even without pre-sliding, lower friction coefficient with smaller fluctuation than that of nonpolished disk was observed, as clearly shown in Figure 9. The temporary high friction coefficient during the sliding process could be attributed to the entrance of small wear particles into contact interface, which can have a great influence on the friction behaviour of the smooth contact interface. Nevertheless, the polished disk still cannot give lower friction coefficient than that observed in two-step friction test. Therefore, the role of smoothen of contact interface is not the main reason in the pre-sliding for achieving stable and low friction coefficients of CNx coatings in N_2 gas stream.

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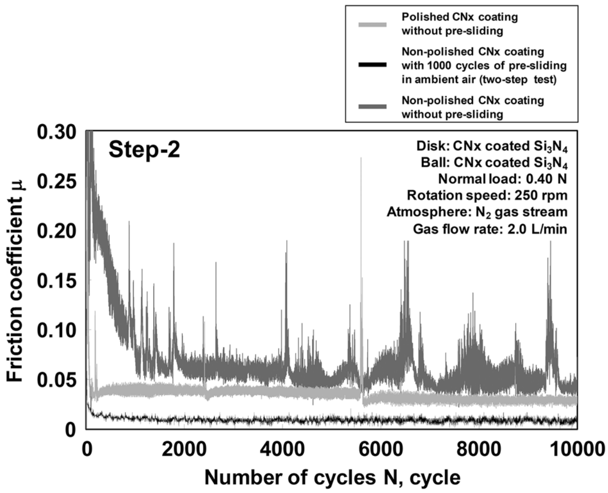

Figure 9. Effect of polishing of CNx-coated Si_3N_4 disk on friction behaviour of CNx/CNx in N_2 gas stream.

The effect of relative humidity of air in step 1 on the friction behaviour of CNx/CNx in N_2 gas stream is shown in Figure 10. Stable and low friction coefficients of less than 0.025 were observed in step 2 under all the three experiments. The steady-state friction coefficients first decreased and then increased with increasing relative humidity from ~10% to ~50% RH in air. The lowest friction coefficient was obtained at relative humidity of ~30% RH. It was suggested that not only gas species but also relative humidity in pre-sliding (step 1) can greatly affect the friction behaviour of CNx/CNx in N_2 gas stream (step 2). Therefore, it is assumed that the structural changes of the contact interface is a major point in the pre-sliding for obtaining stable and low friction coefficients of CNx coatings in N_2

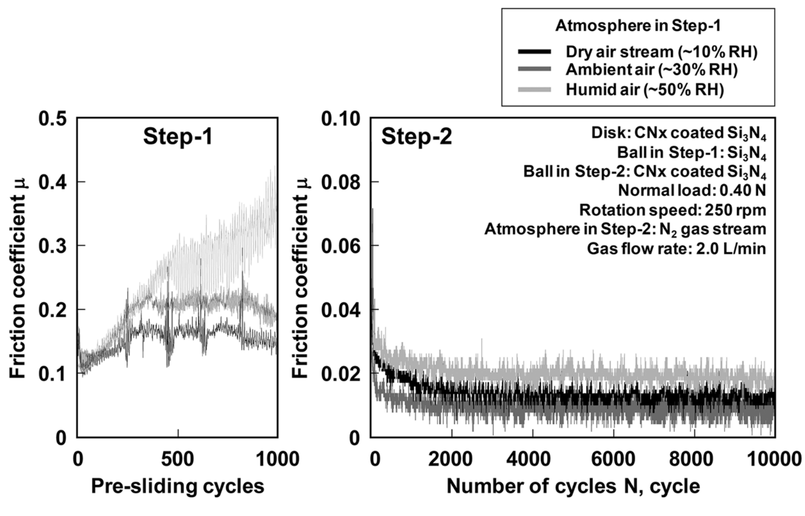

Figure 10. Effect of relative humidity of ambient air in pre-sliding (step 1) on friction behaviour of CNx/CNx in N_2 gas stream (step 2).

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gas stream. As a new CNx-coated Si_3N_4 ball was employed for the friction test in step 2, only the composition and structural changes of the disk surface will be analysed.

Composition and structural changes of disk surface

In order to clarify the composition change of disk surface after pre-sliding, the worn surfaces on the disks after pre-sliding in ambient air, N_2 and O_2 gas with 500 cycles were analysed by X-ray photoelectron spectroscopy, and the atomic concentration of the initial and worn surfaces is shown in Figure 11. A dimensionless parameter 'transformation ratio' was employed to discuss the structural changes of the disk surface. The transformation ratio of N/C or O/C is a comparison of the relative value of N (or O) and C atomic concentration in the worn surfaces and that in the initial surface. The correlation between the transformation ratios of N/C and O/C is shown in Figure 12. The N/C transformation ratio decreased, and the O/C transformation ratio increased on the worn surface after pre-sliding, which suggested that nitrogen desorption and oxygen intake occur on the top surface of the disk.[17] Additionally, nitrogen desorption has been argued to be beneficial for the formation of a graphite-like structure on the top surface of CNx coatings.[18]

To clarify the structural change of disk surface after pre-sliding, these three worn surfaces were also characterised by TEM and a typical TEM cross-sectional image of wear track on the disk surface after 500 cycles pre-sliding in O_2 gas stream is shown in Figure 13. The other two results were identical to that shown in Figure 13. Unfortunately, TEM image does not reveal evidence of graphite-like structure or any other structural change on the top surface of CNx coating. This suggests that structural change was not occurred on the disk surface or the change was too small to be detected by TEM. Recently, partial distribution or noncontinuous lubrication layers have been observed on the contact interfaces, which are strongly claimed to be enough for drastically reducing frictions of CNx coatings in N_2 gas environment[5] and graphene layers on steel surface in air and dry N_2 gas environment.[19,20] Therefore, it is assumed that structural change on the CNx coating will be small if it exists, and the lubrication layer islands on the disk surface are beneficial for achieving stable and low friction coefficients of CNx coatings.

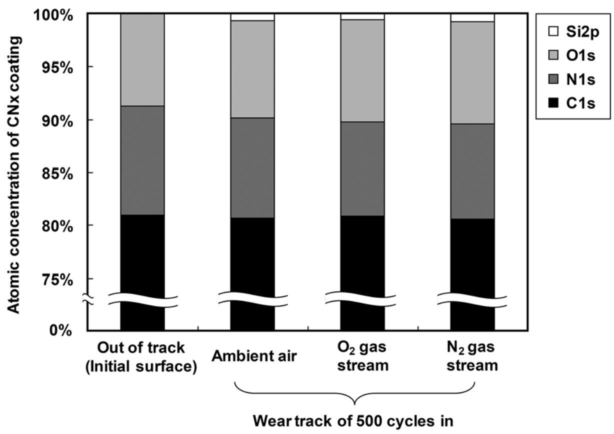

Figure 11. The change of atomic concentration of CNx coating before and after pre-sliding.

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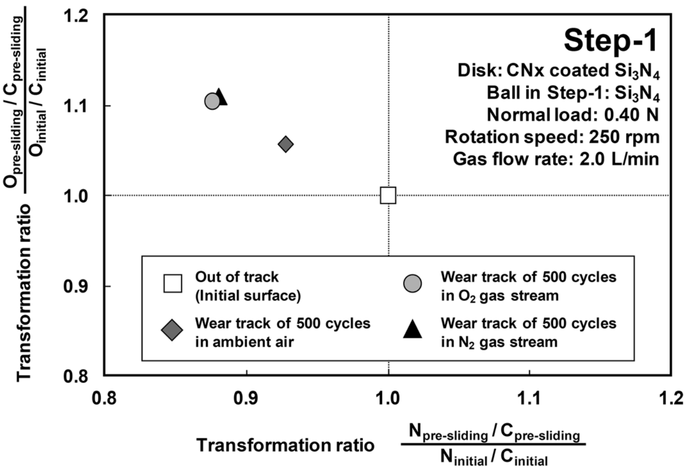

Figure 12. Change of N/C and O/C transformation ratio of CNx surface after pre-sliding under various atmospheric conditions.

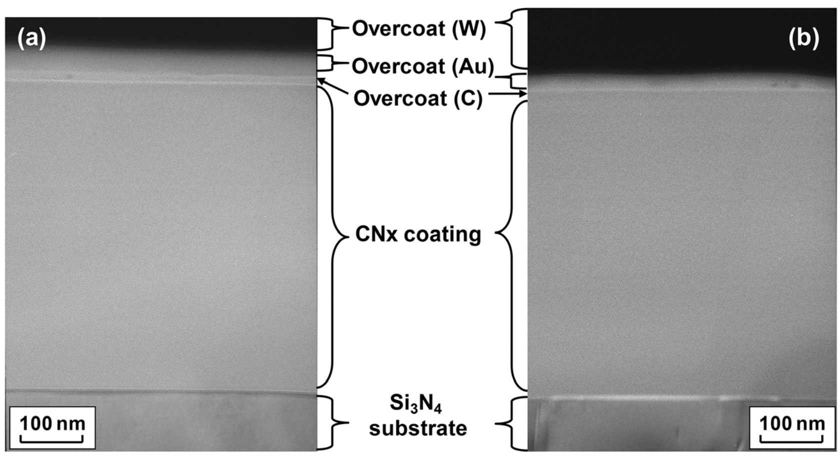

Figure 13. TEM cross-sectional images of CNx coatings before and after pre-sliding. (a) Initial surface and (b) wear track after 500 cycles in O_2 gas stream.

Considering about the evolution of lubrication layer islands on the disk surface in the steady-state friction process, to clearly identify the structural change of disk surface, the worn surface on disk after two-step friction test was analysed by Raman spectroscopy and representative results are shown in Figure 14. The spectrum of initial CNx coating indicated a broad band with two shoulders located at 1350 and 1550 cm^{-1}, corresponding to D band and G band, respectively. The Raman spectra of worn CNx coating on disk surface can be divided into two types; one was identical to that of initial CNx coating; the other was similar to that of the CNx coating with graphitization[4]; a shift of G band peak to a higher wavenumber was obviously identified. It was concluded that the graphite-like

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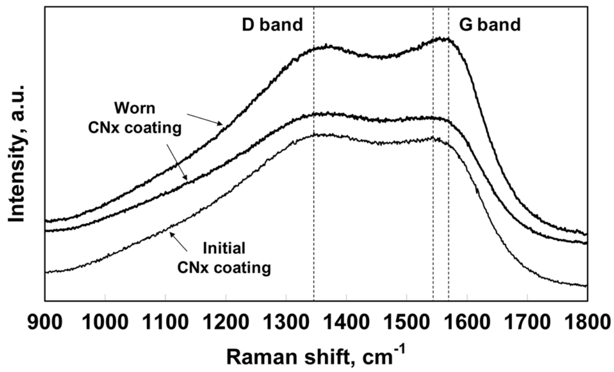

Figure 14. Raman spectra of worn CNx coating on disk surface after two-step friction test in N_2 gas stream. The spectrum of initial CNx coating is also shown for reference.

structure was generated on the top layer of the wear track after two-step friction test. Therefore, in this paper, the suitable running-in process in the two-step friction test results in the formation of a thin lubricious top layer on the sliding surface, facilitating the formation of a graphite-like structure on the disk surface and leading to stable and low friction coefficients of CNx/CNx in N_2 gas stream.

CONCLUSIONS

The effect of running-in process, including pre-sliding with different number of cycles and gas atmospheres, on the friction behaviour of CNx coatings in N_2 gas stream was systematically investigated with a newly introduced two-step ball-on-disk friction test; the following conclusions are drawn:

- The two-step ball-on-disk friction test is clarified to be a promising method for achieving stable and low frictions of CNx coatings in N_2 gas stream. Friction coefficients of CNx/CNx in N_2 gas stream decrease greatly from 0.07 without pre-sliding to less than 0.025 with pre-sliding in two-step friction test. Especially, minimum friction coefficient as low as 0.004 is obtained in N_2 gas stream after giving 500 cycles of pre-sliding to CNx-coated Si_3N_4 disk by a Si_3N_4 ball in ambient air (~30% RH).
- Two-step ball-on-disk friction test is proved to be an effective technique for obtaining the contact material combination of self-mated CNx coatings, which provide a new pathway for studying the potential outstanding tribological performances of self-mated CNx coatings in N_2 gas environment.
- The stable and low frictions of CNx/CNx in N_2 gas stream after proper pre-sliding are mainly attributed to the generation of self-mated CNx coatings on the contact interface and the formation of a lubricious top layer on the disk surface during the pre-sliding, which facilitate the formation of a graphite-like structure on the disk surface and lead to stable and low friction coefficients.

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