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The tribological performance of Ti(C,N)-based cermet sliding against Si_3N_4 in water

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ABSTRACT

Ti(C,N)-based cermets have been attracting much attention because of their good high temperature strength, perfect chemical stability, excellent wear resistance, and relatively better machinability. In this paper, the tribological performance of Ti(C,N)/Si_3N_4 sliding pairs lubricated in water was investigated using a ball-on-disk tribometer at sliding speed of 200 mm/s. The normal loads are controlled at 2 and 5 N, respectively. The experimental results show that, at the normal load of 2 N, ultra-low friction coefficient ($\mu < 0.005$) is obtained at each experiment. At the normal load of 5 N, two kinds of experimental phenomena were observed. One is that an extremely low friction coefficient is achieved within the running-in period of 12 h. The other shows no ultra-low friction after sliding in water even for 24 h. It is proposed that ultra-low friction coefficient mainly owes to hydrodynamic lubrication caused by surface smoothing. The wear mechanism is tribochemical wear while mechanical wear may be the dominating factor in the high friction case at the normal load of 5 N.

1. Introduction

Nowadays, environmental pollution has become a more and more serious problem to human beings. Every year, a vast quantity of industrial lubricants enters the environment, which may be a primary source of pollution. Seeking for a green lubricant remains a great challenge for world. Water lubrication can be an effective way to partially address environmental issues.

In the past decade, there has been a growing interest on the friction properties of ceramics lubricated in water [1,2]. Non-oxide ceramics such as Si_3N_4 and SiC, in particular, are candidates for tribological materials because the self-mated tribopairs in water all have shown low friction coefficient ($\mu < 0.01$) under suitable running-in condition [3–6]. Hsu and Shen [7] investigated the effects of sliding speed and mean contact pressure on wear of self-mated SiC and Si_3N_4 using a pin-on-disk tester and set up wear maps of these ceramics under water lubrication. Xu and Kato [8] studied the formation of a tribochemical layer on silicon nitride sliding in water and the results showed that the silica tribochemical layer formed on the friction surface reduces the friction. In addition, Wong et al. investigated the water lubrication of four kinds of ceramics, Si_3N_4/Si_3N_4, SiC/SiC, Al_2O_3/Al_2O_3 and ZrO_2/ZrO_2, with a ring-on-disc apparatus [9,10]. The results showed that SiC/SiC has the largest lubrication area and is the best combination for water lubrication.

Until now, only SiC/SiC tribopairs have been used as water lubricated sliding bearings or mechanical water seals owing to its higher wear-resistance against abrasive wear. However, due to the long running-in period for SiC/SiC tribopairs in water and the easy occurrence of hydration reaction between SiC (Si_3N_4) and water, severe wear was easily observed for the self-mated SiC (Si_3N_4) tribopairs in water [11]. Moreover, ceramics materials are expensive, hard to machine and sensitive to crack, which seriously restrict their applications.

Cermets, commonly used to describe TiC or Ti(C,N) alloys, have been mostly used for finishing turning operations [12]. At present, much attention was paid to them because of their excellent wear-resistance, good high temperature hardness, perfect chemical stability and very low friction coefficient to metals as well as superior thermal deformation resistance [13]. In addition, the machinability of cermets is better than traditional ceramics.

The tribological properties of Ti(C,N) cermet material in different experimental conditions have been reported in several previous publications. Jeon et al. [14] studied the anti-wear properties of cermet Ti(C,N) at the temperature of 873 K. Under dry sliding conditions, the influence of chemical composition of TiC cermets on wear rate against a steel disk were studied by Pirso et al. [15]. The tribological performance of Ti(C,N)/1045 steel pairs sliding in oil was reported in ref. [16] and it was found that intensive adhesion occurred between the Ti(C,N) and steel surface and the adhesion

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Table 1
Mechanical properties of specimen.

Materials	Ti(C,N) disk	Si_3N_4 ball
Production process	Sintered	HIP
Density, ρ ($\times 10^3$ kg/m^3)	6.0	3.3
Vickers hardness, *Hv*	1420	1053
Fracture toughness, K_{IC} (MPa/m$^{1/2}$)	15	6

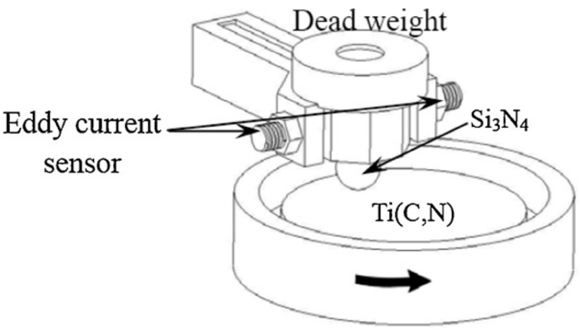


Fig. 1. The scheme of experimental apparatus.

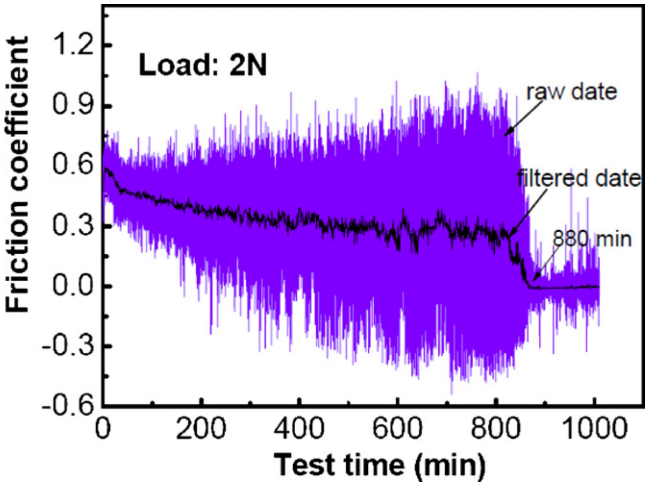


Fig. 2. The variation of friction curve at the normal load of 2 N in water (raw data and filtered data).

junctions were continuously broken with the relative movement of the rubbing pairs.

With the development of science and technology, energy-saving and pollution-reducing have increasingly become the most important trends in machine design. As mentioned before, the friction properties of ceramics in water have attracted keen attention for development of water-lubricated ceramic bearing. However, few reports have been found about the tribological performance of Ti(C,N) under water lubrication. Can ultra-low friction coefficient also be obtained for cermet Ti(C,N) in water? What is the running-in properties of Ti(C,N) in water? There is still no available knowledge about this. Therefore, the present paper focuses on the tribological properties of Ti(C,N)-based cermet under water lubrication. Traditional water-lubricated ceramic Si_3N_4 was selected as sliding pair. The experiments were performed on a ball-on-disk tribometer. More attention was paid to the running-in process of cermet/ceramic rubbing pairs in water based on the observation of their friction coefficients and worn surfaces.

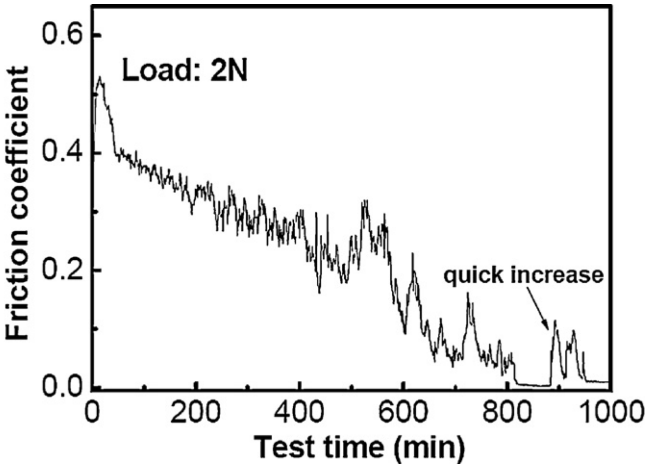


Fig. 3. The friction curve at the load of 2 N, lubricated in water (a shake of the ball leads to a quick increase of the friction).

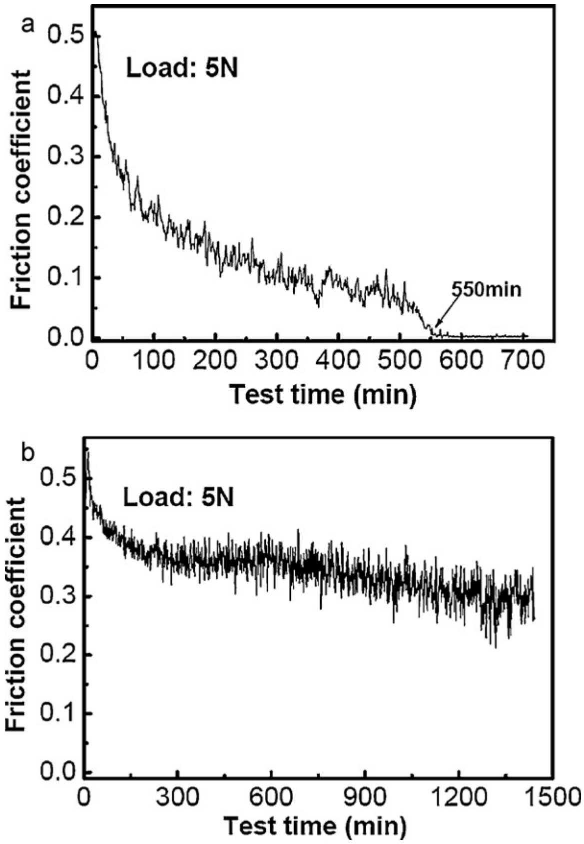


Fig. 4. The friction curve of Ti(C,N)/Si_3N_4 tribopairs at the load of 5 N. (a) Low friction case and (b) high friction case.

Table 2
Composition and particle sizes of the raw materials.

Powder	TiC		TiN		Ni	Mo	WC	Cr_3C_2	C
Particle size (μm)	2.5	20.0×10^{-3}	3.0	14.0×10^{-3}	1.7	3.2	4.5	3.4	5.5
Mass fraction (wt.%)	38.5	2.0	11.4	0.6	25.0	16.0	4.8	0.6	1.1

2. Experimental procedures

2.1. Materials

The materials used in this investigation are characterised in Table 1. The Ti(C,N)-based cermet was produced by vacuum sintering. The composition and particle sizes of the raw materials were shown in Table 2. There are two kinds of particles size of the TiC (2.5 μm, 20 nm) and TiN (3 μm, 14 nm), respectively. The mixed powders were put into a steel mould and pressed into a circular disk. The pressing parameters were as follows: pressure 270 Mpa, duration time 60 s. Sintering was carried out using a furnace at temperature up to 1400 °C with holding time of 4.5 h. The density of the sintered body was determined by the Archimedes method. The structure of sintered Ti(C,N) is dense and the mean carbide grain size is about 3 μm. The surfaces of the sintered samples were machined with a diamond tool and finally polished. The ball material was single phase sintered silicon nitride prepared by Shanghai Research Institute of Material.

2.2. Friction and wear tests

Friction tests were carried out using a ball-on-disk tester. Fig. 1 shows the apparatus used in this experiment. The lower specimen was the disk of Ti(C,N)-based cermet with a diameter of 32 mm and thickness of 7 mm. The surface roughness Ra is less than 53 nm after polishing. The upper specimen is a Si_3N_4 ball, which has a diameter of 8 mm, roughness Ra less than 14 nm. The ball was stationary, held by the arm. The normal load was applied on the ball by dead weight. The disk was driven by a motor with an adjustable rotational speed. The friction coefficient was obtained by measuring the strain of the arm caused by friction force.

Table 3
Test conditions.

Sliding speeds (mm/s)	200
Normal loads (N)	2, 5
Time (min)	720–1440
Temperature (°C)	20
Lubricants	Purified water

Experimental conditions were shown in Table 3. All specimens were cleaned with acetone in an ultrasonic bath for 10 min. The wear scars on disks and balls' surface were studied by scanning electron microscopy (Quanta200); energy dispersive spectrometry was used to detect the elements present on wear scar surfaces. The surface profiles of the wear tracks were measured using a MicroXAM surface map microscope. Cross-section areas of wear track on disk (*A*) were also determined at four different positions for reliable average by MicroXAM surface map microscope. The diameter of wear scar on Si_3N_4 balls under each condition was mea-

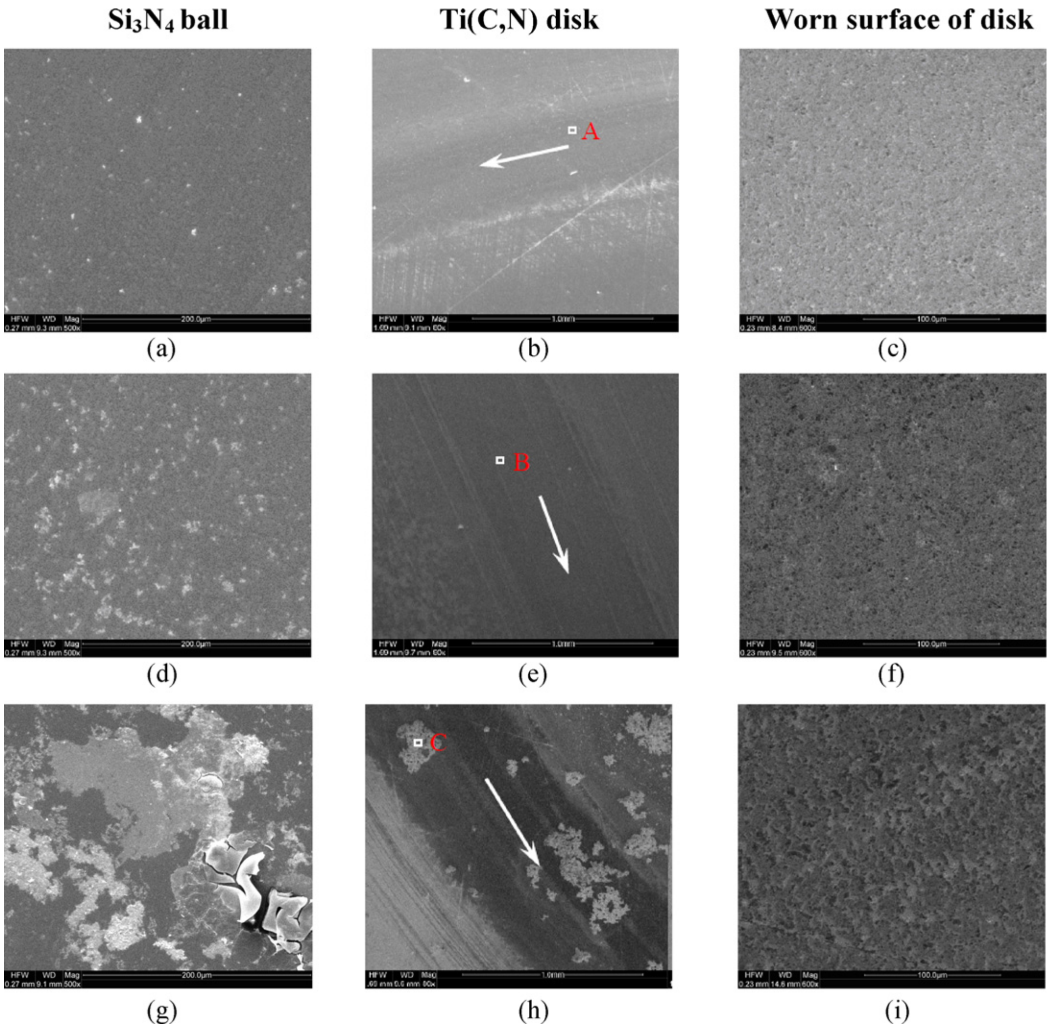
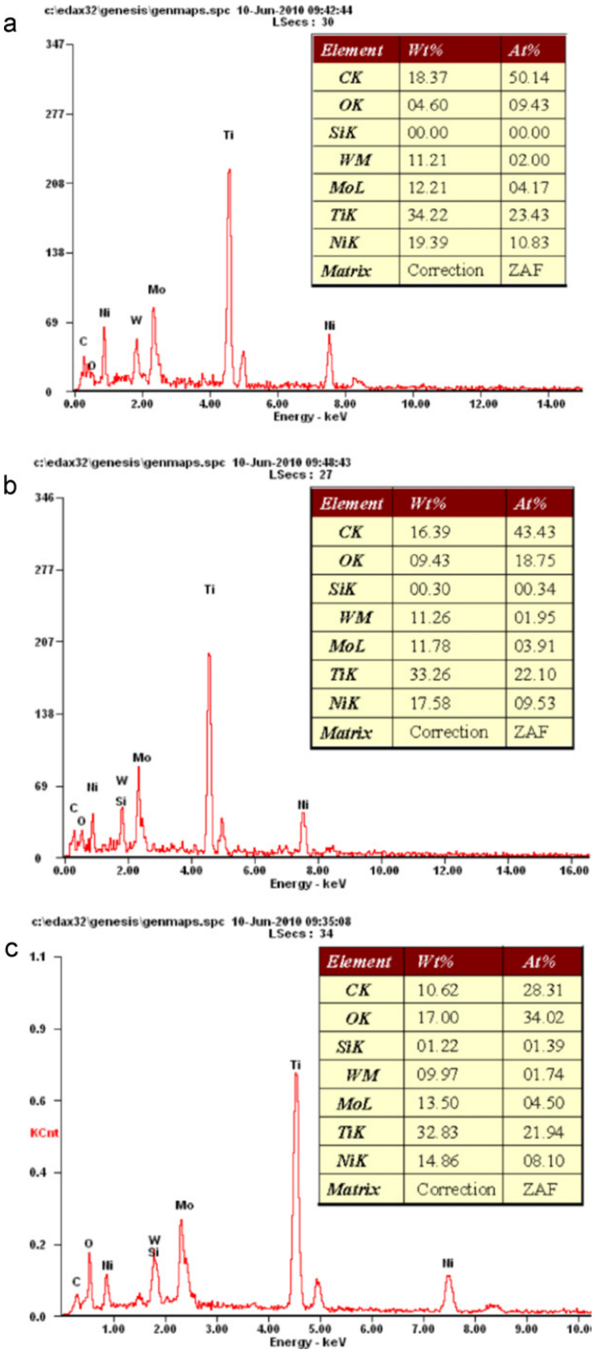


Fig. 5. Scanning electron micrographs of the worn surface of Si_3N_4 and mated disk. (a)–(c), corresponding to Fig. 2 low friction case; (d)–(f), corresponding to Fig. 4a, low friction case; (g)–(i), corresponding to Fig. 4b, high friction case.



a

Element	Wt%	At%
CK	18.37	50.14
OK	04.60	09.43
SiK	00.00	00.00
WM	11.21	02.00
MoL	12.21	04.17
TiK	34.22	23.43
NiK	19.39	10.83
Matrix	Correction	ZAF

b

Element	Wt%	At%
CK	16.39	43.43
OK	09.43	18.75
SiK	00.30	00.34
WM	11.26	01.95
MoL	11.78	03.91
TiK	33.26	22.10
NiK	17.58	09.53
Matrix	Correction	ZAF

c

Element	Wt%	At%
CK	10.62	28.31
OK	17.00	34.02
SiK	01.22	01.39
WM	09.97	01.74
MoL	13.50	04.50
TiK	32.83	21.94
NiK	14.86	08.10
Matrix	Correction	ZAF

Fig. 6. EDX spectrum in the wear tracks of the disks as seen in Fig. 5b, e and h, respectively. (a) corresponding to A; (b) corresponding to B; (c) corresponding to C.

sured using a Mitutoyo optical microscope. Thus, the specific wear volume for balls and disks were determined using the following equations:

$$w_{s,ball} = \frac{\pi d^4}{64R} \tag{1}$$

$$w_{s,disk} = 2\pi r A \tag{2}$$

where R is the Si_3N_4 ball radius, d is the diameter of wear scar on the ball, r is the wear track radius on the disk, A is the cross-section areas of wear track on disk. All friction experiments were repeated for three times, and the wear volume here was the mean value of the three times.

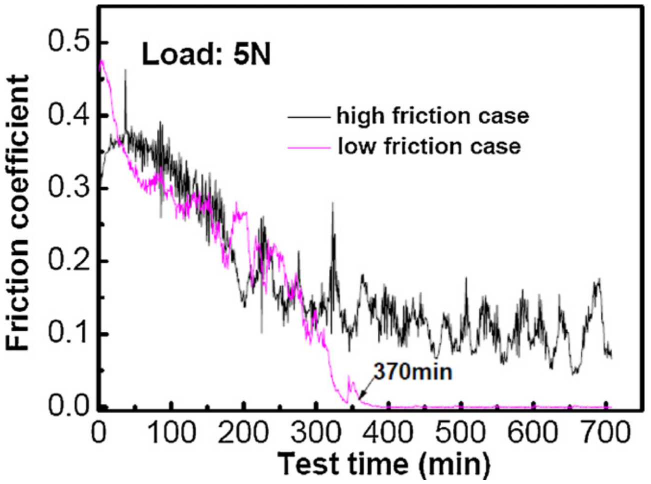


Fig. 7. The friction curve of Ti(C,N)/Si_3N_4 tribopairs at the load of 5 N in water – two cases.

3. Results and discussion

Fig. 2 shows the variation of the friction coefficients of Ti(C,N)/Si_3N_4 pairs with sliding time in water at the normal load of 2 N. Severe stick-slip occurs at the beginning of the test and the friction force response during stick-slip is strongly dependent on the compliance and speed control characteristics of the tribometer. According to Ref. [17], the raw data is treated using a 60-point moving average and the result is also shown in Fig. 2. The friction curve is smoother after filtering. As seen in Fig. 2, filtering has little effect on the plotted data once low friction sliding starts and all the dates of the friction curves in the following text were filtered.

It can be seen in Fig. 2 that the friction coefficient decreases gradually at the beginning of the test. Sharp decrease of the fric-

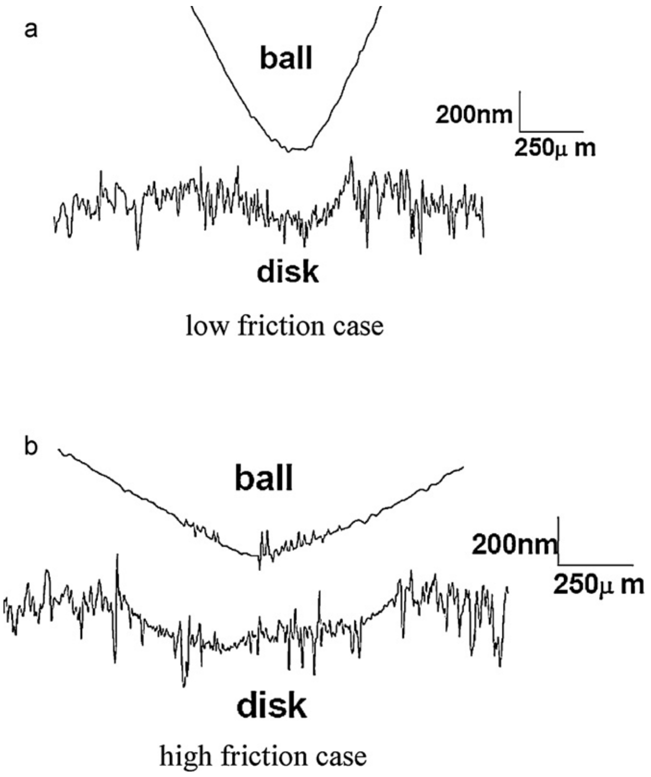


Fig. 8. Two kinds of typical specimens surface profiles after sliding in water for 720 min at the normal load of 5 N.

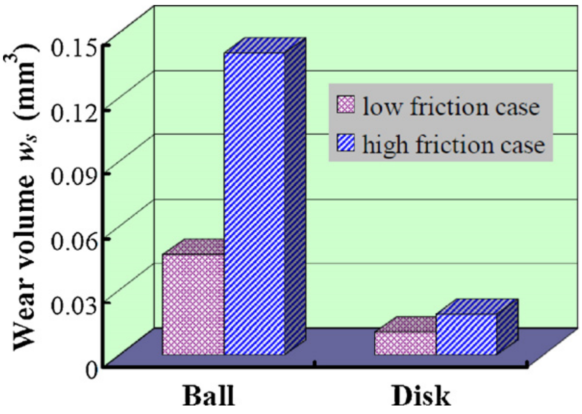


Fig. 9. Comparison of specimens wear volume between high and low friction case.

tion coefficient was observed after sliding in water for 880 min. The initial friction coefficient of Ti(C,N)/Si_3N_4 pairs is about 0.6 while the mean steady-state coefficient after running-in in water is about 0.001, which is lower than the resolution of the measurement (0.005). According to [18], the contact at the beginning is Hertzian; the small contact area and high contact pressure lead to the high friction. Wear creates a smooth and flat surface on the Si_3N_4 ball so that a flat-on-flat friction is established. The result is a reduction of the local stresses responsible for the mechanical wear. When the area of the wear scar is large enough, the friction may drop gradually. The final surface roughness (Ra_{max}) of Ti(C,N) in the wear track was about 45 nm, which is less than the initial roughness. It means that the wear track is smoother than the disk's surface and there exists a polishing effect during the running-in process at the normal load of 2 N.

During the experiment, it was found that when the coefficient is less than 0.005, a slight shake of the ball could lead to a quick increase of the friction (seen Fig. 3). When the rubbing process is stable, friction coefficient gradually decreases to the extremely low value again. As Ref. [3] pointed that slight shake disturbed the previous hydrodynamic lubrication state and solid to solid contact happened, which caused the high friction. After a period time of sliding, the hydrodynamic lubrication was formed again for the worn of the samples, thus, the ultra-low friction was achieved.

At the load of 5 N, two typical variations of the friction coefficient during the running-in process were observed at the same conditions (see Fig. 4a and b). One typical curve is that the friction coefficient decreases gradually with the increasing of sliding distance and then drops to a value of about 0.001. It costs about 550 min to achieve the low friction. The other is that the friction coefficient decreases gradually from 0.54 and finally keeps at a high value of about 0.30 until the end of the test. To ensure the reliable running-in time, the tests were carried out over 1440 min.

Experiments, at the normal load of 5 N, were carried out more than 40 times, among which the appearance probability of the low friction cases was about 20%. This result shows that, under most conditions, ultra-low friction of Ti(C,N)/Si_3N_4 tribopairs cannot appear at the load of 5 N sliding in water.

The SEM images of the worn surfaces of the specimens corresponding to Figs. 2 and 4a and b at the end of the running-in process were shown in Fig. 5, respectively. The arrows show the sliding direction of the counterface. In Fig. 5a, small wear particles dispersed on the worn surface of the Si_3N_4 ball at the load of 2 N. A little larger wear particles appeared on the ball's wear scar when the normal load increased to 5 N in the case of low friction (see Fig. 5d). In Fig. 5b, a few original scratches still remained in the wear track, which suggests that the depth of wear was considerably less than the depth of the deepest scratches.

For the two low friction cases, the contact surfaces become very smooth and flat after different running-in periods and the worn surfaces seem to be compact (see Fig. 5c and f). For Si_3N_4/Si_3N_4 tribopairs, Tomizawa and Fischer [3] attributed the smoothing process to tribochemical wear. Smooth surface is necessary to generate hydrodynamic lubrication, which maintains a low friction coefficient.

For the high friction case, typical morphology (see Fig. 5g) of wear particles in the form of a sheet were observed on worn surface of Si_3N_4 ball at the load of 5 N after 24 h running-in process. From Fig. 5g, plastic deformation, delamination and brittle fracture can be seen on the wear scar. Many wear particles deposited on the worn surface of Ti(C,N) disk (see Fig. 5h). Some of the particles were located at the edges of the wear tracks and a few of large particles were still attached to the bulk material surface. It can be seen that there are waviness and many cavities on the disk's worn surface (see Fig. 5i). These cavities are distributed in the sliding direction on the worn surface, which may attribute to severe wear. The observed high friction is probably associated with the wear particles and cavities. It can be deduced that, for the high friction case, the wear mechanism may be mechanical wear at the normal load of 5 N.

Fig. 6 shows the EDX spectrum corresponding to parts A, B and C on the wear tracks of the disks as seen in Fig. 5. No Si element was detected on the wear track at the normal load of 2 N (Fig. 6a). Silicon content in Fig. 6b is relatively lower compared with that of Fig. 6c. The highest content of silicon is detected for the wear particles in the high friction case. As Refs. [3,19] pointed that, for self-mated Si_3N_4, low friction coefficient is only resulted from tribochemical wear and the majority of wear particles generated by tribochemical wear dissolve in water.

For the high friction case, lots of wear particles (see Fig. 5 h) cannot dissolve in water, which may lead to the high friction. It also suggests that the wear particles might be SiO_2 according to the elements content of silicon and oxygen (see Fig. 6c).

In order to know the influence of Si_3N_4 on the specific running-in process of Ti(C,N) disk in water at the load of 5 N, the detailed wear behavior of Si_3N_4/Ti(C,N) tribopairs was carried out. The wear tracks of Si_3N_4 ball and Ti(C,N) disk, after sliding for 720 min, were analyzed at the load of 5 N. Two kinds of the experimental results were analyzed according to the final friction coefficient: (1) low friction case and (2) high friction case.

Fig. 7 shows two kinds of the friction curves at the load of 5 N lubricated in water. The test time is controlled at 720 min. It cost about 370 min for Si_3N_4/Ti(C,N) tribopairs to reach ultra-low friction, which is shorter than the running-in process in Fig. 4a. For the high friction case, the friction coefficient always maintains at 0.1 or so after 720 min test. We observed that the time spending to get low friction is random variations, but in most cases, it is less than 600 min during all of the experiments.

Fig. 8 gives the surface profiles of the balls and disks corresponding to the two curves in Fig. 7. Fig. 8a is the surface profile of the low friction case. Compared with high friction case, the wear scar on the ball is relatively smooth in this condition. A fairly rougher surface of the ball (Fig. 8b) was obtained in the case of high friction. As mentioned above, the roughness of the two wear tracks on the disks is less than the initial roughness of the surface, which may be due to the polishing effect during the running-in process. In addition, there is no obvious difference between the roughness of the two cross-section worn surfaces, which may come from the higher hardness of Ti(C,N) disk.

Because of the possibility of higher wear rate during the high friction running-in period, comparison of the wear rates based on the total sliding time is misleading. Therefore, the total wear volume of the samples within 720 min running-in process was calculated as shown in Fig. 9. The wear volume of the balls is much higher than that of the disks' for both the high and low friction case. It indicates that wear resistance of Ti(C,N)-based cermet is much higher than that of Si_3N_4 material, which may result from the higher hardness of cermet. Corresponding to the low friction case, wear volume of the ball and disk are both less than that of high friction case. The value of $w_{s,ball}$ in high friction case is about three times larger than in low friction case, which may result from the different wear mechanism. The difference of $w_{s,disk}$ between the two disks is relatively small, but it is still slightly higher for the high friction case.

4. Conclusions

According to the previous reports, it can be seen that severe wear was easily observed for the self-mated ceramics SiC (Si_3N_4) tribopairs in water. Besides, ceramics materials possess self weakness such as hard to machine and sensitive to crack. The characteristics of Ti(C,N)-based cermet may remedy the defects of ceramics at a certain extent. In this paper, the running-in tests of Ti(C,N)-based cermet sliding against Si_3N_4 in water were conducted with a ball-on-disk apparatus. The sliding speed is 200 mm/s and the normal loads are 2 and 5 N, respectively. The main results are as follows:

(1) When the normal load is 2 N, ultra-low friction coefficient less than 0.005 can be achieved.
(2) At the higher load of 5 N, two kinds of results were obtained: (a) low friction case ($\mu < 0.005$); (b) high friction case ($\mu > 0.1$). During the whole times of experiments, the appearance probability of the low friction case was about 20%.
(3) Low friction can only be obtained after a much smoother worn surface achieved. The smoothing process may be attributed to tribochemical wear. Mechanical wear could be the dominating factors at the high friction case.
(4) At the load of 5 N, the wear volume of Si_3N_4 ball is much higher in the high friction case than that of low friction case. While the difference of wear volume between the two disks is relatively small, which may due to the higher hardness of Ti(C,N)-based cermet.

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