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ARTICLE

Tribological Behavior of 1Cr18Ni9Ti Steel under Hydrogen Peroxide Solution against Different Ceramic Counterparts

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Abstract: We investigated the effect of counterface of ZrO_2, Si_3N_4 and SiC ceramics on the tribological behavior of 1Cr18Ni9Ti steel in 90% hydrogen peroxide solution. The results show that the tribological properties of 1Cr18Ni9Ti steel are strongly dependent on the counterfaces. The adhesion behavior affects the tribocouple of 1Cr18Ni9Ti/ZrO_2, leading to a high coefficient of friction (COF) fluctuating from 0.17 to 0.27 and the highest wear loss of 1Cr18Ni9Ti steel. The oxidation and hydrolysis protect the worn surface of 1Cr18Ni9Ti/SiC, inducing a low COF 0.035 and the lowest wear loss of 1Cr18Ni9Ti steel. Both the adhesion behavior and the reactions play important roles in the wear behavior of 1Cr18Ni9Ti/Si_3N_4, leading to a complex COF and intermediate wear loss of 1Cr18Ni9Ti steel. As for the counterparts, ZrO_2 ceramic shows the most severe wear, SiC ceramic shows relatively low wear volume, and Si_3N_4 ceramic presents the lowest wear volume.

Key words: hydrogen peroxide; tribological property; counterpart; tribochemistry; adhesion

High concentration hydrogen peroxide (HCP), including hydrogen peroxide concentrations ranging from 70% to 98% is receiving renewed interest as a monopropellant and the oxidizer for bipropellant systems [1]. However, the strong oxidizing property of H_2O_2 determines the incompatibility of most alloys and excessive wear of tribo-pairs [2,3]. Pure Al and 1Cr18Ni9Ti stainless steel are the rarely compatible materials of H_2O_2[4]. Compared with the soft pure Al, 1Cr18Ni9Ti stainless steel with good mechanical strength, processing property and corrosion resistance, is potential to preserve nice wear-resistance and be moving parts in H_2O_2 solution[5,6]. Wear, as a complex phenomenon during the surface interaction, is affected by the structures of both materials in the tribo-pair[7,8]. Studying the wear mechanism of different materials coupled with 1Cr18Ni9Ti stainless to design the tribo-pairs with nice tribological property in HCP, is important for the new propulsion systems.

C. Q. Yuan *et al* found that the 1Cr18Ni9Ti steel/Si_3N_4 ceramic rubbing pair could preserve nice wear-resistance[3]. In addition, it was reported that SiC ceramic also could preserve low COF (coefficient of friction) and wear loss in HCP [9]. With nice compatibility in H_2O_2 solution [5] and good wear-resistance [10,11], SiC and Si_3N_4 ceramics are potential to be the good counterpart of 1Cr18Ni9Ti steel in the HCP propellant systems. However, very few papers focused on the difference between the wear mechanism of Si_3N_4 and SiC ceramics in HCP.

Aiming at understanding the aforementioned points, we investigated the tribological properties of 1Cr18Ni9Ti stainless steel sliding against ZrO_2, Si_3N_4 and SiC ceramics in 90% hydrogen peroxide solution. The wear mechanisms were studied by the worn surfaces of both 1Cr18Ni9Ti stainless and corresponding ceramics.

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1 Experiment

The XRD pattern and chemical composition (wt%) of 1Cr18Ni9Ti steel are presented in Fig.1. The tribological tests were conducted on a SST-ST pin-on-disc tribometer (Wazau Germany) in 90% hydrogen peroxide solution at average room temperature of (298±2) K. In order to insure required face-to-face mating and perpendicularity between the pin and disc, the ingots were fabricated into pin specimens of *Φ*8 mm×15 mm, and then they were carefully polished on 2000# abrasive paper before test. The ZrO_2 ceramic was selected to be the yttrium stabilized zirconium dioxide (YSZ) with tetragonal structure. The physical and mechanical properties of the ceramic materials are shown in Table 1. The surface roughness (*R*a) of 1Cr18Ni9Ti stainless steel and ZrO_2, SiC, Si_3N_4 ceramic discs were polished to 0.2~0.25 μm. The tests were carried out at an applied load of 35 N, sliding speed of 300 r/min (0.690 m/s), and testing time of 30 min.

The wear calculations of discs and pins were evaluated by wear volume and mass loss, respectively. The radius of the wear track was 22.5 mm. As for the counterpart discs, the cross-section profile of worn surface was measured using white light confocal microscope (CM, Micromeasure2 STLE). The wear volume, $V=AL$, was defined as the cross-section area of wear track (A) multiplied by the circumference of the worn track (L). Four locations were measured in each wear track to determine A. With regards to the pins of the 1Cr18Ni9Ti steel, the mass before and after the test was both measured for calculating the wear loss. To make sure the reproducibility of the experimental results, all the tribological tests were carried out at least three times. Morphologies of the worn surfaces of the alloys and ceramics were analyzed by scanning electron microscopy (SEM JSM-6510LV) equipped with energy dispersive spectrometry (EDS).

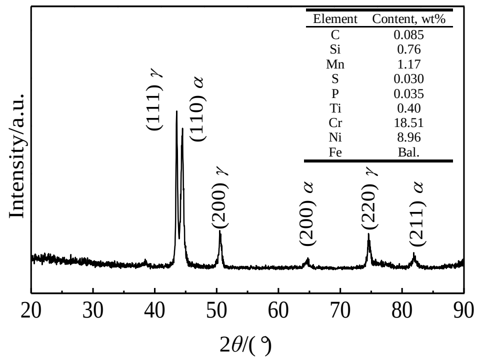

Fig.1 XRD pattern and chemical composition of 1Cr18Ni9Ti steel

Table 1 Physical and mechanical properties of the ceramic

Ceramic	Density/ g cm^{-3}	Hardness, HR_{45N}	Young's modulus /GPa	Fracture toughness /MPa m$^{1/2}$	Thermal conductively/ W (m K)$^{-1}$
ZrO_2	5.8	84	225	7.13	2.5
SiC	3.1	93	441	4.65	58.6
Si_3N_4	3.2	87	294	4.71	12.6

2 Results and Discussion

Fig.2 shows the coefficient of friction (COF) curves of the 1Cr18Ni9Ti steel sliding against different counterparts at applied load of 35 N and sliding speed of 0.690 m/s with sliding time of 30 min. The COF of the 1Cr18Ni9Ti steel/ZrO_2 ceramic pair keeps the stabilization with about a high value fluctuating between 0.17 and 0.27 during the test process. For the 1Cr18Ni9Ti steel/SiC ceramic pair, the COF curve shows a gradual decreasing trend within 250 s at the initiatory stage, and then presents an excitingly low value of about 0.035 with the increase of time. In contrast to the smooth characteristics against ZrO_2 and SiC ceramics, the COF curve of 1Cr18Ni9Ti steel rubbing against Si_3N_4 ceramic is complex. In order to clearly describe the experimental results, the complex COF curve of Si_3N_4 ceramic is decomposed into two regimes in the present work. α regime keeps the high value with distinct fluctuation similar to ZrO_2 ceramic. β regime is a complex process where the COF firstly decreases to the low value similar to SiC ceramic, and then increases sharply to the high value. In the COF curve of 1Cr18Ni9Ti steel/Si_3N_4 ceramic pair, α and β regimes present alternately. With the continuation of sliding process, the duration of α regime decreases gradually. Namely, α regimes displayed in the test maintain at about 600, 400 and 50 s, successively. The different friction behavior indicates that the counterpart plays an important role in the tribological behavior of 1Cr18Ni9Ti steel in 90% hydrogen peroxide solution.

Fig.3 illustrates the wear loss of the 1Cr18Ni9Ti steel pins and the wear volume of the three different ceramic discs. Both the wear loss of pin and the wear volume of disc in the 1Cr18Ni9Ti steel/ZrO_2 ceramic pair are obviously higher than those in other pairs. With low total wear loss, the tribo-pairs with SiC and Si_3N_4 ceramics exhibit different wear properties of steel and ceramic. The wear loss of 1Cr18Ni9Ti steel rubbing against SiC ceramic, is the lowest, which is

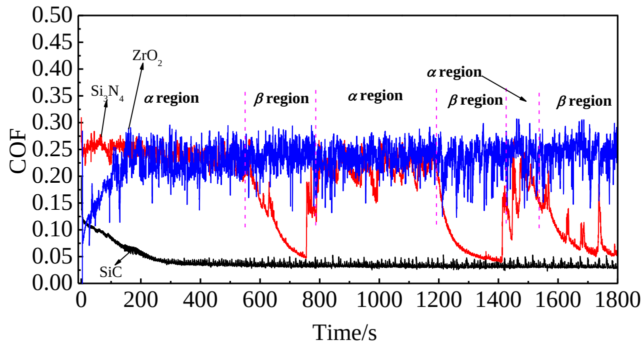

Fig.2 COF curves of the 1Cr18Ni9Ti steel sliding against ZrO_2, Si_3N_4 and SiC ceramics at 35 N and 0.690 m/s with sliding time of 30 min

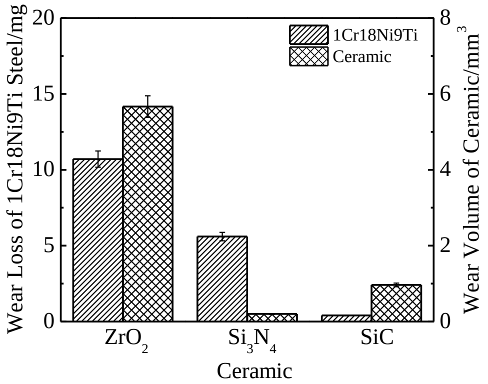

Fig.3 Wear loss of 1Cr18Ni9Ti steel pins, and wear volume of different ceramic discs

about one-fourteenth of that against Si_3N_4 ceramic. With regard to the ceramics, the wear volume of Si_3N_4 ceramic is the lowest, which is about 5 times lower than that of SiC ceramic. The mathematical relationship among the wear loss of 1Cr18Ni9Ti steel against different ceramics is ZrO_2 (10.7 mg) > Si_3N_4 (5.6 mg) > SiC (0.4 mg), and that among the wear volume of the ceramics is ZrO_2 (5.67 mm^3) > SiC (0.47 mm^3) > Si_3N_4 (0.21 mm^3).

Fig.4a and Fig.4b show the worn surfaces of 1Cr18Ni9Ti steel/ZrO_2 ceramic tribo-pair in 90% hydrogen peroxide solution. Rubbing with ZrO_2 ceramic, the flaking-off behavior, parallel to the orientation of the sample sliding is displayed on the surface of 1Cr18Ni9Ti steel (in Fig.4a). EDS analysis taken from region B of ZrO_2 ceramic (in Fig.4b) shows the signal of the Fe element, which indicates the transfer of Fe from 1Cr18Ni9Ti steel to the worn surface of ZrO_2 ceramic during the sliding. The creation of chemical bonds among Zr, O and Fe elements leads to the adhesion behavior [12]. With the relatively low shearing strength, 1Cr18Ni9Ti steel is flaked-off and transformed onto the surface of ZrO_2 ceramic by the relative movement. As for ZrO_2 ceramic, besides the adhesion layer, fatigue cracks perpendicular to the sliding direction is observed in the Fig.5a. The repeated shearing and compressive stresses, caused by the rolling of detached adhesive particles between the contact surfaces [13], results in the fatigue of ZrO_2 ceramic.

The surface tracks for 1Cr18Ni9Ti steel and Si_3N_4 ceramic after the tests are illustrated in Fig.4c and Fig.4d. The friction transfer is also observed between the contact surfaces of the 1Cr18Ni9Ti steel/Si_3N_4 ceramic. Obvious delamination behaviors are also presented on the surface of 1Cr18Ni9Ti steel in Fig.4c. The adhesion layers (region D) on the worn surface of Si_3N_4 ceramic in Fig.4d are proved to contain Fe element by the EDS. A high chemical affinity exists between iron and Si_3N_4 ceramic [14,15] which is reasonable for forming of the adhesion behavior. Apart from the adhesion layers, it is noteworthy that the signal of the O element is observed on the no-adhesion region E of Si_3N_4 ceramic. S. F. Ren *et al.* and J. Xu *el al.* reported that Si_3N_4 ceramic should be tribo-oxidated in oxidizing medium when accelerated by friction[16,17]. The oxidation and hydrolysis reactions, as represented in Eq.(1) and Eq.(2)[11], could occur on the worn surface of Si_3N_4 ceramic when friction takes place in hydrogen peroxide solution. The formed colloidal film of SiO_2 nH_2O and $Si(OH)_4$ could reduce the friction and wear loss. In 90% hydrogen peroxide solution, the wear mechanism of the 1Cr18Ni9Ti steel/Si_3N_4 ceramic rubbing pair, is affected by both adhesion behavior and the protective film formed by oxidation and hydrolysis reactions.

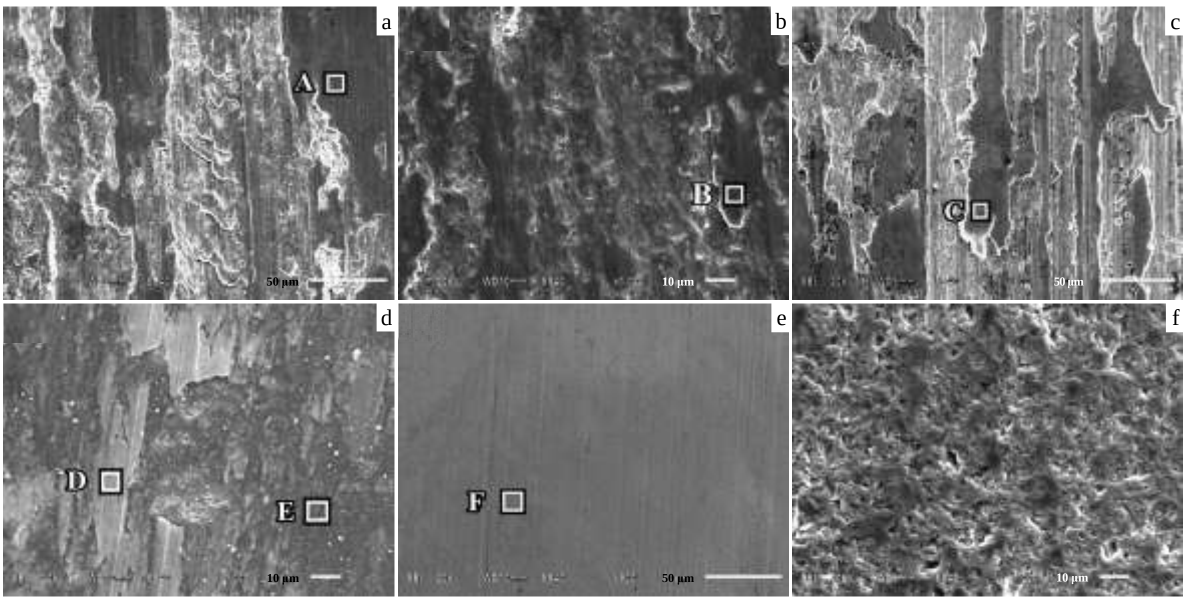

Fig.4 SEM images of the 1Cr18Ni9Ti steels and different counterparts after wear test: (a, b) 1Cr18Ni9Ti steel/ZrO_2 ceramic, (c, d) 1Cr18Ni9Ti steel/Si_3N_4 ceramic, and (e, f) 1Cr18Ni9Ti steel/SiC ceramic

$$Si_3N_4 + 6H_2O \rightarrow 3SiO_2 + 4NH_3 \quad (1)$$

$$SiO_2 + (n+2)H_2O \rightarrow SiO_2 \cdot nH_2O + Si(OH)_4 \quad (2)$$

Fig.4e and Fig.4f show the morphological characteristics of the 1Cr18Ni9Ti steel/SiC ceramic tribo-pair after test in 90% hydrogen peroxide solution. In contrast to the rough worn tracks against ZrO_2 and Si_3N_4 ceramic, the worn surface of 1Cr18Ni9Ti steel against SiC ceramic (in Fig.4e) is very smooth. There are no delamination and adhesion characteristics, only shallow abrasive grooves along the sliding direction observed on the surface of 1Cr18Ni9Ti steel. With regard to SiC ceramic, the characteristic of dispersive pits are presented on the worn surface in Fig.4f. Details reveal that cracks are formed on the surface of SiC ceramic in Fig.5b, and the compositions of different regions such as region G and region H are shown in Table 2. The initiation and the propagation of the cracks during the wear process lead to the formation of pits. By comparing the surfaces of 1Cr18Ni9Ti steel before and after wear in Fig.6, it is found that wear process of 1Cr18Ni9Ti steel/SiC ceramic actually creates a polishing action on the surface of 1Cr18Ni9Ti steel. K. Yagi *et al.* and A. Kubota *el al.* reported that OH radicals (OH·) could be obtained from the reaction of H_2O_2 molecules with the iron. The formed OH· could break the Si-C bonds during tribo-chemical reaction. When attacked by OH·, the surface of SiC was oxidized and hydrolyzed as Eq.(3) and Eq.(2)[18,19]. Rubbing with the iron-based 1Cr18Ni9Ti steel, SiC ceramic reacts seriously with the hydrogen peroxide solution. The producing of SiO_2 creates polishing action, and the existence of colloidal film could protect the surfaces of SiC ceramic and 1Cr18Ni9Ti steel contacting. Under the synergy of SiO_2, colloidal film, the 1Cr18Ni9Ti steel is polished during the wear process.

$$SiC + 4OH\cdot + O_2 \rightarrow SiO_2 + 2H_2O + CO_2 \quad (3)$$

According to the above discussions, the wear mechanisms of 1Cr18Ni9Ti steel/ZrO_2 ceramic and 1Cr18Ni9Ti steel/SiC ceramic pairs are mainly affected by the adhesion behavior, the reactions of oxidation and hydrolysis, respectively. Both the adhesion behavior and the reactions, play important roles in the wear process of 1Cr18Ni9Ti steel/Si_3N_4 ceramic (in Fig.7). Therefore, rubbing against ZrO_2 ceramic, the flaking-off behavior caused by adhesion, leads to the highest wear loss of 1Cr18Ni9Ti steel. The SiO_2 and colloidal film produced between the 1Cr18Ni9Ti steel and SiC ceramic reduce the attrition, and lead to the lowest wear loss of 1Cr18Ni9Ti steel. The wear loss of 1Cr18Ni9Ti steel against Si_3N_4 ceramic is intermediate, due to both the delamination caused by adhesion and the protective film. As for the ceramics, the subsequent propagation of the cracks leads to a high wear volume of ZrO_2

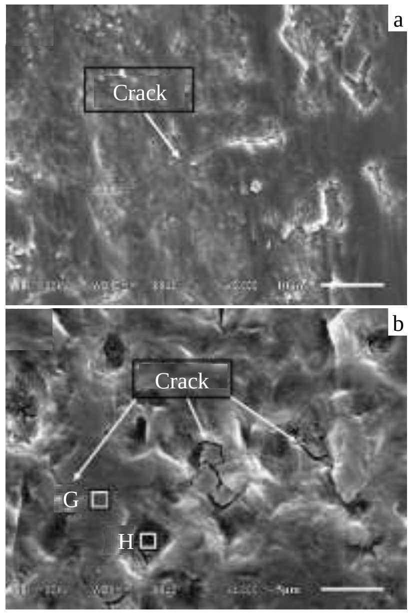

Fig.5 High magnification of the worn surface of ZrO_2 ceramic (a) and SiC ceramic (b)

Table 2 EDS analysis corresponding to the worn surfaces of the 1Cr18Ni9Ti steels and different counterparts in Fig.4 and Fig.5 (at%)

Region	Fe	Mn	Cr	Ni	Ti	Zr	Si	C	O	N
A	50.1	0.7	13.8	6.0		2.1	1.8		25.6	
B	19.8		4.6	1.8		12.6			61.2	
C	59.8	1.2	16.5	6.6	0.4		2.2		13.4	
D	32.7		7.7	3.9			27.5		28.2	
E	0.3						49.2		16.1	34.4
F	64.3	1.2	18.2	7.3			2.1	0.1	6.3	
G							81.6	10.0	8.4	
H	3.2						64.6	7.5	24.7	

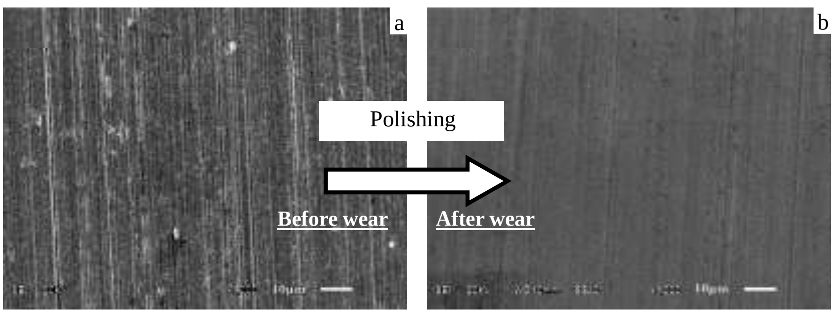

Fig.6 SEM images of the surface of 1Cr18Ni9Ti steel rubbing with SiC ceramic: (a) before wear and (b) after wear

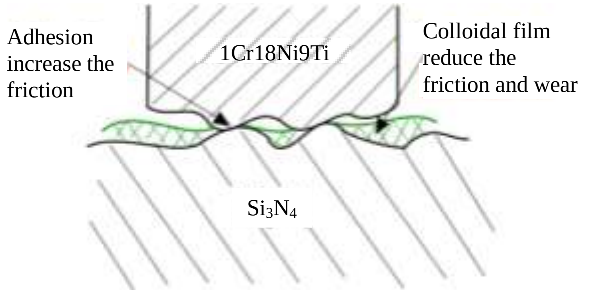

Fig.7 Wear mechanism of 1Cr18Ni9Ti steel/ Si_3N_4 pair in 90% H_2O_2 solution

ceramic. The wear volume of SiC ceramic with the produced pits is higher than that of Si_3N_4 ceramic sticking with 1Cr18Ni9Ti steel.

The junctions, which are the key of the adhesion process and sheared under the applied tangential force result in the frictional force. The formation and the rupture of the junctions control the adhesion component of friction [20]. As a result, coupled with ZrO_2 ceramic, the COF keeps a high value with a distinct fluctuation. With regard to the 1Cr18Ni9Ti steel/SiC ceramic pair, the producing of SiO_2 creates polishing action, and the existence of $SiO_2 \cdot nH_2O$ and $Si(OH)_4$ results in the formation a colloidal film to reduce the friction coefficient. Rubbing with SiC ceramic, the COF keeps an excitingly low value with very slight fluctuation. The friction behavior of the 1Cr18Ni9Ti steel/Si_3N_4 ceramic is complex. The work of X. Z. Zhao *et al.* proved that the adhesion behavior between 1Cr18Ni9Ti steel and Si_3N_4 ceramic could lead to severe friction [21]. At the beginning, the direct contact between the pair causes serious adhesion, inducing the COF with a high value and a distinct fluctuation. With the sliding process, the colloidal film is formed due to the oxidation and hydrolysis reaction. When the colloidal film is integrated enough, the COF decreases to the value similar to that against SiC ceramic. However, as the colloidal film is not compact enough and the adhesion surface is rough, the colloidal film is easily ruptured. The adhesion behavior occurs again, leading to the COF turning high. These two mechanisms appear alternately. By the reason that the oxidation and hydrolysis reaction keeps occurring in the hydrogen peroxide solution, the colloidal film becomes more and more integrated, homogeneous and compact with the increase of wear time. Thus, the duration of α regime with the high COF which is caused by the adhesion shows a trend of decrease.

3 Conclusions

1) Adhesion behavior occurs between 1Cr18Ni9Ti steel/ZrO_2 ceramic pair, leading to the high COF with a distinct fluctuation. Due to the flaking-off behavior by adhesion, 1Cr18Ni9Ti steel against ZrO_2 ceramic exhibits the highest wear loss.

2) The SiO_2 and colloidal film, produced by the oxidation and hydrolysis reactions of SiC ceramic in the hydrogen peroxide solution, protect the worn surface, resulting in the low COF of 1Cr18Ni9Ti steel/SiC ceramic pair and the lowest wear loss of 1Cr18Ni9Ti steel. The wear process against SiC ceramic creates a polishing action on the surface of 1Cr18Ni9Ti steel.

3) The wear mechanism of 1Cr18Ni9Ti steel/Si_3N_4 ceramic pair is affected by both adhesion behavior and hydrolysis reactions. The COF curve contains two alternate regimes, and the wear loss of 1Cr18Ni9Ti steel show an intermediate loss.

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过氧化氢环境下 1Cr18Ni9Ti 不锈钢与不同陶瓷配副的摩擦学行为

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摘　要：研究了 ZrO_2，Si_3N_4 和 SiC 3 种陶瓷配副对 1Cr18Ni9Ti 不锈钢在 90%的 H_2O_2 溶液中摩擦学性能的影响。结果表明，1Cr18Ni9Ti 不锈钢在该环境下的摩擦学性能受配副的影响明显。与 ZrO_2 对磨，发生了粘着行为，导致了大的摩擦系数（0.17~0.27）和最高的 1Cr18Ni9Ti 不锈钢磨损量。与 SiC 对磨，发生了氧化和水解反应，形成的胶体膜起到了润滑作用，导致了小的摩擦系数（0.035）和最低的 1Cr18Ni9Ti 不锈钢磨损量。粘着行为和水解反应均发生于 1Cr18Ni9Ti/Si_3N_4 的磨损过程中，粘着与保护膜的耦合，导致了复杂的摩擦系数。对于配副，ZrO_2 的磨损体积最大，SiC 最小，Si_3N_4 表面有粘着层，因此磨损体积介于上述 2 种陶瓷之间。

关键词：过氧化氢；摩擦学性能；配副；摩擦化学；粘着

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