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Friction and Wear Behaviors of Fe-19Cr-15Mn-0.66N Steel at High Temperature

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Abstract: The friction and wear behaviors of Fe-19Cr-15Mn-0.66N steel were investigated under applied loads of 5 N and 15 N at the wear-testing temperatures of 300 °C and 500 °C using a ball-on-disc tribometer. The wear tracks were evaluated by scanning electron microscopy (SEM) and laser scanning confocal microscopy (LSCM) to reveal the variation in morphologies. Energy-dispersive X-ray spectroscopy (EDS) and X-ray photoelectron spectroscopy (XPS) were used to determine the components of oxide layers formed on wear surfaces. The results demonstrated that the oxide layers are favorable for obtaining a low friction coefficient under all conditions. The average friction coefficient decreased with increasing load at 300 °C, while it increased with the increase in applied load at 500 °C. At 300 °C, severe abrasive wear characterized by grooves resulted in a high friction coefficient with 5 N applied, whereas the formation of a denser oxide layer consisting of Cr₂O₃, FeCr₂O₄, Fe₂O₃, etc., and the increased hardness caused by work hardening led to a decrease in friction characterized by mild adhesive wear. At 500 °C, the transformation of Fe₂O₃ to the relatively softer Fe₃O₄ and the high production of lubricating Mn₂O₃ resulted in a minimum average friction coefficient (0.34) when 5 N was applied. However, the softening caused by high temperature weakened the hardening effect, and thus the friction coefficient increased with 15 N applied at 500 °C.

Keywords: high-nitrogen austenitic stainless steel; wear; friction coefficient; high temperature

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1. Introduction

High-nitrogen austenitic stainless steel (HNSS) can be used for non-magnetic drill collars in oil exploitation, bearing steels for automobile industry, and many other applications due to its excellent properties, i.e., high strength, good ductility, and superior corrosion resistance [1–6]. However, because of harsh service conditions, such as high temperature, heavy load, and high velocity, the demand for surface hardness and wear resistance is enhanced [7,8]. Investigations on preparation methods, mechanical properties, and corrosion resistance of HNSS have been widely carried out [9–12]. However, few reports about its tribological properties, especially at high temperatures, are available. Therefore, it is of great significance to investigate the friction and wear behaviors of HNSS at high temperature.

Wear at high temperature is a serious problem in many industrial applications, such as power generation, materials processing, and high-temperature bearing [13–15]. Metallic materials can be oxidized or softened at high temperature, which plays a significant role, causing a change in overall friction behavior [16]. This is because oxides can reduce metal

wear by reducing or eliminating metal–metal contact, allowing a transition from severe to mild wear [17]. In addition, at elevated temperatures, the increasing dominance of dislocation climb, aided by increased lattice diffusion at high temperatures, results in a significant softening of metals [18,19]. Thus, the wear mechanism at high temperature is different from that at ambient temperature [20,21]. Hemant et al. [22] investigated the sliding friction and wear behaviors of austenitic stainless steel at a temperature of 473 K and 823 K, respectively, and found serious damage at 823 K due to the softening of the rubbing surfaces. Wang et al. [23] evaluated the tribological behaviors of sodium carbonate coating on stainless steel at high temperatures, and the results suggested that the lubrication capability of NCO is partially due to the reconstruction of the oxide scale. The high-temperature wear mechanism of 445 stainless steel was studied, and the Cr-rich oxide scale was able to stabilize the friction coefficient and reduce the wear rate [24]. In addition to temperature, the applied load also affects friction and wear behaviors at high temperature. Wang et al. [25] studied the effect of load on wear behavior at high temperature and found that the oxide layer at a higher load is easier to transfer onto the counterpart surface, leading to higher wear loss. Torres et al. [26] concluded that a higher applied load at high temperatures could lead to higher wear rates for the steels. Stott et al. [27] studied the effects of load on wear-protective layers during sliding at elevated temperatures, and the results showed that the layers were broken down, leading to enhanced wear damage, particularly at higher loads at 550 and 600 °C. Additionally, Razali et al. [28] used a combination of numerical and experimental methods to reveal the relationship between tribological conditions and microstructural evolution. In addition, Lee et al. [29] used similar methods that revealed that the friction coefficient suddenly changes at critical surface strain, and the lubricant film loses its function when the surface strain of the material exceeds the critical surface strain.

At present, there is a lack of research on the tribological properties of HNSS at high temperatures, and the synergistic effects of load and temperature on the wear mechanisms of HNSS need to be further studied. In this work, high-temperature friction and wear tests under the conditions of 5 N at 300 °C, 15 N at 300 °C, 5 N at 500 °C, and 15 N at 500 °C were applied on HNSS of Fe-19Cr-15Mn-0.66N. The microstructure and tribological properties of the samples were systematically characterized and analyzed.

2. Experimental Details

The material used in this study was Fe-19Cr-15Mn-0.66N HNSS. Its chemical composition is listed in Table 1. The material was cut into coupons with dimensions of 20 mm × 20 mm × 5 mm. The samples were gradually ground using 2000# SiC papers, then polished with a diamond paste of 2.5 µm, cleaned in ethanol, and finally dried using cool air.

Table 1. The chemical composition of the HNSS (wt%).

C	Si	Mn	P	S	Cr	Mo	N	Fe
0.044	0.24	15.80	0.017	0.005	18.40	2.19	0.66	bal.

The wear tests were evaluated using a ball-on-disc tribometer (HTC-2, Lanzhou Zhongke Kaihua Technology Co., Ltd., Lanzhou, China). The ball and the disc were Si₃N₄ and the HNSS, respectively. After installing the ball and disc in the holder on the tribometer, they were heated to the target temperatures of 300 and 500 °C. The sliding tests were conducted under loads of 5 N and 15 N (yielding an initial Hertz peak contact pressure of 370–630 MPa) and were conducted continuously for a duration of 1800 s in air without lubrication. The samples were taken out immediately when the sliding tests ceased to prevent further oxidation. The morphologies of the wear tracks were observed using laser scanning confocal microscopy (LSCM, LEXT OLS400, Tokyo, Japan) and scanning electron microscopy (SEM, JEOL, JSM-6480, Tokyo, Japan) under secondary electron image (SEI) mode at an accelerating voltage of 20 kV and a probe current of 50 pA. The oxide layers

formed on the wear tracks were characterized using energy-dispersive X-ray spectroscopy (EDS, X-Max 20, Oxford, UK) and X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi T, ThermoFisher Scientific, Waltham, MA, USA) using an Al $K\alpha$ (1486.6 eV) radiation source. The analysis of the spectra was performed using CasaXPS software version 2.3.15 (CasaXPS Software Ltd.). The background baseline of all the signals was subtracted using a standard Shirley background line type. Before conducting curve fitting, all the peaks were calibrated by shifting C 1s to 284.6 eV.

3. Results

3.1. Friction Coefficient

Figure 1 depicts the friction coefficient as a function of time for the HNSS under different test conditions. It is obvious that almost all the friction coefficient curves exhibited a similar tendency under all the four test conditions with the increase in time. Indeed, the curves could be divided into two stages, as can be seen from Figure 1. The curves of the friction coefficient dropped dramatically in the initial stage for the first 400 s and then rose up slowly without significant fluctuations or maintained a relatively stable trend as the time increased.

Figure 1. Friction coefficients of the HNSS under different conditions.

To observe the magnitude of the friction coefficient under different conditions more visually, the average friction coefficients of all four test conditions are shown in Figure 2. When the samples were tested at 300 °C, the average friction coefficients obtained by applying 5 N and 15 N were 0.44 and 0.38, respectively, indicating that the change in the friction coefficient is directly related to the applied load [30]. When the test temperature was 500 °C, the average friction coefficients obtained by applying 5 N and 15 N were 0.34 and 0.40, respectively. The variation in the friction coefficient shows an interesting phenomenon. When the HNSS was tested at 300 °C, its friction coefficient decreased with increasing load but showed an inverse trend when the temperature was 500 °C. This will be further explained in the Discussion section.

Figure 2. Average friction coefficients of the HNSS under different conditions.

3.2. LSCM of the Wear Tracks

Figure 3 shows the LSCM diagrams of the wear tracks. It can be seen that the surfaces of the wear tracks were seriously damaged. Grooves and oxide layers (dark zone) were observed on the surfaces of the wear tracks under all four conditions. As shown in Figure 3a, the width of the wear track was about 480 μm , and the oxide layer on the surface of the wear track was discontinuous when 5 N was applied at 300 $^{\circ}\text{C}$. When the load was changed to 15 N, the morphology of the wear track was similar to that at 5 N, except that the width of the wear track increased to 682 μm , as shown in Figure 3b. At 500 $^{\circ}\text{C}$, the worn surface was almost completely covered by the oxide layer, and the width of the wear track was about 423 μm with 5 N applied. When 15 N was applied at 500 $^{\circ}\text{C}$ (Figure 3d), the width of the wear track increased to 683 μm , and there were a large number of grooves as well as delamination of oxide on the surface.

3.3. Morphologies of the Wear Tracks

Figure 4 shows the SEM morphologies of the wear tracks for the HNSS under different conditions. It could be found that the surface was rough when 5 N was applied at 300 $^{\circ}\text{C}$. The micrograph of the wear track showed a variety of superficial damages, such as grooves and stripping areas. The material near the grooves was subjected to severe plastic deformation and plastic damage, which subsequently formed the wear debris [31], which potentially acted as a third-body abrasive source, resulting in severe abrasive wear. When the load was increased to 15 N, the morphology of the wear track was as can be seen in Figure 4b. Some small particles and stripping areas could be found. Meanwhile, work hardening caused by dislocation accumulation occurred due to the increased load, resulting in an increase in hardness [5,32–35]. Thus, the wear resistance of the sample was better than that of the sample with 5 N applied at the same temperature.

Figure 3. Morphologies of wear tracks for the HNSS under different conditions: (a) 300 °C–5 N, (b) 300 °C–15 N, (c) 500 °C–5 N, and (d) 500 °C–15 N.

Figure 4. Wear track microstructure of samples under different conditions: (a) 300 °C–5 N, (b) 300 °C–15 N, (c) 500 °C–5 N, and (d) 500 °C–15 N.

Figure 4c shows the micrograph of the wear track when 5 N was applied at 500 °C. It is shown that the worn surface was relatively smooth. However, some shallow grooves along the sliding direction could still be seen, which was mainly caused by wear debris

under dry-friction conditions. Local stripping areas could also be seen on the surface. Figure 4d shows the surface of the wear track when 15 N was applied at 500 °C; a large number of wear debris and some grooves could obviously be seen on the surface, which showed relatively serious wear behavior.

3.4. Microstructure Characteristics of the Wear Tracks

The SEM morphologies and corresponding distributions of elements on the worn surfaces of the HNSS are shown in Figure 5. It is obvious that the O element was distributed in the wear tracks of all conditions, indicating that the samples were oxidized at both 300 °C and 500 °C. It can be found from the EDS mapping that the oxide layers mainly consisted of the elements Fe, Cr, Mn, and O. Although there was no obvious change in the distribution of elements, a more serious discontinuous distribution could be observed on the oxide layer at 300 °C, which was consistent with the results of LSCM, indicating an oxidation–scratch–reoxidation mechanism in the process of friction [36]. It could also be found that oxidation was more severe with increasing temperature, and this was demonstrated by the distribution of the O element, which was distributed from being mainly present in the wear track at 300 °C to also being significantly distributed on the substrate at 500 °C.

Figure 5. SEM morphologies of worn surfaces and corresponding element mapping: (a) 300 °C–5 N, (b) 300 °C–15 N, (c) 500 °C–5 N, and (d) 500 °C–15 N.

XPS was used to investigate the components of oxide layers on the worn surfaces under different conditions. Figure 6 presents the XPS spectra of the oxide layers. The peaks of Fe 2p, Mn 2p, Cr 2p, and O 1s were found in all the survey spectra. In addition, the intensity of Cr 2p dropped obviously when the temperature increased to 500 °C, while the intensity of Mn 2p showed a slight increase.

Figure 6. XPS spectra of oxide layers at wear tracks for the HNSS under different conditions.

The high-resolution XPS spectra of Fe 2p of the wear tracks under different conditions are shown in Figure 7. The element of Fe has two common oxidation states, ferrous (Fe^{2+}) and ferric (Fe^{3+}) [37], and the two oxidation states exist under all conditions. The binding energy (BE) of Fe^{3+} (2p $_{3/2}$) in Fe_2O_3 and Fe^{2+} (2p $_{3/2}$) in Fe_3O_4 was 711.6 eV and 709.2 eV, respectively [38,39]. The peak of metallic Fe^0 (2p $_{3/2}$) was located at 706.6 eV [40]. It can be concluded from Figure 7 that Fe is mainly present in the above three forms under different conditions, with Fe_2O_3 and Fe_3O_4 in all cases. However, metallic Fe could only be detected at 300 °C, indicating more severe oxidation with increasing temperature, which is also consistent with the result in Figure 5.

Figure 7. Spectra of Fe 2p of wear tracks under different conditions. (a) 300 °C–5 N, (b) 300 °C–15 N, (c) 500 °C–5 N, and (d) 500 °C–15 N.

The high-resolution XPS spectra of Cr 2p of the wear tracks under different conditions are shown in Figure 8. The BE of Cr^{3+} (2p_{3/2}) in Cr_2O_3 was 576.4 eV [41], and a peak at ~ 2.6 eV lower than the Cr^{3+} (2p_{3/2}) peak was observed, which was attributed to the BE of metallic Cr^0 [42,43]. FeCr_2O_4 with a BE of 577.9 eV could also be detected at 300 °C [38]. However, there only existed Cr_2O_3 on the wear tracks at 500 °C, as seen in Figure 8.

Figure 8. Spectra of Cr 2p of wear tracks under different conditions. (a) 300 °C–5 N, (b) 300 °C–15 N, (c) 500 °C–5 N, and (d) 500 °C–15 N.

The high-resolution XPS spectra of Mn 2p of the wear tracks under different conditions are shown in Figure 9. The binding energy (BE) of Mn^{3+} (2p_{3/2}) in Mn_2O_3 and Mn^{2+} (2p_{3/2}) in MnO was 641.9 eV and 643.2 eV, respectively [38,39]. The peak of metallic Mn^0 (2p_{3/2}) was located at 639.6 eV [38]. In addition, the above three states of Mn were present under all four conditions.

Figure 10 shows the contents of Fe, Mn, and Cr oxides under different conditions based on the results of XPS. It could be observed that the composition of the oxides changed significantly with the change in the test conditions. When the temperature was 300 °C, the oxides of Cr were the most abundant among the oxides, while they decreased significantly when the temperature increased to 500 °C, which could also be proved by the survey spectra. In addition, the oxides content of Mn increased significantly with increasing temperature. Although the oxide content of Fe also increased at high temperatures, the increase was not as great as that of Mn.

Figure 9. Mn 2p spectra of wear tracks under different conditions. (a) 300 °C-5 N, (b) 300 °C-15 N, (c) 500 ° C-5 N, and (d) 500 ° C-15 N.

Figure 10. Contents of oxides under different conditions.

4. Discussion

The friction coefficient curves show some significant variations in trends and values under different conditions. Detailed studies of the wear tracks have shown that these changes are associated with the oxide layers formed on the wear surfaces. Oxidation wear is a common phenomenon in a high-temperature environment, characterized by the presence of an oxide layer on the sliding surface [44,45]. The surfaces of the samples are easily oxidized under high temperature due to the high affinity of Cr, Mn, and Fe to

O [23,24]. These oxides usually became wear particles due to the normal stress and shear force from the friction couple during the friction test, and the wear particles, which act as a third body, often plough the wear tracks as the sliding develops, resulting in a fluctuation of the friction coefficient and a relatively high value [16]. After the generation of debris particles, some are lost from between the surfaces, while most are retained and they are involved in the development of a compact oxide layer [23]. This layer can provide wear protection for long periods due to its high smooth and hardness, resulting in a decrease in the friction coefficient. The development of this layer from the agglomeration and compaction of the wear debris particles is described elsewhere [46].

As is seen, oxide layers were formed on the surfaces of all wear tracks (Figures 3 and 4), but the stability of the oxide layers was different under different conditions. In general, this compact oxide layer can form at a temperature higher than 150 °C [16]. In addition, the rate of establishment of this layer usually increases with the increase in temperature [17], which can explain the phenomenon that the friction coefficients at 500 °C were lower than those at 300 °C for the first 400 s. It can be seen from the results of the friction coefficient that there was no significant fluctuation after 400 s (Figure 1), indicating that the oxides formed on the wear track surface play a crucial role in the wear process. However, there existed a little difference in the friction coefficient trend after 400 s. Most of them increased slowly as the time increased, except for the curve of 300 °C–15 N, which maintained a relative constant at 0.38. After the establishment of the compact oxide layer, there were two competitive processes, breakdown and reconsolidation of this layer, indicating that the layer established at 300 °C with 15 N applied may be the most stable one.

When a load of 5 N was applied at 300 °C, an oxide layer consisting of Fe_2O_3 , Fe_3O_4 , Cr_2O_3 , FeCr_2O_4 , MnO , and Mn_2O_3 was formed on the wear track, which can be concluded from the results of EDS and XPS. It can be revealed that the content of Cr_2O_3 is higher than XPS (Figures 6 and 10), and it is usually used as the third body to grind the surface of the wear tracks due to its high hardness [24]. Thus, severe abrasive wear occurred at this time. In addition, part of the Cr_2O_3 can react easily with Fe to form FeCr_2O_4 at 300 °C [47], which has high hardness and can be easily removed from the surface during the process of friction, thus greatly reducing the protective ability of the oxide layer [37,48]. With the plowing of Cr_2O_3 , the transformation of Cr_2O_3 to FeCr_2O_4 and the long-term shear action of the friction couple, the layer was destroyed, which resulted in the exposure of the matrix and the formation of a large amount of wear debris on the surface of the wear track (Figure 4a) [37]. The HNSS matrix was ploughed by hard debris under the extrusion of the friction couple, forming some grooves parallel to the sliding direction on the surface. Thus, the friction coefficient increased continuously and the average friction coefficient (0.44) was the highest under this condition with the wear mechanism of oxidation wear and severe abrasive wear [49].

The addition of the N element could further reduce the stacking fault energy of austenitic stainless steel, which increased the ability of work hardening. When 15 N was applied at 300 °C, the increased load facilitated plastic deformation of the HNSS, which led to an increase in the dislocation density and an increment in the dislocation, resulting in the effect of work hardening and the improvement of the hardness of the matrix [5]. A material with higher hardness usually shows a lower friction coefficient to that of a material with lower hardness [50–53]. The higher load also makes it easier to crush oxide particles, such as Cr_2O_3 , which are usually squeezed into previously formed grooves, which in turn makes the formed compact layer denser and also enhances the load-bearing capacity [17,54]. During the period of 0–400 s of the test, the curve of the friction coefficient was almost coincident with that at 300 °C–5 N, but it became much more stable after 400 s due to the increased hardness and compact oxide layer (Figure 1). At this time, a significant reduction in the content of Cr_2O_3 and FeCr_2O_4 also occurred, and the grinding effect on the surface was reduced. However, a partial oxide layer may peel off and adhere to the surface of the friction couple during friction due to shear action and the accumulation of plastic deformation in the contact area. The peeling oxide layer was then gradually

removed from the surface, and the exposed metal matrix under the stripping area was oxidized to form a new oxide layer (Figure 5b). The exfoliated oxidation products hindered direct contact between the friction couple and the matrix. The effects of increased hardness, the formation of a denser oxide layer, and the exfoliated oxidation products under this condition make the HNSS have better wear resistance than that of 300 °C–5 N, showing the wear mechanism of oxidation wear and mild adhesive wear and forming a stable friction state (Figure 1).

When a load of 5 N was applied at 500 °C, only a few oxide particles, shallow grooves, and sporadic stripping areas could be observed, as seen in Figure 4c. It could also be found that the content of Cr oxides was significantly reduced, and Cr₂O₃ had the effect of inhibiting the diffusion of O to the inside. Thus, a large amount of Fe and Mn oxides were formed when Cr₂O₃ was reduced. Among them, Mn has a stronger affinity with O compared to Fe, so the oxides of Mn, i.e., MnO and Mn₂O₃, are more abundant [23,24]. The latter, in particular, is generated in large quantities and is beneficial for lubrication during friction [55]. In addition, at 500 °C, Fe₂O₃ is easily transformed into Fe₃O₄, which has lower hardness than Fe₂O₃, and also plays a certain protective effect [24,56]. Additionally, the oxide layer is softened due to the high temperature, which can reduce friction to some extent [38,57,58]. Due to the accumulation of plastic deformation, the partial layer peeled off and adhered to the friction couple. Therefore, the friction process mainly occurred between the oxides and the friction couple, which could play a better role in tribological properties [59–61], and the average friction coefficient (0.34) was the smallest under this condition in all cases (Figure 2) [62,63].

When a load of 15 N was applied at 500 °C, the composition of the oxide layer did not change much compared to that at 500 °C–5 N, while the increased temperature softened the matrix, resulting in the weakening of work hardening [64]. In addition, the wear of the oxide layer was aggravated and the layer was destroyed due to the increased load, leading to large amounts of wear debris and some grooves (Figures 3d and 4d), showing typical abrasive wear. The increase in Mn content on the wear track of EDS indicated that the compacted oxide layer on the surface was slightly damaged, which led to an increase in the friction coefficient (0.40) [26], so the wear resistance weakened. However, the oxide layer still played a certain role in protection, so the wear resistance at this time was better than that at 300 °C with 5 N applied. The wear mechanism of the HNSS under this condition was mainly oxidation wear and severe abrasive wear.

5. Summary

The friction and wear behaviors of the Fe-19Cr-15Mn-0.66N HNSS at high temperature were tested under different conditions with different loads and temperatures, and the conclusions can be drawn as follows:

- (1). The friction coefficient of HNSS decreased with time up to 400 s but rose up slowly or maintained a relatively stable trend as the time increased. The average friction coefficient decreased with increasing load at 300 °C, while it showed a reverse trend at 500 °C, which depended on the wear mechanism that reflects the competitive processes, including breakdown and reconsolidation of the oxide layer formed on wear tracks.
- (2). At 300 °C, the friction coefficient curve of HNSS with 15 N applied showed a stable friction state after 400 s due to the formation of a denser oxide layer consisting of Cr₂O₃, Fe₂O₃, and Mn₂O₃ and increased hardness caused by work hardening, showing the wear mechanisms of oxidation wear and adhesive wear.
- (3). Under the conditions of 500 °C–5 N, softening at high temperature and the production of large amounts of Mn₂O₃, which has a lubricating effect, minimized the friction of the sample, showing the lowest friction coefficient with a value of 0.34. In addition, the increased temperature also weakened the work hardening, resulting in severe abrasive wear at 500 °C–15 N.

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