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Adhesion, friction and wear analysis of a chromium oxide scale on a ferritic stainless steel

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Abstract

The aim of this study is to explore the potential engineering application of a thermally grown chromium oxide scale on a ferritic stainless steel. The friction and wear behaviour of the oxide scale were investigated by scratch and pin-on-disk tests. The failure mechanisms of the oxide scale subjected to scratch tests under both progressive and constant loading were investigated. It shows that the oxide scale adheres well to the steel substrate and is in a ductile failure. The oxide scale leads to an increase in the coefficient of friction (COF) under different constant loads when compared to what occurs when there is no oxide scale in the scratch test. The pin-on-disk tribological tests show that the chromium oxide scale on the ferritic stainless steel causes abrasive wear of the high-speed steel (HSS) pin, but it enhances the wear resistance of the ferritic stainless steel disk. The COF is slightly lower when there is oxide scale on the disk which is different from that by the scratch test. The ferritic steel substrate fine wear particles generated during the dry sliding and the formation of new compound layers on the surface can stabilise the COF. The wear mechanism is proposed and the tribological properties of the thermally grown chromium oxide by the two techniques are discussed.

Keywords: Ferritic stainless steel; Chromium oxide scale; Powder-like debris; Pin-on-disk

1. INTRODUCTION

When iron or Fe-rich alloy is heated in highly oxidising gases above 572 °C, a multi-layered oxide scale is formed with wüstite (FeO) at the metal-oxide interface, an intermediate layer of magnetite (Fe₃O₄), and hematite (Fe₂O₃) at the oxide-gas interface [1-

4]. Usually, the iron oxides exhibit higher hardness than the steel substrate at room temperature, but their hardnesses decrease when the temperature increases.[5]. It is known that FeO and Fe₃O₄ can behave like lubricants at high temperature, whereas α -Fe₂O₃ has a higher hardness that promotes abrasive wear [6, 7]. The oxide scale formed on different steels may have different thicknesses and compositions, showing different tribological behaviours that depend on the temperature [8], atmosphere, and chemical composition of the alloys [9]. On stainless steels, the Cr₂O₃ scale protects them from being oxidising quickly at high temperature. The thickness of the Cr₂O₃ scale varied from nano to micrometres before the occurrence of breakaway oxidation, e.g., oxide thicknesses on AISI

441 and 444 stainless steel was between 0.6 and 1.3 μm at 800-1000 °C during 100 or 200 h [10]. Over time breakaway oxidation occurs, and iron-rich oxides or spinel formed quickly [11-13], causing an abrupt increase in the oxide growth rate/thickness. The oxide scale on the steels may have beneficial or detrimental effects on the tribological behaviour according to its characterisation on the steels. Oxide scale as a solid layer on the metal may decrease metal-to-metal contact, thus protecting the metallic parts against wear [14, 15]. If the oxide scale breaks or delaminates from the steel substrate, the wear becomes severe, and this would be associated with its adhesion strength. Oxide debris may act as free third bodies abrading the antagonistic surfaces if it is hard and brittle [16]. In a certain situation,

when wear particles and oxides form mechanically mixed harder layers or tribolayers at the contact surface, wear can be reduced [17].

The adhesion of the oxide scale was determined qualitatively by way of scratching on the surface oxide [18]. By a constant increase of loads, it is able to measure the scale adhesion accurately. The adhesion strength depends on many factors: the specific oxide growth mechanism, the stresses in the oxide metal system, the ability of the system to relieve such stresses, and the fracture resistance of the oxide and the metal and the metal/scale interface [10, 19, 20]. Wear behaviour in the scratch test is usually the abrasive/ploughing wear due to a coated material is always softer than the diamond pin. Coating cracking and spalling can be determined at different loads. Cracks exhibit different characterisations such as angular, parallel and transverse semi-circular cracks. Severe coating failure such as chipping, spalling and whole layer breakthrough may occur [21]. Alloy elements, like Si, Mo [22], or rare earth elements are known to improve the adhesion of Cr_2O_3 scale [11, 23].

The thermally grown Cr-rich oxide scale on the ferritic stainless steel 445 played a major role in friction and wear at high temperature [24]. Cr_2O_3 , whatever it is a solid or particles, showed ductility, prevented adhesive wear and stabilised coefficient of friction at high- temperature range 850-950 °C. The adhesion of the oxide scale had an effect on its tribological performance. It is known that the chromium oxide was used as coating though techniques such as chemical vapour deposition and radiofrequency reactive magnetron sputtering[25, 26]. The chromium oxide is hard, exhibiting a low coefficient of friction, and has properties to reduce wear and corrosion [25, 26]. The purpose of this study is to form

the Cr_2O_3 scale on this stainless steel by an oxidation process, and to investigate its adhesion strength by macro-scratch tests and its tribological behaviour by pin-on-disk dry sliding tests. It could give us an understanding of the possible potential of this thermally grown chromium oxide scale in engineering applications such as its effect in reducing wear or coefficient of friction.

2. EXPERIMENTAL METHODS

2.1 Oxidation process

The chemical composition of the ferritic stainless steel 445 is listed in Table 1. The steel 445 samples were machined to the disks with a diameter of $\varnothing 40$ mm and a thickness of 5 mm. All disks were placed in an electric furnace at 1100 °C for 120 min then slowly cooling them down to room temperature. In this way, the thickness of the oxide scale formed on all the oxidised disks was kept the same. In an iron-based alloy containing chromium as a solute, an outer layer of chromium oxide Cr_2O_3 will preferentially form during the early stages of oxidation but the poorly protective Fe-rich oxides will form which depends on the oxidation temperature and time, the oxygen partial pressure, the water vapour content of the atmosphere and the Cr content of the alloy [11, 27-29]. A large amount of Cr_2O_3 evaporation occurs in a mixed atmosphere of O_2 and H_2O and induce the breakaway oxidation, therefore Cr_2O_3 scale will be thinned and the surface will become uneven due to the formation of Fe-rich oxides [30, 31]. The heating process adopted in the study is according to our previous oxidation research of the steel. For example, the Fe-rich oxides were formed on the steel 445 at 1120 °C in a humid air. The heating process in the study ensures that the Cr_2O_3 scale is formed to a certain thickness and the surface is even.

The characterization of the oxide scale by oxidation can be found in our earlier reported work [32]. On the oxidised disk surface, some coarse tetrahedral $(\text{Cr, Mn, Fe})_3\text{O}_4$ spinel crystallites are embedded in the fine chromium oxide scale matrix. Discontinuous SiO_2 particles exhibits at the steel/oxide interface on the 445 steel. Chromia scale decreases oxygen activity at the scale/metal interface to the dissociation partial pressure of Cr_2O_3 according to the thermodynamic aspect. Then, only elements forming more stable oxide such as Si can be oxidised selectively underneath the Cr_2O_3 scale. The amount of SiO_2 is little compared to the big volume of Cr_2O_3 in the oxide scale. SiO_2 particles are also known as hard and abrasive. The amount of Fe in the spinel is small. The main oxide scale composition is Cr_2O_3 . The oxide scale thickness is $6.1 \pm 0.23 \mu\text{m}$. This thermally grown Cr_2O_3 oxide scale is thicker than that reported elsewhere [10]. The hardness of the Cr_2O_3 oxide scale was measured at the cross-section by a nanoindenter, showing a value of $14 \pm$

1.5 GPa. A Topographic feature and surface roughness of the bare and the oxidised stainless steel surface were examined using an atomic force microscope (AFM). Fig.1 shows oxidised disks from the furnace and the comparison of topography and surface roughness of the clean and the oxidised surface. At the area of $20 \times 20 \mu\text{m}^2$, the oxidised surface has higher Ra and RMS (Rq), indicating that the surface becomes rougher after oxidation. The disk hardness has not changed after the oxidation process.

Table 1 Chemical composition (wt. %) of the ferritic stainless steel 445, disk material.

C	Si	Mn	P	Cr	Cu	Mo	Ti	Nb	Fe
≤ 0.01	0.30	0.15	0.03	21.50	0.10	0.60	≤ 0.20	0.12	Bal

Fig. 1.(a) oxidised 445 steel disk from the furnace, (b) The cross-section of the oxide scale formed on the 445 steel from [32] (c) AFM image and surface roughness of clean bare surface and (d) AFM image and surface roughness of oxidised 445 surface

2.2 Scratch tests

Scratch tests were utilised to study the friction behaviour and damage to the oxidised surfaces under sliding contact. A scratch tester from CSM Instruments SA equipped with a Rockwell C diamond stylus was used. The pin has a cone apex angle 120° and tip radius $200 \mu\text{m}$. Two types of tests were completed: progressive and constant load. The progressive load was used to obtain the critical load at which failure occurred along the scratch track by applying a load from 0 up to 20, 50, 100 and 150 N for a length of 5 mm and the lateral displacement speed was 6 mm/min. In order to verify the results, the test was carried out

three times. The instrument was equipped with an acoustic emission monitoring system to detect crack formation and a device to measure the horizontal frictional force F_t in the scratching direction from which the COF can be obtained ($F_t = \mu F_n$). The failure of the oxide scale was determined by *post facto* microscopic examination of the scratch tracks. The first acoustic emission peak observed and the variation of the frictional force provided complementary information for critical load measurements [33]. Constant loads of 5, 10, 15, 20, 50 N were performed specifically to analyse the friction behaviour and the sliding mechanisms of the oxidised surfaces and the test repeated three times to verify the results.

2.3 Pin-on-disk tribo tests

Tribological tests were carried out under a pin-on-disk configuration on a multi-function tribometer from Rtec-instruments, USA. The chemical composition of the high-speed steel is listed in Table 2. The hardness of the high-speed steel (pin) is 835 ± 35 HV and the hardness of the ferritic stainless steel 445 (disk) is 172 ± 9 HV at room temperature. The pin was manufactured into a 6 mm radius with a ball surface and a length of 20 mm. The pin tips were machined by a lathe, so that the surface was not very smoothing, but every pin has the same surface topographical feature. Fig. 2 shows the dimensions of the pins and the top view of the pin tip. The spiral marks were caused by lathe machining. The test was carried out at a linear speed of 50 mm/s for a total linear sliding distance of 90 m. Each test was repeated three times to verify the results.

Table 2 Chemical composition (wt. %) of the high-speed steel, pin material.

C	W	Mo	Cr	V	Sr	Mn	S	P	Impurities	Fe
1.6	6.0	5.0	4.0	4.5	1.2	0.3	0.03	0.03	≤ 0.1	Bal

Fig. 2 The dimensions of the high-speed steel pin and the top view of the pin tip

X-ray diffractometer (XRD) was used for phase detection of the wear debris. A KEYENCE 3D laser-scanning microscope and a JEOL JSM-6490 type scanning electron microscope (SEM) equipped with an energy dispersion spectroscopy meter (EDS) were used to characterise the structure, morphology, and composition of the worn surface.

3. RESULTS AND DISCUSSION

3.1 Scratch test

The diamond stylus was drawn across the oxidised surface as the applied load increased linearly from 0 to 20, 50, 100 and 150 N while acoustic emission (AE) signals and frictional forces (FF) were recorded. Fig. 3a shows an example of the coefficients of friction (COF) on the oxidised and the clean bare surface of the steel 445 under different progressive loads. Fig. 3b shows an example of the acoustic emission output (bare and oxidised surfaces) and the area of the oxide scale cracks during the scratch test at a progressive load of 150 N. COF was higher when the diamond stylus was in contact with the oxidised surface. The higher the progressive load the higher variation of COF is between the two different surfaces. Actually, AE signals were not stable and not consistent for each test. This is caused by the uneven oxidised surface of the 445 steel or the non-fragile coating failure [34]. The bare 445 surface without oxide scale also caused higher

variation of AE signals. Acoustic emissions are the stress waves generated by the sudden internal stress redistribution materials or structures when changes in their internal structure are produced, e.g, crack initiation, deformation, interfacial failure, etc. This indicates that the steel substrate cracks or fractures from the bulk. It can be seen from Fig. 3b that the friction force on the oxidised surface was higher but more stably increased than that on the bare steel surface as the applied load increased. The images on the right of Fig.3 show the horizontal and the vertical profiles at the end of the scratch track at a progressive load of 50 N. The wear profile of the steel substrate was irregular and not consistent compared to that of the oxidised steel surface. The evolution of the friction coefficient with scratch length is much more tortuous for the substrate samples are because its surface morphology has changed during the scratch. The critical load was mainly determined by the change in friction and the failure of the oxide scale which was observed by a microscope and SEM. The crack initiated at a critical normal load (L_c) of 39 N. More steel substrate was exposed as the normal load increased, and some oxides were incorporated into the base steel substrate, but more brittle. The mean critical load of the oxide scale on the 445 surface was 36 ± 13 N. It is a significant variation. Because the oxide layer is formed by the thermal oxidation, the stress in the oxide scale is more complicated than the coatings. Oxide scales are usually subject to mechanical stress [35]. It is developed during oxidation or during temperature change such as cooling. The growth stresses in the oxide can be also caused by the changes in the oxide composition [36]. The Cr_2O_3 oxide on Fe-Cr alloys is compressed because the volume differences between the oxide and the steel, and its Pilling-Bedworth ratio (PBR) is greater than unity [37]. The test results may be affected by the stress in the oxide scale.

Fig. 3 (a) coefficients of friction with applied progressive loads from 20 to 150 N on oxidised and clean bare surface of the steel 445. The thicker line indicates COF curves of the oxidised surface. (b) Friction force (FF) and acoustic emission (AE) signals of two surfaces under progressive load with the scratch track images of the oxidised stainless steel surface. Applied load: 0-150 N, the lateral displacement speed: 6 mm/min, and scratch distance: 5 mm. The images on the right show the horizontal and the vertical profiles at the end of the scratch track at 50 N under the progressive load.

The failure mode in the scratch testing is complex. The crack pattern in a scratch test has been investigated and classified by Bull et al. [38]. It is classified as a ductile failure mode for a hard coating on a soft ductile substrate [21]. It was observed that the soft substrate material was piled up ahead of the stylus when there was no oxide scale on the steel 445.

The failure mode of the chromia scale occurs ahead of the moving indenter as shown in Fig. 3b. According to the AE signals, both bulking and spallation failure would occur. When the diamond stylus past over the chormia failure, the damage associated with it was constrained within the scratch track. No small spalled chips at the track edges or in the track were observed. The spalled or bulked oxide scale would be impressed into the wear track. In some cases, AE did show some abrupt high peaks, however, this may be caused by the indenter tip in contact with coarse spinel structures along the path. The high intensity of AE signal on the bare steel surface without the oxide scale occurred at the beginning of the scratch, as shown in Fig. 3b, indicating that the damage to the steel substrate took place quickly without the protection of the chromia scale.

Based on the results of the progressive load test, another scratch testing method was performed in order to investigate the frictional behaviour and cracking initiation and development under constant loading conditions. Fig. 4a shows the average coefficients of friction for the 445 stainless steel with or without oxide scale using constant loading. Fig. 4b shows the scratch track depths. The depth measurement has been used for material hardness evaluation [39]. As the scratch depth increased, the hardness was more inclined to the substrate's hardness. Fig. 4a indicates that there was a substantial increase in the average COF when there was an oxide scale on this steel. The layer and interfacial adhesion

have restrained the plastic flow and pile-up behaviours of the substrate layer [40]. The COF increased significantly at a load of 50 N as this load was close to the critical load. The COF is 3.2 times than that at a load of 20 N. Fig. 5 shows the 3-D profiles with scratch tracks on the surface of the oxidised 445 steel under different applied loads. From 5 to 20 N, the black oxide scale was still attached to the steel substrate. At a load of 50 N, as seen in Fig. 5d, on the bottom of the scratch track, transverse cracks were observed inside the scratch path. The colours of the oxide and the steel substrate are different. No small spalled black chips, which indicated oxide, were observed at the track edges or in the track. The spalled or bulked oxide scale would be impressed into the wear track. According to the measurements of the morphologies and the profiles in the scratch tracks, the oxide scale was to be pressed and appeared attached to the steel substrate. The steel substrate is ductile and undergoes plastic deformation when the scratch tip ploughs the material, forming a periodic concave damage feature pointing toward the scratch direction [41]. The cracks formed behind the indenter, and this occurred to a softer substrate [42, 43].

The oxide scales with Fe_2O_3 , Fe_3O_4 and FeO formed on a low carbon steel between 1100 and 1250 °C spalled easily [4]. The critical adhesion load of the oxide scale did not reach 20 N. However the critical load is strongly affected by many parameters related to testing conditions and the coating/substrate system [44]. From the scratching tests in our study, the failure of this thermally grown Cr-rich oxide scale is not easily identified by the progressive load, but much clearly observed by the method of using constant load.

Fig. 4 (a) Variation of coefficient of friction and (b) scratch track depth with applied constant loads by scratch test

Fig. 5 3-D Profiles with scratch tracks on the surface of oxidised ferritic stainless steel 445 under different applied constant loads (a) 5 N, (b) 10 N, (c) 20 N, and (d) 50 N. The black colour in the tracks indicates the Cr-rich oxide scale

3.2 Wear and friction

Two loads, 5 and 50 N, were selected to investigate the tribological behaviour of oxide scale on the ferritic stainless steel 445. High-speed steel was chosen as a pin material because it is used as a roll material in the steel industry and it displays high hardness and abrasion resistance. Fig. 6 shows the macro-worn surfaces of HSS pins under loads of 5 and 50 N after pin-on-disk tribotests. When there was oxide scale on the steel disk, the wear morphologies of the pin tips were different from that on the clean steel disk. It can be seen that some materials covered the HSS pin tips from the 3-D profiles in Fig. 6a and c. The area of the material covered more on the surface of the HSS pin at a load of 50 N. Figs. 6b and d show clearly the removal of the HSS pin material when the disks have oxide scale. The wear scars on the pins were circular so that the top portion of the sphere which was abraded was the wear volumes. Wear volume loss is $(2.42 \pm 0.56) \times 10^{-2}$ and $(6.63 \pm 1.07) \times 10^{-2}$ mm³ under loads of 5 and 50 N, respectively. The wear volume loss of the HSS pin at 50 N is more than that at 5 N but the surface of the worn HSS pin is metallic shiny at 5 N, while it is dull at 50 N.

Fig. 6 Worn surfaces of pins under loads of 5 and 50 N after dry sliding pin-on-disk tests (a) and (c) HSS pin on clean disk, and (b) and (d) HSS pin on oxidised disk (the arrows indicate sliding direction)

Fig. 7a shows the variation of COF as a function of the sliding time under loads of 5 and 50 N. The COF is higher in the early stage but then stabilises to a lower COF by the end of the test, and this occurs to the different surface conditions, with or without oxide scale. In the initial stage of sliding, when HSS pin contacts on the clean FSS disk, it is the metal-to-metal contact so that severe adhesive wear occurs. COF curves fluctuate significantly when the clean disks are used. Usually, this phenomenon is called the “running-in” process. Running-in involves a tribological transition in wear from high rates to low rates with sliding time [45]. If surfaces are ‘run-in’ under load, the projecting irregularities are gradually removed and the actual area of contact is increased. It depends on the tribosystem, including changes in surface composition, microstructure, and third-body distribution. Later, a mild wear occurs over time because the metal surface becomes oxidised or hardened [14]. This phenomenon does not occur at the initial stage when the FSS disk has an oxide scale. At the stabilised stage, the COF is slightly lower when there have oxide scales on the disks at the same load. The COF increases when the load increases. Fig. 7b shows the average COF values at different loads. COF decreases marginally when there is a Cr_2O_3 scale on the FSS disk at two loads. However, considering the variance of the data, the average friction coefficient can be thought to be not affected by the presence of the oxide layer. This also indicates the tribosystems that show the same COF can also have different wear behaviour and wear rates because the energy is partitioned differently into heat, fracture, deformation, and the creation of wear particles [46, 47]. The COF values by pin-on-disk tribological tests showed different results compared to the scratch tests. It has to be noted, in scratch tests, the counterpart tip material is diamond, which is harder than the Cr-rich oxide scale. The scratch speed is slow and the

distance is short and the tip on the track is not overlapped. Although COF can be obtained by the scratch test, it shows the friction force between the diamond and steel or the Cr-rich oxide scale. Only the abrasive/ploughing wear was shown on the 445 steel with or without oxide scale. Wear is influenced significantly by several parameters such as load, velocity, counterparts, environment and others. Scratch tests in this study can give us some aspects on the material properties in sliding and on the initial contact of the pin-on-disk test. For example, the ferritic stainless steel substrate was easily cracked or deformed under the high loads (Fig.5d).

Fig. 7 (a) Coefficient of friction versus sliding time for HSS pins sliding against clean and oxidised ferritic stainless steel disks under 5 and 50 N and (b) COF at different loads

Black wear debris powder-like particles were generated during dry sliding on both disks. The particles were collected for the XRD analysis. A conventional diffraction meter operating with Cu K_{α} radiation was used for X-ray analysis. Fig. 8 shows that the wear debris generated on the clean disk is mainly stainless steel substrate powder, while on the oxidised disk they are mainly stainless steel substrate powder with a small amount of Cr_2O_3 . The particle size of the wear debris is very small and some are nanometres. The

powder can be attracted to a magnet. The black powder-like particles were visible during testing after 5 min, which had an effect in stabilising the COF for the rest of the test. This indicates that not only oxides but also the Fe-Cr substrate powder can stabilise the COF in the dry sliding. Hiratsuka et al. [48] analysis the role of wear particles in severe-mild wear transition. Morphologies of wear particles changed and resulted in smaller particle sizes when the wear mode changed from severe to mild, and some wear particles are oxidised.

Fig. 8 The XRD analysis of wear debris on clean and oxidised disk after dry sliding pin-on-disk tests under 50 N.

Fig. 9 shows the SEM micrographs of the worn surface of the HSS pins on both the clean and the oxidised disks at 5 N. The HSS pin surfaces have been cleaned in an ultrasonic bath for 5 min. Fig. 9a shows that the pin surface is covered with the ferritic stainless steel transferred from the clean disk. Substrate powder was compacted and sintered on the pin surface. It shows that the HSS pins had adhesive wear because the Fe-Cr powder sintered on the pin surface. Fig. 9b shows the scratch marks and carbides in HSS, indicating the abrasive wear of the pin and the grinding effect of the oxide scale on the stainless steel 445.

Fig. 9 SEM micrographs of worn surface of HSS pin under 5 N after dry sliding pin-on-disk tests (a) on clean disk and (b) on oxidised disk

Fig. 10 shows the SEM micrographs of the worn surface of the HSS pins on the clean and the oxidised disks at 50 N. Fig. 10a shows that the HSS pin surface is covered with the ferritic stainless steel transferred from the clean disk, which is similar to that at 5 N. EDS spectra 1 and 2 show an oxidised surface. Fig. 10b shows the scratch marks in the insert image, indicating the abrasive wear of the HSS pin. EDS spectra 2 and 3 show that the powder-like wear particles of Cr-rich stainless steel substrate are sintered on the surface, and also oxidised. Spectrum 4 shows the composition of the HSS substrate because the intensity of Cr is weak and it indicates a composition of W. The worn surface was slightly oxidised, showing oxidative wear.

Fig. 10 SEM and EDS analyses of worn surfaces of the HSS pins under 50 N after dry sliding pin-on-disk tests (a) on the clean disk and (b) on the oxidised disk

Fig. 11 shows the SEM micrographs of the worn surface in the wear tracks of the clean and the oxidised disks under loads of 5 and 50 N. The high pressure compressed air was used to clean the disk tracks. The worn surfaces of the wear tracks had a similar appearance. The plough signs and the delamination of the ferritic stainless steel were dominant in all wear tracks. Fig. 11b shows the ferritic stainless steel squeezed from the cracks of the oxide scale. According to the sliding counterpart HSS pin surface (Fig. 9b), there was no sign of transferred ferritic stainless steel substrate, indicating less adhesive wear at 5 N when there has oxide scale on the disk. The same phenomenon took place on the HSS pin sliding

against the oxidised disk at 50 N (Fig. 10b), where only small amounts of stainless steel powder- like wear debris was present. This means that the oxide scale on the ferritic stainless steel can reduce adhesive wear between the ferritic stainless steel 445 and the HSS. Fig. 11c and d show that there is more powder-like wear debris, mainly the steel substrate powders sintering in the wear track. This powder-like wear particle was generated more at the higher load. The powder-like wear particles started agglomerating in certain locations, filling into the grooves of the pins and disks caused by abrasive wear, and became load-bearing and prevented metal-metal contact [49].

Fig. 11 SEM micrographs of worn surfaces of disks after dry sliding pin-on-disk tests (a) clean disk and 5 N, (b) oxidised disk and 5 N, (c) clean disk and 50 N, and (d) oxidised disk and 50 N (the arrows indicate sliding direction)

Fig. 12 shows two-dimensional surface profiles of the wear track of the disks. The disk having oxide scale showed the shallowest wear track at 5 N. The wear of the disk was less

when there was oxide scale on the disks at the different loads. The thermally grown chromia scale on the ferritic stainless steel was useful for improving its wear resistance in dry sliding. A hard and compact oxide coating on the steel substrate can enhance its abrasive wear resistance [15, 50]. Wear debris particles were formed in our pin-on-disk dry sliding tests. It was found that wear debris particles were dominantly involved in the wear processes of the transitions in wear and contact resistance with sliding time [51]. The wear debris particles took part in the formation of high-resistance load-bearing layers, and developed by compaction of the wear debris particles on the rubbing surfaces. Adhesion between the wear debris particles is an important factor in determining whether the layers are maintained or break down [52]. Increased sintering and adhesion between the debris particles can enhance the oxidation of these particles. If the wear debris particles are not sintered or compacted it may lead to abrasive wear. A complex mechanism involving wear debris from both the disks and the counter bodies that are fractured, oxidised, attached to other particles and finally, contribute to the formation of new compound layers on the surface. Fig.13 shows the wear mechanisms in the two cases.

Fig. 12 Two-dimensional profiles of wear tracks of the 445 disks after dry sliding pin-on-disk tests

Fig. 13 schematic illustration of the wear mechanism (a) HSS pin on bare 445 disk, mainly adhesive wear. Wear debris particles are sintered, compacted and oxidised to the formation of new compound layers on the surface, (b) HSS pin on the oxidised 445 disk, mainly abrasive wear. Wear debris particles include more Cr_2O_3 particles may influence the sintering and attachment.

At room temperature, steel substrate powder-like particles were generated and became more overtime during the pin-on-disk test (Fig.7). These powder-like particles had effects in stabilising the COF quickly. It is noted that tribological performance of the Cr_2O_3 scale on the 445 stainless steel was attributed to its adhesion to the steel substrate and its mechanical oxide scale stability [53]. The oxide scale formed on the steel 445 is made of the irregular Mn-Cr spinel on top of the Cr_2O_3 scale [24]. High concentration of Cr (21.5 wt.%) and alloying elements in the steel play big roles, e.g., Mn can lower the activity of Cr_2O_3 by the formation of Mn-Cr spinel on the top, which can protect the Cr_2O_3 scale from evaporation in an atmosphere containing oxygen and/or H_2O [54]. The rough surface and weak compaction of the chromium oxide scale also have an effect on its friction and wear behaviour.

4. CONCLUSIONS

Scratch and pin-on-disk dry sliding tests were carried out to investigate the adhesion and the tribological behaviours of the chromium oxide scale on the stainless steel 445.

The oxide scale formed on the 445 stainless steel showed various adhesion strength and was in a ductile failure under progressive loading by a scratch test. The COF increased with increasing constant load and showed higher values for the oxidised stainless steel 445. The test shows friction between the counterparts and the abrasive wear on the steel 445.

In the dry sliding pin-on-disk test, high-speed steel was used as a pin material. The generated powder-like fine wear particles, mainly the ferritic Fe-Cr substrate, had played roles in forming high-resistance load-bearing layers and in stabilising the COF. The chromium oxide scale formed on the steel 445 can reduce adhesive wear but induce abrasive wear of the HSS steel. The COF was similar when there were oxide scales on the disks. The HSS pin showed mainly abrasive wear at the low load; while it showed abrasive and oxidative wear at the high load. The HSS pin showed severe adhesive wear when there were no oxide scales on the disks.

The thermally grown Cr-rich oxide scale which adhered well to the ferritic stainless steel substrate was beneficial to improving its wear resistance but its ability to reduce the COF is limited.

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Table captions

Table 1 Chemical composition (wt. %) of the ferritic stainless steel 445

Table 2 Chemical composition (wt. %) of the high speed steel

Figure captions

Fig. 1 (a) oxidised 445 steel disk from the furnace, (b) The cross-section of the oxide scale formed on the 445 steel from [32] (c) AFM image and surface roughness of clean bare surface and (d) AFM image and surface roughness of oxidised 445 surface

Fig. 2 The dimensions of the high-speed steel pin and the top view of the pin tip

Fig. 3 (a) coefficients of friction with applied progressive loads from 20 to 150 N on oxidised and clean bare surface of the steel 445. The thicker line indicates COF curves of the oxidised surface. (b) Friction force (FF) and acoustic emission (AE) signals of two surfaces under progressive load with the scratch track images of the oxidised stainless steel surface. Applied load: 0-150 N, the lateral displacement speed: 6 mm/min, and scratch distance: 5 mm

Fig. 4 Variation of coefficient of friction and (b) scratch track depth with applied constant loads by scratch test

Fig. 5 3-D Profiles with scratch tracks on the surface of oxidised ferritic stainless steel 445 under different applied constant loads (a) 5 N, (b) 10 N, (c) 20 N, and (d) 50 N. The black colour in the tracks indicates the Cr-rich oxide scale

Fig. 6 Worn surfaces of pins under loads of 5 and 50 N after dry sliding pin-on-disk tests (a) and (c) HSS pin on clean disk, and (b) and (d) HSS pin on oxidised disk (the arrows indicate sliding direction)

Fig. 7 Coefficient of friction versus sliding time for HSS pins sliding against clean and oxidised ferritic stainless steel disks under 5 and 50 N

Fig. 8 The XRD analysis of wear debris on clean and oxidised disk after dry sliding pin-on-disk tests

Fig. 9 SEM micrographs of worn surface of HSS pin under 5 N after dry sliding pin-on-disk tests (a) on clean disk and (b) on oxidised disk

Fig. 10 SEM and EDS analyses of worn surfaces of the HSS pins under 50 N after dry sliding pin-on-disk tests (a) on the clean disk and (b) on the oxidised disk

Fig. 11 SEM micrographs of worn surfaces of disks (a) clean disk and 5 N, (b) oxidised disk and 5 N, (c) clean disk and 50 N, and (d) oxidised disk and 50 N (the arrows indicate sliding direction)

Fig. 12 Two-dimensional profiles of wear tracks of the 445 disks after dry sliding pin-on-disk tests

Fig. 13 schematic illustration of the wear mechanism (a) HSS pin on bare 445 disk, mainly adhesive wear. Wear debris particles are sintered, compacted and oxidised to the formation of new compound layers on the surface, (b) HSS pin on the oxidised 445 disk, mainly abrasive wear. Wear debris particles include more Cr_2O_3 particles may influence the sintering and attachment.