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In-situ interfacial tribochemistry toward eliminating red-scale of silicon steel in friction process

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ABSTRACT

Irregularly striped red-scale defects are often formed on hot rolled silicon steel strips, and some still remain on the surface even after pickling. In this study, an in-situ interfacial tribochemistry concept was proposed to eliminate red-scale silicon steel using ball-on-disc test, by transforming harder eutectic Fe_2SiO_4/FeO eutectic into other compounds with sodium polyphosphate lubricant. The results show that polyphosphate can lubricate the high speed steel/silicon steel interface effectively with significant reduction of the friction and wear by 50% and 90% respectively. And in situ formed compounds of $Na_3Fe_2(PO4)_3$ and $NaFePO_4$ at the rubbing interface can eliminate red-scale on silicon steel by weakening the adhesion of oxide scale to the substrate.

1. Introduction

Silicon steels have been used extensively as soft magnetic materials in generators, motors, and transformers, and also as high strength steel in automobiles [1–3]. The current trend in their production is to increase the composition of silicon above 5 wt% to optimize the property of silicon steels as required. However, silicon steels with a high concentration of silicon element become brittle and exhibit poor workability [4]. If silicon steels are deformed by hot rolling, the silicon in steels becomes more active and produces higher fayalite content in addition to more generated oxide scale at high temperature and oxygen containing atmosphere.

It has been claimed that the fayalite in the secondary scale is hard and sticky, which causes the non-uniform wear of roll, and leads to poor efficiency of secondary scale descaling and poor surface quality of strips [5]. In fact, about 30% of the yield loss stems from scale-related defects. Red scale defects resulting from the effects of silicon are difficult to remove by conventional hydraulic descaling approaches [6]. It is reported that the formed compound FeO/Fe2SiO4 eutectic (melting point: 1173 °C) with a morphology that anchors to the oxide scale and steel base after solidification and enables the scale to connected firmly to the steel substrate that makes it difficult to descale the red scale [7,8]. So the invasion of the FeO/Fe2SiO4 eutectic compound into the steel substrate greatly hinders the ability to descale [8,9].

Many scientific papers about silicon steels have focused on the formation of red-scale, improving descaling capability by adding alloying elements [10–12] (e.g. P, Ni, Cu, Cetc.) in steel substrate, and also optimize parameters of rolled surfaces (phase constitution, structure and coating, etc.) [5,13,14]. Some researchers reported the lubrication result from in situ tribochemical reaction formed tribolayer [15,16]. Ge et al. [17] reported that in situ tribochemical layer with phosphates included formed at the Si_3N_4 interfaces in IL ([Li (EG)]PF_6) lubrication condition is beneficial to superlubricity and wear protection. Ripoll et al. [18] mentioned that in situ tribo-chemical formation of WS_2 from sulfur carrier additive and tungsten carbide particles contributes to low coefficient of friction of about 0.06 for steel/steel contact. Berman et al. [19] reported that the stress-induced reactions at (OLCs) hydrogenated diamond-like carbon (H-DLC) surface sliding against molybdenum disulfide along with nanodiamonds, lead to the formation of onion-like carbon structures that reduces the contact area and incommensurate contact, which enables superlubricity. Phosphate is well known anti wear and lubrication additives, such as ZDDP, which is likely to in situ form a protective tribofilm induced by thermomechanical pressure and temperature at the contact interface [20–22].

In this work, we plan to make the red-scale more deformable and easily descaled through in situ formed compounds by the introduction of

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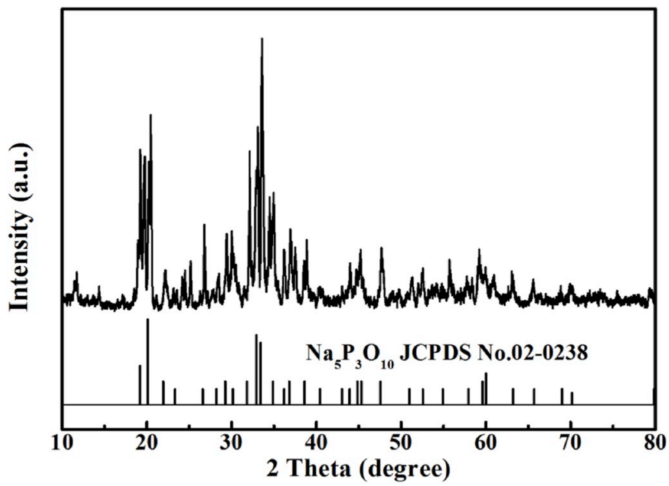

Fig. 1. X-ray diffraction (XRD) pattern of commercial sodium polyphosphate detected at room temperature, standard card of $Na_5P_3O_{10}$ (JCPDS No. 02–0238).

sodium polyphosphate. The aim of this study is to identify the mechanics to reduce red-scale during friction by sodium polyphosphate lubrication, and the in-situ tribochemical reactions at the rubbing interface were studied. Meanwhile, the wear and lubrication mechanism is also provided.

2. Experiment section

2.1. Materials

High speed steel (HSS) is widely used roller material for hot rolling process, so HSS ball was used as the upper counterpart representative of the roller for the ball-on-disc sliding process, its hardness and young's modulus are 803 Hv and 203 GPa, respectively. The disc is made of Si steel (SST) with 1 wt% silicon, with the hardness and young's modulus of 201 Hv and 192 GPa, respectively. The composition of the HSS ball and the Si steel disc (analysed by optical emission spectroscopy) was shown in Table 1. Sodium polyphosphate (NPO) purchased from Sigma-Aldrich were used as lubricant/coating, with the X-ray diffraction (XRD) pattern shown in Fig. 1. This commercial sodium polyphosphate is mainly $Na_5P_3O_{10}$, which is in agreement with reference PDF cards (JCPDS No. 02–0238).

2.2. Preheating process

The Si steel (SST) discs with a diameter of 50 mm and 3 mm in thickness were ultrasonically cleaned by acetone and ethanol, respectively, for 5 min prior to the preheating process. Before heating, the furnace was vacuumed to reach a vacuum of 10^{-1} Pa, and then N_2 gases were introduced with a flow of 2 L/min, then the temperature increases at a rate of 20 °C/min from room temperature to 850 °C, followed by 6.6 °C/min to 1200 °C. After holding at the maximum temperature for 20 min, they were cooled down in the furnace to room temperature. The aim of reheating of the discs is to prepare the analogous fayalite and oxide layer with factory reheating process for subsequent friction tests.

Heat treatment was also carried out in air atmosphere, the SST bulks (10 × 10 × 3 mm) were coated with 16 wt% NPO aqueous solution to reach 0.01 ml/cm^2. Heat treatments temperatures are 1150 and 1200 °C, to make a comparison, the heat treatment experiments were also carried out on the naked bulks without any coating. After heating experiments, the samples were mounted in resin and polished with SiC polishing paper (200–2400 meshes) for cross section observation.

2.3. High temperature friction tests

High temperature friction tests were carried out using a ball-on-disc UMT tribolab machine (Bruker, USA). A HSS ball (minor polished, HRV 65, 6.35 mm in diameter) was fixed in the upper ball holder against the preheated Si-containing disc. This friction tests were conducted at 950 °C to simulate the finishing mill rolling process, during which the temperature of the plate is also 950 °C. Tests were performed at a load of 10 N (maximum Hertz pressure 0.74 GPa), with a sliding radius of 15 mm, sliding velocity of 60 RPM (0.094 m/s). After sliding 50 s, 0.5 g of NPO was introduced into the wear track through a ceramic tube which is above the wear track region. The ceramic tube was located beside the ball holder and inserted through the insulated cover of the heating chamber. The same amount of NPO was introduced every 200 s, and each friction test lasts for 600 s. Dry friction tests were also conducted for comparison. The coefficient of friction is the ratio of Fx (lateral force) and Fz (normal force), which were automatically recorded throughout the duration of the test. Wear rate of the ball were calculated by $W=V/FL$, where V is the wear volume the ball, corresponded with the wear scar diameter [19], which was measured using 3D profilometer, F is the load and L is the sliding length. The tests were repeated three times to confirm the repeatability.

2.4. Descaling experiment

After high temperature friction test, the discs were cut into small coupons for subsequently descaling experiments. The acid solution for pickling is 18 wt% HCl solution at a stable temperature of 70 ± 1 °C. Then the coupons were submerged in a thermostatic and stirred batch reactor containing the hydrochloric acid bath. The images of the disc surface were captured for every 20 s.

2.5. Characterisation

X-ray diffraction technique (GBC MMA) with Cu-Ka radiation source was used to reveal the structure of commercial sodium polyphosphate. The morphology and chemical composition of the cross section and worn surfaces were analysed by JSM-6490LV scanning electron microscopy (SEM) attached with energy dispersive spectroscopy (EDS).

The small lamellar including both phosphate layer and iron oxide was prepared using Dual beam FEI Helios NanoLab G3 CX with Gallium ion source. The produced specimen with a carbon and platinum protective coating from the target area was inserted into a thin foil and stored in a high vacuum instrument to avoid the undesired pollution until subsequent microscopy testing.

The structure and chemical composition of the phosphate and iron oxide layers were assessed by an aberration-corrected JEOL ARM 200f scanning transmission electron microscopy (STEM). High angle annular dark field (HAADF) and bright field (BF) imaging of the specific area were obtained simultaneously. The bonding state of P was identified by an electron energy loss spectroscopy (EELS). The selected area electron diffraction (SAED) was performed at phosphate rich region. The EELS peaks identification was carried by the EELStools in Digital Micrograph

Table 1
Nominal composition of Si steel (SST) disc and high speed steel (HSS) ball (wt.%).

Element	Fe	C	Mn	Si	W	Cr	Mo	V	Mg
SST	97.33	0.0798	1.336	1.001	0.1008				
HSS	87.92–90.15	0.80–0.85		<0.25	<0.25	4.00–4.25	4.00–4.50	0.90–1.10	0.15–0.35