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Oxide scale characterization of ferritic stainless steel and its deformation and friction in hot rolling

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Keywords

characterization, scale, oxide, its, steel, hot, rolling, stainless, friction, ferritic, deformation

Disciplines

Engineering | Science and Technology Studies

Publication Details

Cheng, X., Jiang, Z., Wei, D., Hao, L., Zhao, J. & Jiang, L. (2015). Oxide scale characterization of ferritic stainless steel and its deformation and friction in hot rolling. *Tribology International*, 84 61-70.

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Oxide scale characterization of ferritic stainless steel and its deformation and friction in hot rolling

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Abstract

The oxidation kinetics of ferritic stainless steel 430 was studied in dry and humid air at 1090 °C by Thermo Gravimetric Analysis (TGA). Different atmospheres and heating times were adopted for reheating to obtain different compositions and thickness of the oxide scale. Hot rolling was performed on a 2-high Hille 100 experimental rolling mill at various reductions. Oxide scale thickness and composition were analysed with optical microscopy (OM), scanning electron microscope (SEM) and X-ray diffraction (XRD). The surface profiles were examined by a digital microscope, and the topographic features of the thin oxide scale surface were examined with an atomic force microscope (AFM) before and after rolling. The oxide scale surface and steel/oxide interface roughness were measured after rolling. Inverse calculation of the coefficient of friction was employed to analyse and the effect of oxide scale on friction in hot rolling. The coefficient of friction depends not only on the thickness of the oxide scale, but also on its composition and surface topography before hot rolling.

Keywords: Ferritic stainless steel; High temperature oxidation; Hot rolling; Oxide scale; Friction

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1. Introduction

Surface quality and appearance are especially important for stainless steels [1]. The oxide scale structure formed upon hot rolling is of crucial importance in the downstream performance of the steel strips in such processes as pickling, cold rolling, tempering and electrolytic tinning. In hot rolling, the strip is exposed not only to high temperatures but also to a very humid atmosphere. During hot rolling, water vapour content levels in the air vary between 7.0 and 19.5 vol. % [2]. Oxide scale on the surface of the steel strip separates the rolls and the metal substrate and affects not only the friction conditions but also the heat transfer between the strip and the rolls [3-4]. High friction usually results in excessive load and damage to the surface of the rolled products [5].

Few studies have been carried out to characterise the oxidation and deformation behaviour of the oxide scale in the hot rolling of stainless steels. The oxidation of stainless steels is significantly affected by alloying elements and atmosphere, and this means that its oxidation is more complex than that of carbon steels. At ambient temperature, stainless steels have a thin, dense and tightly adhering chromium oxide layer which resists corrosion and oxidation [6]. In a reheating furnace, however, where the temperature can reach 1200 °C, the chromium oxide gradually loses its protective capability and breakaway oxidation occurs. The formation of non-protective iron-rich oxide scale marks the failure of the chromia scale and the start of breakaway oxidation [7]. The growth of the oxide scale follows a parabolic relationship over time, due to the diffusion of cations or anions in the oxides [8]. The two major potential components of a stainless steel oxide scale are M_2O_3 rhombohedral phase and M_3O_4 spinel phase [9]. The oxide scale formed on 304 hot rolled strips [10] contains spinel constituents

such as FeCr_2O_4 , NiFe_2O_4 , Fe_3O_4 and Fe_2O_3 while the oxide scale formed on 430 hot rolled strips [11] consists of FeCr_2O_4 , Fe_3O_4 and Fe_2O_3 .

The deformation behaviour of the oxide scale depends on its thickness [12], chemical composition [13-14] and rolling parameters [3, 15-17]. These components affect the friction and surface qualities of the steels. Most studies on oxide scale deformation have been based on carbon steels which consist of iron oxides, and FeO is always the main constituent of the oxide scale [18]. Munther and Lenard [3] found that the oxide scale thickness appears to have a greater effect on friction than the oxide scale composition, and thicker scales give lower friction values. The same result was also confirmed by Luong and Heijkoop [13]. Suárez et al. [19] concluded that the deformation temperature is the important factor affecting the plastic behaviour of the oxide scale formed on ultra low carbon steel. Krzyzanowski et al. [20-21] have investigated the failure of oxide scales formed on mild steel using high temperature tensile tests because of a longitudinal tensile stress in the stock surface ahead of contact with the roll. The oxide scale can have through-thickness cracks or delamination [20, 22]. Jin et al. [23] have conducted hot rolling experiments on ferritic stainless steel 430 and have found that the thick oxide scale does not always play a positive role in reducing the coefficient of friction. However, their experiments were only concerned with the thickness of the oxide scale. Oxide scale failure on AISI430 ferritic stainless steel by mechanical de-scaling was investigated both experimentally and numerically [24-25], but it is a process after hot rolling. Wei et al. [26-27] have investigated the deformation of oxide scale and the roll-strip interface characteristics in the hot rolling of austenitic stainless steels. It was found that oxide scale thickness decreases with an increase of rolling reduction, and higher reduction and thinner oxide scale result in better surface at the rolling temperature of 1050 °C.

This study focuses on the oxide scale grown on ferritic stainless steel 430. An oxidation kinetics study was carried out by using a thermogravimetric analyser (TGA) under two kinds of atmospheres: air enriched with water mist and static dry air. TGA results show that different thicknesses, surface morphologies and compositions of the oxide scale were formed. During the hot rolling experiments, various atmospheres and times in the reheating furnace were adopted. The oxide scale generated on the 430 steel surfaces was examined with respect to its effects on the hot rolling characteristics. The objectives of this study were to examine the oxide scale thickness, surface morphology, composition, rolling reduction on the coefficient of friction and surface roughness of the steel.

2. Experimental details

2.1. Material

A 430 ferritic stainless steel was selected in this investigation and its chemical composition is shown in Table 1. All the material used in the experiment was taken from hot rolled strips.

2.2. Oxidation kinetics test

The sample for the oxidation kinetics study was $20 \times 10 \times 1 \text{ mm}^3$ small plates. Prior to the oxidation experiments, the plates were ground on all sides with 1200 grit SiC sandpaper, then cleaned in acetone and subsequently rinsed with alcohol.

The oxidation kinetics of stainless steel was investigated using a thermogravimetric analyser (TGA). Oxidation tests in humid air were performed in the apparatus shown in Fig. 1. Before

the test, temperature calibration was carried out. The sample was put in a vertical tube furnace and isothermally heated for 120 min. The mass change of the sample was measured using a Sartorius CP124S microbalance with a resolution of 10^{-4} g and the data was logged onto a computer. The device in Fig.1 which can generate humid air was connected to the bottom of the tube furnace. 18% water vapour content was selected to flow into the furnace at a rate of 1 litre/min by bubbling synthetic air through the water bath and the water temperature was set at 58.4 °C. The gas inlet lines were heated to prevent water vapour condensation. The total gas pressure was 1 atm.

The tube furnace was heated to the set temperature at a heating rate of 20 °C/min, then humid air flowed through the tube furnace. The furnace was held at the set temperature for 30 min, then the steel sample was lowered into the hot zone of the furnace suspended on a silica wire and the weight change logged. An oxidation test in static dry air was performed in the same vertical tube furnace but with an open top end in order to allow air to enter the tube furnace. After a set time, the experiments were completed by immediately removing the sample from the furnace and cooling it in air to room temperature.

Above 850°C the gas velocity also affects the oxidation rate on low carbon steel [28]. The oxidation rate increases with an increase of gas velocity until a critical gas velocity is reached. Basabe et al. [29] have investigated the concentration of free oxygen in the heating atmosphere, and shown that it has a significant effect on the scale morphology. The oxygen pressures do not vary the thickness of the FeO layer when the FeO accounts for 95% of the scale thickness [30]. In our study, flow gas was used to bring humidity to the furnace in order

to simulate a humid environment, and to compare the steel oxidation behaviour with that in a dry air environment.

2.3. Reheating and hot rolling test

The material used was cut from the hot rolling slab and the specimens were machined to the size of 100(W) × 400(L) × 8(H) mm. In order to help bite the sample, the front of the specimen was machined to a thickness of 1 mm and a length of 80 mm. The surface of the specimens was ground and the measured surface roughness R_a was 0.25 μm . Small samples of 15(W) × 15(L) × 8(H) mm³ were cut from the same material with the same surface roughness for the oxidation test and were put with the rolling specimens in the electric resistance furnace to study the composition and the thickness of the oxide scale before hot rolling.

Reheating was carried out in a high temperature electric resistance furnace with a chamber size of 350(W) × 330(H) × 870 (D) mm³. Three heating environment programs were adopted: (a) static dry air for 120 min; (b) humid air for 120 min; (c) humid air for 25 min. In order to simulate a humid atmosphere in the reheating furnace, the device shown in Fig. 1 was connected to the furnace and 18% water vapour content was selected to flow into the furnace at 15 litres/min. Like the TGA experiment, the gas inlet line was heated to prevent water vapour condensation. The reheating temperature was 1090 °C and the entry temperature was 1050 °C.

Hot rolling experiments were carried out on a 2-high Hille 100 experimental rolling mill with rolls of 225 mm diameter and 254 mm roll body length. Rolling forces were measured by load cells on the mill. Rolling speed can be set from 0.12 to 0.72 m/s. In this study, the rolling

speed was kept as 0.72 m/s. Reductions of 25, 35 and 45% were selected. All the specimens were thoroughly cleaned with acetone before heating, and all the specimens were placed immediately in a cooling box with nitrogen gas after rolling or reheating to prevent further oxidation.

2.4. Observation and analysis

The surface of the small and the rolled samples was covered with a mixture of epoxy resin and hardener to protect the oxide scale.. After 24 hours the resin coagulated, and then the protected oxide scale part was sectioned along the rolling direction by a Stuers Accutom50 Cutting Machine to obtain the cross-section of the oxide scale. Finally, the oxide scale sample was cold mounted, ground and polished and then etched for metallographic examination. To analyze the scale cross-section, all oxidised samples were prepared using a technique reported by Chen and Yuen [31] to minimise preparation damage to the oxide scale. The scale morphology was revealed by using an etchant containing 1% HCl diluted in ethanol.

The microstructures, composition and thickness of the oxide scale were examined by LEICA optical microscopy (OM) and a JEOL JSM 6490 scanning electron microscope (SEM) equipped with an Energy Dispersive Spectrometer (EDS). The surface profiles were examined using a VHX-1000 digital microscope with 2D/3D imaging and measurement capability. Topographic features of the thin oxide scale surface were examined using an atomic force microscope (AFM) before and after rolling. X-ray diffraction was used for the phase identification of the oxide scale.

3. Results and discussion

3.1. Oxide scale growth kinetics at 1090 °C in two atmospheres

Fig. 2a presents the isothermal oxidation kinetics, the weight-gain curve vs. time for stainless steel 430 specimens oxidised at 1090 °C for 120 min in humid air containing 18% water vapour, and also in static dry air. Fig. 2b is the parabolic plot of weight gain as a function of the square root of the time. The incubation time is 4 min in humid air and 6 min in static dry air before breakaway oxidation occurs, as indicated by the arrows in Fig. 2b. The breakaway oxidation is determined by the turning point of the high oxidation rate, and it means the failure of protective chromia scale and the formation of iron rich oxide [6]. Compliance with the parabolic rate indicates that the reaction is of a diffusion-controlled character at two atmospheres [32]. High temperature oxidation kinetics of metals or alloys is commonly controlled by the diffusion of cationic or anionic species through the oxide scale [8]. Such control leads to a parabolic rate constant, K_p , expressed in $\text{mg}^2 \text{cm}^{-4} \text{s}^{-1}$, and defined by $(\Delta m/s)^2 = A + K_p t$ [8], where $\Delta m/s$ is the specific weight gain per area unit [$\text{mg} \cdot \text{cm}^{-2}$], t is the oxidation time and A is a constant. In the process of breakaway oxidation, (see Fig. 2b) the oxidation rate is higher in humid air than in static air.

The parabolic rate constant for steel 430 oxidised in humid air containing 18% water vapour after breakaway oxidation is $0.93 \text{ mg}^2 \text{cm}^{-4} \text{s}^{-1}$ and in static dry air, it is $0.07 \text{ mg}^2 \text{cm}^{-4} \text{s}^{-1}$. Water vapour has a significant effect on the oxidation rate of steel 430 after breakaway oxidation, facilitating rapid inward transport of oxygen and outward transport of Fe ions through the Fe-Cr spinel to form iron oxides. Some researchers have observed that the water vapour induces a breakaway oxidation during high temperature oxidation of iron-based

chromia forming alloys [33-37]. This phenomenon depends upon the oxidation time, the atmosphere and the chromium alloy content [36].

3.2. Composition and morphology of oxide scale before hot rolling

Before the hot rolling test, the small samples placed in the reheating furnace with the rolling specimens were examined. The small samples were removed from the furnace by the operator and placed in the cooling nitrogen box, and then the rolling specimen was removed from the furnace and immediately rolled. Uniform surface appearances were observed on all oxidised samples. The oxide scale on the surface of the sample oxidised in static dry air for 120 min appears black, and part of its oxide scale is spalled off during cooling. The oxide scale on the surface of the sample oxidised in humid air for 120 min appears bright grey, and in humid air for 25 min the surface appears dark grey with a small area covered with bright grey oxide. The oxide scale spallation was not observed for the latter two samples, indicating that the adhesion of the oxide scale is improved in a humid reheating environment.

A conventional diffraction meter operating with Cu K α radiation was used for X-ray analysis. The external layers of the oxide scale formed in static dry air and humid air for 120 min were peeled off for the phase detection, and surface scan was carried out for the thin oxide scale oxidised in humid air for 25 min.

The outer oxide layer is mainly composed of Fe₂O₃ and Fe₃O₄ in dry air and Fe₂O₃, Fe₃O₄ and FeO in humid air, as shown in Fig. 3a. The inner oxide layer is mainly composed of

spinel $(\text{Fe, Cr})_3\text{O}_4$. For the sample oxidised for 25 min in humid air, the oxide scale is composed of Cr_2O_3 and Fe_2O_3 , as shown in Fig. 3b.

The cross section, surface morphology and profile of oxide scale of stainless steel 430 formed in static dry air for 120 min, humid air for 120 min and 25 min are shown in Fig. 4.

In the study, the outer oxide scale was defined as iron oxides, including Fe_2O_3 , Fe_3O_4 and FeO . The inner oxide scale was defined as the spinel $(\text{Fe,Cr})_3\text{O}_4$. The cross-section of the oxide scale in Fig.4 a-b indicates a clear boundary between the outer and the inner oxide scale. The separation of the multi-scale was caused by the fast cooling. During temperature changes, the difference between the thermal expansion coefficients of the metal and the oxides result in stresses being developed within the oxide layer [38]. Compressively stressed oxide layers, e.g. during cooling, are susceptible to spallation [39].

The oxide scale surface formed in static dry air is rougher than that in humid air. The forest-like surface formed in static dry air leads to a dark appearance, displaying the roughest surface. Denser and bigger oxide grains with fewer pores formed in humid air appear bright grey and the surface is not as rough as that formed in dry air. In Fig. 4a and b, pores can be seen in the outer and the inner oxide layers. The lowest surface roughness is observed for oxide scale formed in humid air for 25 min. Fig. 4c shows that grinding marks can be seen from the oxidised surface, indicating that the oxide scale is thin.

3.3. Deformation of oxide scale

Fig. 5 shows the surface morphologies of the oxide scale after cooling when the rolling reduction changed from 0 to 45%. The photos were taken from the samples rolled at the highest reduction. Fig. 5a shows that the oxide scale formed in static dry air remains integral after rolling, but the oxide scale without deformation is spalled off. The surface appears reddened because the Fe_2O_3 is pulverised into small particles [40]. For the oxide scale formed in humid air for 120 min, the surface is reddened gradually under deformation from low to high reduction and spalling does not occur. The surface of the oxide scale formed in humid air for 25 min gradually brightens from low to high reduction. The area close to the sample edge appears red due to the excessive oxidation and the formation of Fe_2O_3 on the edges. Extra stresses are generated within the scale grown near the edges of the specimen, allowing cracks to form in the chromia scale, which can accelerate the breakaway oxidation [41].

Fig. 6 shows the surface morphology and cross-section of the oxide scale (along the rolling direction) on the samples that were rolled at the low reduction (20-25%) at 1050 °C. Fig. 6a shows the deformation of the sample oxidised in static dry air for 120 min. Transverse outer oxide layer cracks appear close to the sample edges. SEM surface morphology shows the deformed inner oxide layer between the outer oxide layer cracks (The area is indicated by enclosed purple dots), displaying no inner oxide layer cracks. The cross-section of the deformed oxide scale exhibits a very clear boundary between the outer and the inner oxide layer, indicating no interference of iron oxides or the spinel oxide. The outer Fe_2O_3 oxide is pulverised, and the Fe_3O_4 is fragmented. The Fe-Cr spinel is deformed uniformly and is cracked. Fig. 6b shows the deformation of the sample oxidised in humid air for 120 min. The surface appears grey and some Fe_2O_3 oxide grains can still be seen, exhibiting no cracks. The

cross section of the deformed oxide scale exhibits a very clear boundary between the outer and the inner oxide layer. The outer iron oxides are fragmented. The inner Fe-Cr spinel is fractured and exhibits integral through-thickness cracks. Fig. 6c shows the deformation of the sample oxidised in humid air for 25 min. The elongated surface appears uniform and the grinding texture is similar to that before rolling. The cross-section of the deformed oxide scale shows that it is deformed uniformly.

Fig. 7 shows the surface morphology and cross-section of the oxide scale (along the rolling direction) on the samples that were rolled at the medium reduction (37-38%) at 1050 °C. Fig. 7a shows the deformation of the sample oxidised in static dry air for 120 min. Some transverse outer oxide layer cracks appear close to the sample edges and some cracks in the middle part of the sample. There are still no surface cracks in the deformed inner oxide layer (The area is indicated by enclosed purple dots). The cross-section of the deformed oxide scale shows that the inner Fe-Cr spinel is extruded from the cracks of the outer iron oxide scale layer and some Fe-Cr spinel is also fragmented. Fig. 7b shows the deformation of the sample oxidised in humid air for 120 min. The surface appears to be red and most Fe₂O₃ oxide is pulverised. The cross-section of deformed oxide scale shows some but not complete separation of the Fe-Cr spinel and the steel substrate extruded from it. The steel/oxide interface becomes rough but the outer iron oxides still cover the surface evenly. Fig. 7c shows the deformation of the sample oxidised in humid air for 25 min. The surface appears uniform and the grinding texture can still be seen but not as clearly as that in Fig. 6c. The cross-section of the deformed oxide scale shows that the thin oxide scale is deformed uniformly without exhibiting cracks.

Fig. 8 shows the surface morphology and cross-section of the oxide scale (along the rolling direction) on the samples that were rolled at the high reduction (45-50%) at 1050 °C. Fig. 8a shows the deformation of the sample oxidised in static dry air for 120 min. Transverse outer oxide layer cracks appear all over the surface and some surface cracks begin to appear in the deformed inner oxide layer. The cross-section of the deformed oxide scale shows that the inner Fe-Cr spinel is fragmented and interferes with the outer pulverised iron oxides. Fig. 8b shows the deformation of the sample oxidised in humid air for 120 min. The surface appears to be red and the Fe_2O_3 oxide is pulverised. The cross-section of the deformed oxide scale shows that more and more Fe-Cr spinel separates from the cracks and appears like solid blocks. The steel substrate is extruded from the Fe-Cr spinel cracks but is covered with outer fragmented iron oxides. The steel/oxide interface is very rough. Fig. 8c shows the deformation of the sample oxidised in humid air for 25 min. The surface appears uniform and no grinding texture can be seen. It can be seen from the insert in the SEM surface micrograph that through-thickness oxide scale cracks begin to appear. The cross-section of deformed oxide scale shows that the scale is not deformed uniformly and the steel/oxide interface is not as even as it is at the lower reductions.

The measured thicknesses of the oxide scales before and after hot rolling are shown in Fig. 9. For the multi-scale layers formed in static and humid air for 120 min, the inner Fe-Cr spinel has approximately half of the whole oxide scale. The deformation of the Fe-Cr spinel has major effects upon the deformation of the whole oxide scale, affecting the uniformity of the deformed oxide scale and the roughness of the steel/oxide interface. The oxide scale of 397.6 and 245 μm display good ductility at the reduction lower than 25%. However, the oxide scale of 397.6 μm in which the Fe-Cr spinel lacks plasticity at higher reductions, leads to the reduction of the oxide scale less than the bulk steel reduction. The Fe-Cr spinel formed in

static dry air with the thickness of 200 μm shows better ductility than that in the oxide scale of 397.6 μm because the thinner Fe-Cr spinel is fragmented at high reduction, and then its flow stress is improved. The thinnest oxide scale of 11.5 μm which was formed in humid air for 25 min, with Cr_2O_3 and Fe_2O_3 as the main components, shows better ductility than the multi-layer thick oxide scales. At the bulk reduction of 21.2, 37.8 and 46%, the oxide scale reduction is 33, 63 and 76%, respectively.

At the low reduction of 25%, the multi-layers of oxide scale display ductility-like behaviour. The outer and inner oxide layers are deformed uniformly in their own layers. This can be explained by the compatibility of the oxides during hot rolling. Before rolling, the pores are observed in the oxides, after rolling, the porosity and minor cracks in the oxide disappear. The inner porous layer with high porosity is weak [29] and has the potential to cause delamination within the oxide scale by tensile stress [20]. The pores observed in the Fe-Cr spinel, instead, are small and are expected to ease the compression stress caused by rolling [40].

3.4. Surface and steel/oxide interface roughness

After rolling, the surface roughness R_a (Arithmetical mean roughness) and the steel/oxide interface roughness R_y (Maximum peak from the steel/oxide interface of the scale cross-section) were measured. The effects of rolling reduction on the oxide surface and the steel/oxide interface roughness were examined. The results are shown in Fig. 10.

Bulk deformation significantly affects the surface finish of the sample [42]. It can be seen from Fig. 10a, with increased bulk deformation, the oxide surface roughness is greatly decreased when the oxide scale is thick. In the case of the thin oxide scale formed in humid air for 25 min, however, the oxide surface roughness does not decrease at the high reduction of 46%, but rather increases. The oxide surface roughness is consistent with the steel/oxide interface roughness when this thin oxide scale is deformed. For the oxide scale of an initial thickness of 397.6 μm , the steel/oxide interface roughness R_y increases with increasing reduction. For the scale of an initial thickness of 200 μm , the steel/oxide interface roughness R_y increases at the reduction of 37% but decreases at the high reduction of 47.5%.

Hidaka et al.[40] concluded that plastic deformation by dislocation would occur to some extent in Fe_3O_4 and FeCr_2O_4 crystals, but they would be broken when under high rolling reduction. The dislocation density of FeCr_2O_4 was observed comparatively low, and this type of plasticity was classified as work hardening [43] and the mechanism is explained as dislocation glide or grain boundary sliding [44]. In our study, the deformation of the oxide scale formed in static dry air agrees with their study. The fragmented Fe-Cr spinel seems to have better ductility to improve the steel/oxide interface (Fig. 10b). However, the very thick Fe-Cr spinel formed in humid air for 120 min, instead of being fragmented at the high reductions, was fractured. Therefore, the steel substrate was extruded through the Fe-Cr spinel cracks, causing a rough steel/oxide interface.

Fig. 11 shows the topographic 3D images and section analysis of the oxide scale surface by AFM when the oxide scale was formed in humid air for 25 min. Before rolling, the oxide scale surface exhibits crystallite structures. After rolling, the crystallite-like surface

disappears and displays fine particles. However, as shown in Fig. 11c, at the highest reduction there are some big particles appearing and the surface looks rougher. This phenomenon is caused by the extruding of steel substrate from the cracks in the oxide scale. The value of the maximum vertical distance between the peak and the valley is consistent with the value measured in macro-scales.

3.5. Inverse calculation of coefficient of friction

The coefficient of friction is calculated by a formula developed by Alexander [45] on the basis of Oroman's model.

$$\frac{d(\sigma_x h)}{dx} + p \frac{dh}{dx} \mp 2\mu p = 0 \quad (1)$$

where p is the roll pressure, h is the strip thickness, σ_x is the stress in the rolling direction and x is the distance in the direction of rolling. $\mp$ sign indicates the conditions of equilibrium between the neutral point and the entry (negative sign) or between the neutral point and the exit (positive sign) [46].

The coefficient of friction is calculated until the calculated separating rolling force is less than 1% error with measured value. The detailed calculation can be found in elsewhere [47].

According to [48], the yield stress of the steel can be described as

$$\sigma = \sigma_0 \cdot e^{-aT} \cdot k_1 \varepsilon^{m_1} \cdot k_2 \dot{\varepsilon}^{m_2} \quad (2)$$

where σ is the yield stress, σ_0 the base yield stress, ε and $\dot{\varepsilon}$ the true strain and strain rate (s^{-1}) respectively, T the temperature in K , and a , k_1 , k_2 , m_1 and m_2 are constants.

In Alexander's program, the flow stress is modified in the following form [45].

$$\sigma = A \cdot (1 + B\varepsilon)^{n_1} \cdot (1 + D\dot{\varepsilon})^{n_2} \quad (3)$$

in which $A = \sigma_0 \cdot e^{-aT}$, where B , D , n_1 and n_2 are constants. Assuming $D = B = 1000 \gg 1$, Equation (3) still complies with Equation (2). Sufficiently, A , n_1 and n_2 in Equation (3) are determined by multiple-regression.

The output parameters are total rolling force, roll pressure, roll torque per width, angle in the roll bite, nominal pressure, neutral angle in radians, and the forward slip, defined as the relative difference in roll surface/strip exit velocity. The heat transfer coefficient of the scale is not considered in this model.

Fig. 12 shows the effect of reduction on the separating rolling force and the calculated coefficient of friction for different oxide scales on ferritic stainless steel 430. As can be seen from Fig. 12b, the calculated coefficients of friction increase with an increase of reduction. The thickest oxide scale formed in humid air for 120 min shows the lowest coefficient of friction at all different reductions, indicating that the steel/oxide interface has little effect on the contact friction during hot rolling. At reduction lower than 40%, the oxide scale formed in static air displays the highest coefficient of friction, and this is caused by its rough surface before hot rolling (Fig. 4a) and the outer oxide scale layer cracks in hot rolling (Figs. 7a, 8a, and 9a), although it is thicker than that formed in humid air for 25 min. However, at the highest reduction of 46%, the surface with the thinnest oxide scale formed in humid air for 25 min displays the highest coefficient of friction. The SEM micrograph (Fig. 8c) and the AFM surface topographic 3D micrograph (Fig. 11d) show the extruding steel substrate from the oxide scale cracks. This phenomenon has a significant effect upon its surface roughness after rolling (Fig. 10a) and also the contact friction in hot rolling.

The oxide scales formed on stainless steels at high temperature are complicated. In our study, different heating atmospheres and heating times resulted in forming oxide scales with different compositions, thickness and surface topographies. The adherence property between the oxide scale and the steel substrate is also affected by the alloy elements and heating atmosphere [9, 14]. All these factors cause the complex effects of oxide scale of stainless steels on friction in hot rolling [27, 49].

When the oxide scale is formed on carbon steel at high temperature, the relative thickness of FeO: Fe₃O₄: Fe₂O₃ are in the ratio of roughly 95:4:1 at 1000° C [50]. Funke et al. [2] concluded that the hardness of oxides is temperature-dependent. The hardness of FeO, Fe₃O₄, and Fe₂O₃ are 460, 540 and 1050 HV, respectively, at room temperature [13]. However, at 900°C, the above values drop to 105, 366 and 516 HV, respectively [51]. Plastic deformation has occurred for FeO tested above 700 °C [52]. In our study, the ratio of iron oxides and Fe-Cr spinel formed on steel 430 is roughly 1:1 at 1090 °C. FeO is only present in the oxide scale formed in humid air for 120 min. Previous research has shown that the hardness of the oxides formed on the steel is affected by the alloy elements, oxidising atmosphere and time [53]. In our study, the measured vicker hardness of Fe-Cr spinel is between 452 and 565HV at room temperature. Iron oxides display the same characteristics during hot rolling of the stainless steel, especially at high reductions. They are pulverised or fragmented and cover evenly on the rolled steel, acting likes a lubricant. The properties of the Fe-Cr spinel, however, are different. The thin Fe-Cr spinel is prone to be fragmented and the thick Fe-Cr spinel tends to fracture at the high reduction during hot rolling, but this phenomenon has little

effect on the coefficient of friction because the extruding steel substrate from the cracks in the Fe-Cr spinel is still covered with iron oxides.

4. Conclusions

In this paper, the oxidation kinetics of ferritic stainless steel 430 was studied in dry and humid air by TGA. Hot rolling tests were carried out on a 2-high Hille 100 mill.

The incubation time before breakaway oxidation is within 6 min in both atmospheres at 1090 °C but shorter in humid air. The parabolic rate constant for steel 430 oxidised in the humid air is much higher than that in static dry air.

Before hot rolling, the oxide scales formed in static dry air for 120 min at 1090 °C are constituted of Fe_2O_3 , Fe_3O_4 and Fe-Cr spinel. The oxidised sample surface has the highest roughness. In humid air for 120 min, the oxide scales are constituted of Fe_2O_3 , Fe_2O_3 , FeO and Fe-Cr spinel. The oxidised sample surface has lower surface roughness than that in dry air. In humid air for 25 min, the oxide scales are mainly constituted of Cr_2O_3 and Fe_2O_3 . The oxidised surface displays the lowest surface roughness.

Thick Fe-Cr spinel is difficult to deform plastically and tends to be fractured whereas thinner Fe-Cr spinel is prone to be fragmented at a high reduction and displays improved ductility. The outer layers of iron oxides are pulverised or fragmented, covering the surface evenly in hot rolling. The thin scale with Cr_2O_3 and Fe_2O_3 as its main component shows the best

ductility in rolling, decreasing lineally with an increase of reduction, but with a high reduction of 46%, steel substrate is observed extruded from the cracks of the oxide scale, causing higher surface roughness.

The coefficient of friction increases with an increase of reduction. The coefficient of friction is not only dependent on the thickness of the oxide scale, but also on its composition and surface topography before hot rolling. When the reduction is lower than 40%, the medium thickness oxide scale formed in static dry air for 120 min displays the highest coefficient of friction. As the reduction increases to 46%, the thinnest oxide scale formed in humid air for 25 min displays the highest coefficient of friction.

Acknowledgements

The authors acknowledge the Baosteel-Australian Joint Centre financial support for the current project under Grant number BA110017. The authors are grateful for the assistance from Dr. Brian J. Monaghan and Dr. Raymond J. Longbottom while the TGA oxidation tests were carrying out. The authors wish to gratefully acknowledge the help of Mr. Bob De Jong and Dr. Madeleine Strong Cincotta in the final language editing of this paper.

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Table Captions:

Table 1 Chemical composition (wt. %) for 430 steel specimens

Figure Captions:

Fig. 1. Schematic diagram of the experimental apparatus

Fig. 2. Oxidation kinetics of the steel 430 at two atmospheres. (a) $\Delta m/s-t$ plots and (b) $\Delta m/s - t^{1/2}$ plots.

Fig. 3. XRD patterns of the oxide scales formed on the steel 430. (a) The peeled off external oxide scale in dry and humid air for 120 min and (b) Humid air for 25 min.

Fig. 4. The cross section of the oxide scale formed on the steel 430 (a) in static dry air for 120 min; (b) in humid air for 120 min; and (c) in humid air for 25 min. (a1-c1)-surface morphology without removing surface scale and (a2-c2)-surface profile. O and I in (a) and (b) indicate the outer and inner oxide scale respectively.

Fig. 5. Surface morphologies of oxide scale when reduction is from 0 to 45%: (a) static dry air for 120 min; (b) humid air for 120 min and (c) humid air for 25 min. RD-rolling direction.

Fig. 6. (a) Oxide scale formed in static dry air for 120 min at the reduction of 24%; (b) Oxide scale formed in humid air for 120 min at the reduction of 23.7%; and (c) Oxide scale formed in humid air for 25 min at the reduction of 21.2%. 1-Surface morphology; 2-SEM surface micrograph (deformed inner oxide scale is indicated by the area with enclosed purple dots); 3-Cross- section of the oxide scale along the rolling direction.

Fig. 7. (a) Oxide scale formed in static dry air for 120 min at the reduction of 37%; (b) Oxide scale formed in humid air for 120 min at the reduction of 38%; and (c) Oxide scale formed in humid air for 25 min at the reduction of 37.8%. 1-Surface morphology; 2-SEM surface micrograph (deformed inner oxide scale is indicated by the area with enclosed purple dots); 3-Cross section of the oxide scale along the rolling direction.

Fig. 8. (a) Oxide scale formed in static dry air for 120 min at the reduction of 47.5%; (b) Oxide scale formed in humid air for 120 min at the reduction of 50%; and (c) Oxide scale formed in humid air for 25 min at the reduction of 46%. 1-Surface morphology; 2-SEM surface micrograph; 3-Cross section of the oxide scale along the rolling direction.

Fig. 9. Thickness of oxide scale on the steel 430 after the hot rolling tests. D-the oxide scale formed in static dry air for 120 min; D-S-Fe-Cr spinel in D; H-the oxide scale formed in humid air for 120min; H-S-Fe-Cr spinel in H; H-T-the oxide scale formed in humid air for 25 min.

Fig. 10. Effect of reduction on (a) surface roughness R_a and (b) steel/oxide interface roughness R_y . D-the oxide scale formed in static dry air for 120 min; H-the oxide scale formed in humid air for 120min; H-T-the oxide scale formed in humid air for 25 min.

Fig. 11. AFM 3D image and section analysis of the rolled steel product surface with oxide scale formed in humid air for 25 min: (a) before rolling; (b) at the reduction of 21.2%; (c) at the reduction of 37.8%; and (d) at the reduction of 46%.

Fig. 12. The effect of reduction on (a) the rolling separating force and (b) the calculated coefficient of friction. D-the oxide scale formed in static dry air for 120 min; H-the oxide scale formed in humid air for 120min; H-T-the oxide scale formed in humid air for 25 min.

Table 1 Chemical composition (wt. %) for 430 steel specimens

	C	Si	Mn	S	P	Cr	Ni	Cu
Steel 430	0.04	0.3	0.4	0.002	0.021	16.2	<0.20	0.03

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10

Fig. 11

Fig. 12