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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.

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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 [7]. The formation of non-protective iron-rich oxide scale marks the failure of the chromia scale and the start of breakaway oxidation [8]. The growth of the oxide scale follows a parabolic relationship over time, due to the diffusion of cations or anions in the oxides [9]. The two major potential components of a stainless steel oxide scale are M_2O_3 rhombohedral phase and M_3O_4 spinel phase [10]. The oxide scale formed on 304 hot rolled strips [11] 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 [12] consists of $FeCr_2O_4$, Fe_3O_4 and Fe_2O_3 .

The deformation behaviour of the oxide scale depends on its thickness [13], chemical composition [14,15] and rolling parameters [3,16–18]. 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 [19]. 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 [14]. Suárez et al. [20] 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. [21,22] 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 [21,23]. Jin et al. [24] have conducted hot rolling experiments on

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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 [25,26], but it is a process after hot rolling. Wei et al. [27,28] 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

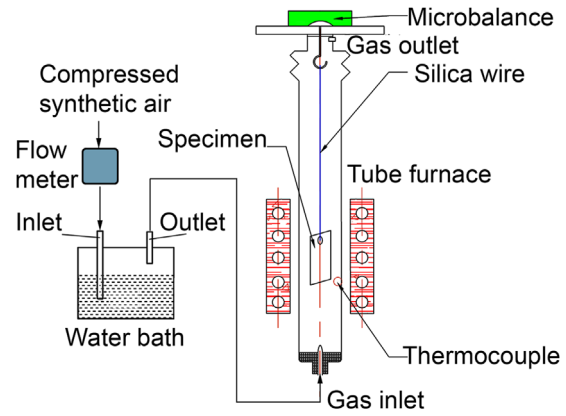


Fig. 1. Schematic diagram of the experimental apparatus.

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 [29]. The oxidation rate increases with an increase of gas velocity until a critical gas velocity is reached. Basabe et al. [30] 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 [31]. 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 $100(W) \times 400(L) \times 8(H) \text{ mm}^3$. 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 $15(W) \times 15(L) \times 8(H) \text{ mm}^3$ 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 $350(W) \times 330(H) \times 870(D) \text{ mm}^3$. Three heating environment programs were adopted: (a) static dry air for 120 min; (b) humid air for 120 min; and (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.

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

	C	Si	Mn	S	P	Cr	Ni	Cu
Steel 430	0.04	0.30	0.40	0.002	0.021	16.20	< 0.20	0.03

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