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# Characteristics of oxide scale formed on ferritic stainless steels in simulated reheating atmosphere

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#### ABSTRACT

The aim of this study is to optimise the stainless steel oxidation behaviours during hot rolling. The high temperature oxidation behaviours of ferritic stainless steels B443NT and B445J1M were studied over the temperature range from 1000 to 1150 °C in a humid atmosphere containing 18% water vapour, as measured by a thermogravimetric analyser (TGA). The results indicate that breakaway oxidation occurs at 1090 °C for the B443NT steel, which is 60 °C lower than that for the B445J1M steel. The occurrence of iron oxide nodules on the steels marks the onset of breakaway oxidation; however, the breakaway oxidation phenomenon of B445J1M is different from that of B443NT due to a compact and continuous Mn–Cr spinel which is formed on the surface of B445J1M. The oxide nodules with regenerated  $Cr_2O_3$  scale underneath the Fe–Cr spinel display better adhesion without showing pores at the metal–scale interface.

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#### 1. Introduction

Ferritic stainless steels containing little or no nickel are widely used because of their excellent resistance to oxidation and corrosion, as well as their lower cost. Ferritic stainless steels have a lower thermal expansion coefficient than austenitic steels do [1], and this is clearly an advantage when temperature cycling resistance is needed, such as in ferritic stainless steel exhaust catalysis units for automobiles. It is more difficult for oxide scale to be generated on the surface of stainless steels than on the surface of carbon steels at processing (e.g. hot rolling) temperatures because of their excellent oxidation resistance. The rolls and strips may stick together due to the lack of oxide scale protection during the hot rolling of stainless steel strips, and the surface quality of the final products will be worse [2]. Sticking refers to a phenomenon occurring in the hot rolling process in which fragments of a rolled material are detached and get stuck onto a work roll surface, deteriorating the surfaces of both the rolls and the rolled material. With the development of new ferritic stainless steels with a high amount of Cr, it is necessary to understand the oxidation characteristics of such ferritic stainless steels in the reheating environment.

In the reheating furnace, the temperature can be as high as 1200 °C. The atmosphere in such furnace generally contains moisture due to natural gas combustion and/or humid conditions. The effect of water

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http://dx.doi.org/10.1016/j.surfcoat.2014.09.019 0257-8972/© 2014 Elsevier B.V. All rights reserved. vapour on stainless steel oxidation has already been described in the literature [3–7]. Douglass et al. [8] stated that the water vapour has a major effect on the oxidation rate of chromia-forming alloys and that this is most marked for iron-based alloys. Generally, the exposure of Fe–Cr alloys to  $O_2/H_2O$  mixtures at temperatures above 500 °C results in a degradation of Cr oxide by the evaporation of Cr species  $CrO_2(OH)_2$  or  $CrO_3$  [9]. The evaporation rate of  $Cr_2O_3$  is linearly dependent on time in  $N_2$ – $O_2$ – $H_2O$  atmospheres, and a higher temperature results in a higher evaporation rate [10]. Chromium evaporation can lead to its depletion from the protective oxide scale [11], resulting in the formation of poorly protective Fe-rich oxide scale and thus an increased oxidation rate. The faster oxidation rates can be attributed to lower Cr/Fe ratios in the scales, and consequently higher solid-state diffusion rates [12].

The long-term oxidation behaviour of some ferritic stainless steels has been intensively studied because of their applications to the solid oxide fuel cell (SOFC) stack [13–15]. However, the operation temperature for these studies was always lower than 1000 °C and this research was not related to the reheating environment. Steel grades B443NT and B445J1M, new ferritic stainless steels developed by Baosteel, China, were found to cause severe sticking during hot rolling. In order to understand the mechanism of surface deterioration and reduce the occurrence of the sticking of stainless steel strips during hot rolling, a fundamental research is necessary to clearly understand the mechanism of oxide scale formation on the strip surface and to develop effective control strategies for optimising the oxide scale structures. Significant benefit will be obtained in the form of product quality improvement and production cost reduction.

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The objective of this study is to investigate the characteristics of oxide scale formed on the surfaces of two stainless steels grades B443NT and B445J1M over the temperature range from 1000 to 1150 °C in a simulated reheating atmosphere with 18% water vapour. Oxidation kinetics as well as the evolution of oxide scale composition and microstructure are systematically studied. The mechanism of the formation of oxide scale on both steels is discussed. The research has practical applications, and the outcomes will be helpful in the design of hot rolling processes of these stainless steels.

#### 2. Experimental method

#### 2.1. Materials

Two ferritic stainless steels, B443NT and B445J1M, were used in this study. The chemical compositions of the steels are given in Table 1. Samples with size of 20 mm  $\times$  10 mm  $\times$  1 mm were machined from a hot-rolled slab with a small hole of 2 mm in diameter near one edge. Prior to the oxidation experiments, all the samples were ground on all sides with 1200 grit SiC sandpaper, then cleaned in acetone and rinsed with alcohol.

#### 2.2. Oxidation experiments

The oxidation kinetics of the stainless steels was investigated using a thermogravimetric analyser (TGA). Oxidation tests were performed in the apparatus shown in Fig. 1. Before the tests, temperature calibration was carried out at the test temperatures. Samples were put in a vertical tube furnace and isothermally heated for 120 min, which is consistent with the real time of reheating. The mass change of a sample during the TGA experiment was measured using a Sartorius CP124S microbalance with a resolution of  $10^{-4}$  g, and the value was recorded automatically by a connected computer. A simulated humid atmosphere with 18% water vapour was generated by bubbling synthetic air through a water bath held at 58.4 °C and then flown into the furnace at a rate of 1000 ml/min. The gas inlet line was heated to prevent water vapour condensation. The total gas pressure was 1 atm.

The furnace was heated to the set temperature at a heating rate of 20 °C/min, then humid air flowed through the 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 and the weight change logged. After the set time of 120 min, the experiment was ended by removing the sample from the furnace and cooling it in air to room temperature.

#### 2.3. Analysis of oxide scale

Surface 3D profiles of the oxide scales formed on the samples were examined by a VHX-1000E Digital Microscope. After surface analysis, all the samples were cold mounted in epoxy resin, then sectioned and polished. The microstructures, compositions and thickness of the oxide scale were examined by a JEOL JSM 6490 scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS). Oxide scales formed on the samples were surface scanned using X-ray diffraction (XRD) for the phase identification.

Table 1	
Chemical compositions of the ferritic stainless steels (wt.%).	

	С	Si	Mn	Р	Cr	Cu	Мо	Ti	Nb
B445J1M	≤0.01	0.30	0.15	0.03	21.50	0.10	0.60	≤0.20	0.12
B443NT	≤0.01	0.35	<0.10	0.00	21.00	0.40	0.00	0.14	0.20



Fig. 1. Schematic diagram of the experimental apparatus.

#### 3. Results

#### 3.1. Oxidation kinetics

Fig. 2a and b presents the variations of mass gain with time for the B443NT and B445J1M stainless steels oxidised from 1000 to 1150 °C in a humid atmosphere with 18% water vapour, respectively. As shown in Fig. 2a, the curves of the B443NT steel present a tendency of parabolic behaviour from 1000 to 1060 °C and the mass gains at 120 min are 1.02, 1.21 and 1.40 mg  $\cdot$  cm<sup>-2</sup>, respectively. At 1090 °C and 1120 °C, the oxidation rates have an abrupt increase indicating



Fig. 2. Mass change versus time curves for the (a) B443NT and (b) B445J1M steels oxidised for 120 min at different temperatures in humid atmosphere containing 18% water vapour.

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