1. Introduction

In hot strip mills using conventional thick slab casting and rolling technology, a thick oxide scale of around 2 mm is formed on the steel surface during reheating. Slabs can be charged to the reheat furnace at different temperatures, cold (from the slab yard) or hot (directly from the continuous caster). The slabs are then reheated to 1250–1260°C for approximately 2 to 3 h. In hot strip mills using thin slab casting and rolling technology, a thin oxide scale of around 200 μm thick is formed in the tunnel furnace. In this type of hot strip mill, tunnel furnaces are the central link between the caster and the rolling mill. They are used to heat and/or equalize the slab temperature in reheating operations of about 12–20 min. In general, tunnel furnaces operate over the temperature range of 1000–1200°C.

During hot rolling of steel strip, oxide scales can generate surface defects. To prevent these defects, hydraulic descaling is necessary to remove oxide scales formed during reheating and ensure the required quality of the final product. There has been considerable work relating hydraulic descaling to oxide scale thickness, steel temperature, steel composition, and scale morphology. However, there is relatively little information available on how the heating atmosphere influences hydraulic descaling. Industrial reheating operations are usually carried out in atmospheres of relatively low oxidizing potential, composed mainly of some free oxygen, carbon dioxide, water vapour, and nitrogen. Therefore, hydraulic descaling studies of steels oxidized in atmospheres composed of O2–CO2–H2O–N2 are of great interest to better understand hydraulic descaling. In the present work, the effect of O2 in heating atmosphere on hydraulic descaling of low carbon steel was analyzed, and results were discussed in relation to the scale morphology.

2. Experimental Procedure

Low carbon steel specimens were obtained with the following chemical composition: 0.06% of C, 0.27% of Mn, 0.005% of Si, and 0.006% of Cu (all in wt%). Before oxidation, the specimens were polished with SiC paper and cleaned with alcohol. Previous experiments showed that the oxidation rate increases with an increase of gas velocity until a critical gas velocity is reached. The critical gas velocity for air is 4.2 cm/s, (5 ft/min) 2.54 cm/s for carbon dioxide, and (23 ft/min) 11.68 cm/s for steam. Therefore, in the present work, a gas velocity of 11.7 cm/s was selected to ensure high oxidizing conditions.

A tubular horizontal furnace was used to heat the specimens (50 mm×50 mm×13 mm) in an argon atmosphere to 1050, 1100, 1150, 1200 and 1250°C. Then, the inert gas was replaced by the selected oxidizing gas mixture. The specimens were isothermally oxidized for 15, 30, 60,
and 120 min in $\text{O}_2$–$15\text{CO}_2$–$10\text{H}_2\text{O}$–$\text{N}_2$ and $5\text{O}_2$–$15\text{CO}_2$–$10\text{H}_2\text{O}$–$\text{N}_2$ gas mixtures.

After taking the specimen out of the furnace, the specimen was placed in a rotary disc and accelerated to an angular velocity of 110 rpm (equivalent to a linear velocity of 1.11 m/s), Fig. 1. The oxide film was removed using a high-pressure water jet with a descaling angle of 15°, spray width of 7 mm (at the sample surface), water pressure of 11.37 MPa and flow rate of 6.8 L/min. Immediately after the hydraulic descaling test, the specimen was placed into a steel tube with a flow of argon and cooled to room temperature. Fast cooling in an argon atmosphere was used to avoid further oxidation and minimize the decomposition of wustite. After cooling, scale/steel cross-sections were cut from the slab and mounted for metallographic analyses. In addition to the specimens for the hydraulic descaling test, extra specimens (15 mm × 30 mm × 13 mm) were oxidized for scale morphology observations.

An optical microscope equipped with an image analyzer was used to determine the porosity of the scale by measuring the pore areas and subtracting them from the total scale area. Before the hydraulic descaling test, the optical microscope was used to measure the scale thickness and inner porous layer thickness, Fig. 2. After the hydraulic descaling test, the thickness of the residual scale was measured. All the measurements were carried out at ten different points.

3. Results and Discussion

3.1. Scale Morphology

In the present work, two types of scale were observed: crystalline scale and classical three-layer scale, Fig. 3 and Table 1. These results correspond with previous works reported by several researchers and the current authors, who have observed that in the oxidation of steel in weakly oxidizing atmospheres, scales have a crystalline structure or the classical three-layered formation.

The mechanisms of formation of crystalline scale and three-layer scale are well understood and documented. Crystalline scale has an irregular outer surface (Fig. 3(a)). It is formed during the initial oxidation in weakly oxidizing atmospheres, when the rate of incorporation of oxygen into the scale is slower than the rate of arrival of iron by outward diffusion through the scale. In this situation, when a phase-boundary reaction is rate-controlling, the adsorption of oxygen atoms or absorption of ions would occur faster at preferred planes of the oxide crystal, leading to idiomorphic crystals at the surface and a scale with an irregular outer surface. Crystalline scale is composed mostly of wustite and a negligible amount of magnetite. The three-layered formation is deposited at high temperatures on the steel substrate when the diffusion of iron is the slowest step, and therefore the rate-controlling mechanism. The innermost layer is wustite, the intermediate layer is magnetite, and the outer layer is hematite.

In the $\text{O}_2$–$15\text{CO}_2$–$10\text{H}_2\text{O}$–$\text{N}_2$ gas mixture, a transition from crystalline scale to three-layer scale was observed at 1150, 1200, and 1250°C, Table 1. As has been shown in previous studies, this transition occurs when the oxidation time is prolonged, increasing the thickness of the scale. The thicker scale reduces the diffusion of iron, changing the rate-controlling mechanism from the surface reaction (crystalline scale) to the diffusion of iron (classical three-layer scale).

The scales were porous, particularly close to the metal.
substrate, forming an inner porous layer between the metallic core and dense scale, Fig. 3. These results correspond to the work of several researchers that observed a concentration of pores close to the metal substrate in the oxidation of steel in laboratory conditions simulating industrial furnaces, and in industrial furnaces. The scales were denser close to the gas phase. This dense scale has been attributed to its formation exclusively by outward diffusion of iron.

It seems that there are two major reasons for the development of porosity: idiomorphic growth and growth stress. Sheasby et al. reported that in the initial oxidation of Fe–C alloys in weakly oxidizing atmospheres, pores are formed when some favored grains overgrow others as larger idiomorphic crystals, leaving small pores trapped at the crystal junctions. They also observed that in oxidizing gas mixtures containing water vapour, the pores migrated from the scale/steel interface to the wustite layer. This pore migration has been attributed to oxidation and reduction reactions that take place in opposite sides of pores filled with H2O–H2 or CO2–CO.

The major source of growth stress in scales is the stress originated by a change in volume as the metal is converted into oxide. The ratio of the volume per metal ion in the oxide to the volume per metal atom in the metal has commonly been termed PBR, the Pilling–Bedworth ratio. Compressive stress is developed in the oxide when PBR > 1 and tensile stress when PBR < 1. The Pilling–Bedworth ratio for iron oxides is PBR > 1. Therefore, during oxide growth, compressive stress is present in the oxide scale. The model proposed by Kofstad explains the formation of pores in scales by growth stress. In this model, growth stress in the scale increases with time and is relieved by plastic deformation, forming pores at the grain boundaries.

The scale/steel cross-sections revealed that pores were continuously formed at the grain boundaries (see Fig. 4) during the 120 min of oxidation, time at which the investigations ceased. Pore formation by idiomorphic growth takes place in the initial minutes of oxidation. This suggests that the pores observed in the present work were formed mainly by growth stress in agreement with the model proposed by Kofstad.

Table 1. Mode of scale removal and scale morphology for the different temperature, gas mixture and oxidation time.

| Temp. (°C) | Gas mixture | Time = 15 min | Time = 30 min | Time = 60 min | Time = 120 min |
|-----------|-------------|---------------|---------------|---------------|---------------|
| 1050      | O2–15CO2–10H2O–N2 | T | L | T | M | 2 | T | H | 2 | T | H |
| 1100      | O2–15CO2–10H2O–N2 | T | M | 1 | T | M | 2 | T | H | 2 | T | H |
| 1150      | O2–15CO2–10H2O–N2 | T | M | 1 | T | M | 2 | T | H | 2 | T | H |
| 1200      | O2–15CO2–10H2O–N2 | T | H | 2 | C | H | 2 | C | H | 2 | T | E |
| 1250      | O2–15CO2–10H2O–N2 | T | H | 2 | T | H | 2 | T | H | 2 | T | E |
| 1300      | O2–15CO2–10H2O–N2 | T | H | 2 | T | H | 2 | T | H | 2 | T | E |

Scale structure: T = three-layer scale; C = crystalline scale. Scale porosity: L = low (porosity ≤ 5%); M = medium (5% < porosity ≤ 10%); H = high (10% < porosity ≤ 20%); E = extra high (20% < porosity ≤ 30%).

In the oxidation of iron, porous scales are formed during prolonged oxidation at 850°C and during progressively shorter times at higher temperatures. The results of this study showed the same tendency; the porosity increased with an increase in oxidation time and/or temperature, Table 1.

3.2. Modes of Scale Removal

In all the hydraulic descaling tests, the scale was removed in the jet impact zone, where the thickness of the residual scale was measured, Fig. 5. Sheppard and Steen observed that during hydraulic descaling of steel oxidized in air, the removal of scale occurred when thermal and mechanical impacts of the water jet propagated a horizontal undercutting (crack) at the scale/metal interface. The position of the horizontal undercutting determines the thickness.
of the residual scale. In the present work, the position of the horizontal undercutting was controlled by the scale morphology. During the hydraulic descaling tests, two modes of scale removal were observed.

3.2.1. Mode I

In mode I, the horizontal undercutting occurred at the boundary of the inner porous layer and dense scale, Figs. 6 and 8. Mode I was observed in three-layer scales that developed a solid inner porous layer with low or medium porosity that resisted the impact of the water jet, Table 1.

Previous works on the oxidation of low carbon steel and steels with various Si content in $\text{O}_2$–$\text{CO}_2$–$\text{H}_2$–$\text{N}_2$ atmospheres showed that a rise in free oxygen results in higher oxidation rates. In the present work, the rate of oxidation was followed by measuring the scale thickness. A rise in free oxygen from 1 to 5% increased the oxidation rates as reported in previous studies (Fig. 7(a)). In addition, it was observed that the thickness of the inner porous layer increased with increasing free oxygen, oxidation time and/or temperature (Fig. 7(b)).

In mode I, the thickness of the inner porous layer determined the thickness of the residual scale. Therefore, an increase in the inner porous layer thickness with increasing free oxygen, oxidation time, and/or temperature, resulted in a thicker residual scale. This point will be taken up in more detail later.

3.2.2. Mode II

In mode II, the horizontal undercutting occurred at the first plane of large pores relative to the scale/steel interface, Figs. 9 and 12. Mode II was observed in three-layer scales that developed an inner porous layer with high porosity, and in crystalline scales, Table 1. In the hydraulic descaling of three-layer scales with high porosity, the inner porous layer was weak and yielded to the impact of the water jet, resulting in a horizontal undercutting at the first plane of large pores relative to the scale/steel interface (mode II).

Hematite and magnetite form rigid layers in three-layer scales. The irregular outer surface and the lack of the rigid layers in crystalline scales, which are mostly formed by wustite (the softest of the iron oxides), resulted in a deep impact of the water jet. Consequently, in the hydraulic descaling of crystalline scales, the horizontal undercutting always occurred deep in the scale in mode II, independently
of the state of porosity.

In mode II, the proximity of the first plane of large pores to the scale/steel interface determined the thickness of the residual scale in both three-layer scales with high porosity and crystalline scales. Therefore, in mode II, a close first plane of large pores to the scale/steel interface resulted in a thin residual scale, while a distant first plane of large pores from the scale/steel interface resulted in a thick residual scale.

3.3. Removability of Scale

Figure 11 illustrates the mean thickness of the residual scale, standard deviation, and mode of scale removal. For the hydraulic descaling test, the low carbon steel was oxidized for 15, 30, 60, and 120 min over the temperature range 1050–1250°C in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures.

![Fig. 10. Cross-section of three-layer scale after hydraulic descaling in mode II. The three-layer scale was formed in SO2–15CO2–10H2O–N2 for 60 min at 1250°C. Outside of the jet impact zone some dense scale was removed together with the scale of the jet impact zone.](image)

![Fig. 11. Mean thickness of the residual scale, standard deviation, and mode of scale removal as a function of temperature, free oxygen, and oxidation time. The low carbon steel was oxidized for 120 min in 1O2–15CO2–10H2O–N2 at (a) 1050°C, (c) 1100°C, (e) 1150°C, (g) 1200°C, (i) 1250°C, and in 5O2–15CO2–10H2O–N2 at (b) 1050°C, (d) 1100°C, (f) 1150°C, (h) 1200°C, (j) 1250°C.](image)
porous oxide at the scale/steel interface. Therefore, after the impact of the water jet and yielded at the weak and the inner porous layer with high porosity could not resist increased from medium to high porosity, Table 1. As a result, during the initial 30 min of oxidation in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures, the porosity in-

Figures 11(a) and 11(b) reveal the results for the low carbon steel oxidized at 1 050°C in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures respectively. During the initial 30 min of oxidation in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures, the horizontal undercutting occurred in mode I. Therefore, thicker residual scales were observed when the free oxygen was increased from 1 to 5%, the oxidation time from 15 to 30 min and/or the temperature from 1 050 to 1 100°C (Figs. 11(a)–11(d)).

Similar transitions to the ones observed at 1 050°C occurred after 30 min of oxidation when the porosity increased, changing the mode of scale removal from mode I to mode II, Table 1. As a result of the transition from mode I to mode II, the position of the horizontal undercutting became controlled by the proximity of the first plane of large pores to the scale/steel interface. After 120 min of oxidation, the proximity of the first plane of large pores to the scale/steel interface was similar in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures, resulting in small differences in the residual scale thickness.

Figures 11(e) and 11(f) illustrate the results at 1 150°C in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures respectively.

In the 1O2–15CO2–10H2O–N2 gas mixture, crystalline scales formed during the initial 60 min of oxidation, and three-layer scales with high porosity formed after 60 min of oxidation had the horizontal undercutting at the first plane of large pores relative to the scale/steel interface (mode II), Table 1. During the initial 30 min of oxidation, the hydraulic descaling in mode II resulted in a thinner residual scale in the 1O2–15CO2–10H2O–N2 gas mixture; the horizontal undercutting took place closer to the scale/steel interface than in the scales grown in the 5O2–15CO2–10H2O–N2 gas mixture, which were removed farther away from the scale/steel interface in mode I. Figure 12(c) shows a typical crystalline scale removed in mode II.

In the 5O2–15CO2–10H2O–N2 gas mixture, mode I was observed during the initial 30 min of oxidation; therefore, a rise in oxidation time from 15 to 30 min increased the thickness of the residual scale. In addition, a rise in temperature from 1 050 to 1 100°C increased the thickness of the inner porous layers (see Fig. 7(b)), which in turn resulted in thicker residual scales in the hydraulic descaling in mode I (Figs. 11(a)–11(d) and 11(f)). As in the experiments at 1 050 and 1 100°C, an increase of porosity from medium to high resulted in a transition from mode I to mode II after 30 min of oxidation, Table 1.

After 120 min of oxidation, pore migration was observed

After 30 min of oxidation in 1O2–15CO2–10H2O–N2 and 5O2–15CO2–10H2O–N2 gas mixtures, the porosity increased from medium to high porosity, Table 1. As a result, the inner porous layer with high porosity could not resist the impact of the water jet and yielded at the weak and porous oxide at the scale/steel interface. Therefore, after

Fig. 12. Cross-sections of scales and their residual scales after hydraulic descaling in mode II. The pores (arrowed) of the first plane of large pores relative to the scale/steel interface are indicated in the middle column. The scales were formed in (a) 1O2–15CO2–10H2O–N2 for 60 min at 1 050°C, three-layer scale, (b) 1O2–15CO2–10H2O–N2 for 120 min at 1 050°C, three-layer scale, (c) 1O2–15CO2–10H2O–N2 for 30 min at 1 150°C, crystalline scale, (d) 1O2–15CO2–10H2O–N2 for 120 min at 1 150°C, three-layer scale, (e) 1O2–15CO2–10H2O–N2 for 60 min at 1 250°C, crystalline scale, (f) 5O2–15CO2–10H2O–N2 for 30 min at 1 250°C, three-layer scale.
in the scales formed in low \(1\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) and high \(5\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) oxygen content gas mixtures. The migration of pores from the scale/steel interface to the wustite layer is counteracted in the opposite direction by creep of the oxide which accompanies scale growth.\(^{13}\) In general, creep increases with temperature; therefore, it is reasonable to assume that a rise in temperature from 1050 and 1100 to 1150°C increased creep in wustite, facilitating the migration of pores into the wustite layer. Pore migration after 120 min of oxidation at 1150°C shifted the horizontal undercutting in mode II farther away from the scale/steel interface, resulting in thicker residual scales. In the migration process, pores usually acquire a cap shape with the convex surface in the direction of scale growth.\(^{13}\) Figure 12(d) shows a typical scale with migrated pores (cap shape).

Figures 11(g) and 11(h) show the results at 1200°C in \(1\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) and \(5\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) gas mixtures respectively. In the oxidation of steel at high temperatures, pore coalescence forms large pores in thick scales.\(^{26}\) At 1200 and 1250°C, pore coalescence formed large pores in the scales. The high porosity and pore coalescence masked the migrated pores in the wustite layer. The pores were large and distributed across the inner porous layer. Figures 10, 12(e) and 12(f) show typical scales with large pores formed by coalescence. The inner porous layers with high porosity could not resist the impact of the water jet and were removed. Therefore, mode I was absent in all the experiments at 1200 and 1250°C. The extremely weak oxide close to the scale/steel interface was the potential location for the horizontal undercutting at 1200°C and 1250°C. Consequently, the horizontal undercutting occurred at the first plane of large pores relative to the scale/steel interface (mode II). In general, at 1200 and 1250°C the concentration of free oxygen in the gas mixture had little effect on the proximity of the first plane of large pores to the scale/steel interface. As a result, the differences in the residual scale thickness with increasing free oxygen were small at 1200°C.

Figures 11(i) and 11(j) reveal the results at 1250°C in \(1\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) and \(5\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) gas mixtures respectively. As mentioned above, at 1250°C, the concentration of free oxygen in the gas mixture had little effect on the proximity of the first plane of large pores to the scale/steel interface, resulting in residual scales with similar thickness in the \(1\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) and \(5\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) gas mixtures.

4. Conclusions

The effect of \(\text{O}_2\) in heating atmosphere on hydraulic descaling of low carbon steel oxidized in conditions similar to those used in reheat furnaces was investigated, and the results are summarized as follows:

(1) Two modes of scale removal have been observed. In mode I, observed in classical three-layer scale that developed an inner porous layer with low or medium porosity, the horizontal undercutting occurs at the boundary of the inner porous layer and dense scale. In mode II, observed in classical three-layer scale, which developed an inner porous layer with high porosity, and in crystalline scales, the horizontal undercutting occurs at the first plane of large pores relative to the scale/steel interface.

(2) The concentration of free oxygen in the heating atmosphere influences the removability of scale. At 1050°C, a rise in the concentration of free oxygen results in an increase in thickness of the residual scale. At 1100°C and 1150°C, the increase in residual scale thickness with a rise in free oxygen is significant in the initial 60 min of oxidation; after this period of time the differences in the thickness of the residual scale become smaller. At 1200°C, the influence of the concentration of free oxygen on the thickness of the residual scale is small, and at 1250°C, it is negligible.

(3) The oxidation time and temperature influence the removability of scale. During the initial 30 min of oxidation, an increase in the oxidation time and/or temperature in the formation of three-layer scales in the temperature range 1050–1150°C results in thicker residual scales. After 30 min of oxidation, an increase in the oxidation time causes a transition from mode I to mode II in three-layer scales formed in the temperature range 1050–1150°C. At 1200°C and 1250°C, the high temperatures lead to the formation of scales with high porosity that are removed in mode II.

(4) The phase composition of the scale influences the mode of scale removal. Crystalline scales mostly composed of soft wustite always have the horizontal undercutting at the first plane of large pores relative to the scale/steel interface (mode II), independently of the state of porosity.

(5) Typical delivery temperatures of a tunnel furnace are in the range of 1050–1150°C.\(^{27}\) The experiments showed that during the initial 30 min of oxidation at 1050, 1100 and 1150°C, scales formed in the low oxygen content \(1\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) gas mixture had thinner residual scales than scales formed in high oxygen content \(5\text{O}_2-15\text{CO}_2-10\text{H}_2\text{O}-\text{N}_2\) gas mixture. This suggests that reducing the concentration of free oxygen in the tunnel furnace atmosphere is important for increasing scale removability.

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