The pickling ability and adhesion edge scale of hot-rolled carbon steel produced using different entry temperatures

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Abstract

The objective of this work was to investigate the pickling ability and the strain provoking the edge scale on hot-rolled carbon steel. The pickling test was conducted by immersing the steel samples in 10% v/v HCl solution both with and without industrial inhibitor at 80 °C. It was found that the addition of the inhibitor retarded the dissolution rate of the oxide scale, and this effect is more pronounced when the entry temperature was reduced from 1011 °C to 990 °C. However, in the solution without the inhibitor, the dissolution rates of the oxides formed using different entry temperatures were identical. From the previous work on scale adhesion assessment, the tensile test was applied to evaluate the scale of adherence. As a result, the derivative of the spallation ratio as a function of the imposed strain was calculated in order to determine the strain provoking the maximum spallation. The values were 3.78 ± 0.13% and 4.2 ± 0.28% for the hot-rolled steel produced using the entry temperatures of 1011 °C and 990 °C, respectively. The results showed that the entry temperature did not affect the edge scale adherence and pickling ability.

Keywords: oxide scale, hot-rolled steel, pickling, adherence

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Introduction

The pickling ability and adhesion edge scale were influenced by hot-rolling process parameters. At the initial step, a slab with thickness of 250 millimeter was heated at a temperature range from 1200 °C to 1250 °C in reheating furnace. At this high temperature, the surface of the slab is oxidized. The reaction between metal and atmosphere at this temperatures in the hot-rolling process results in a thermal oxide scale covering its surface, called scale (Kofstad, 1988). This scale could be removed by using a high-pressure water jet before sending the slab to the roughing mill for first step thickness reduction (transfer bar). In the next step, the scale was sent to the finishing mill to finally reduce its size to the required thickness. Before the transfer bar was rolled in this step, the oxide scale was formed again and also removed by the same method as in the previous step. The temperature before entering the finishing mill was called *entry temperature*; ET which could be in the range of 900-1100 °C. After it passed the finishing mill, it was called *finishing temperature*; FT which could be in the range of 820-910 °C and it was moved to pass through the laminar flows at the cooling bed and then coiled at the down coiler. This temperature was called *coiling temperature;* CT which could be in the range of 540-720 °C (Chen, & Yuen, 2001). There were three important parameters that could influence the structure and properties of the oxide scale (Figure 1).

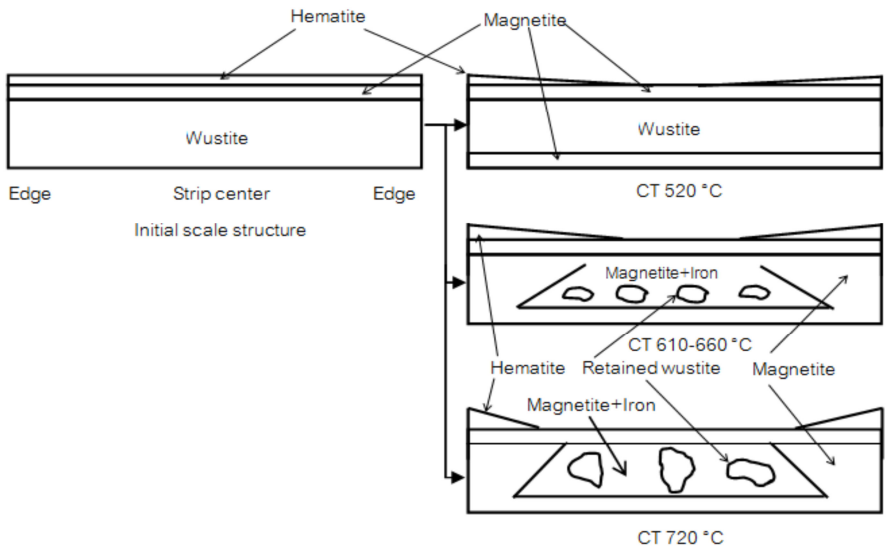


Figure 1 Evolution of the oxide scale on hot-rolled steel strips coiled at different temperatures. (Chen, & Yuen, 2001)

Crossection along the width of strip revealed the structure of oxide scale which consist of wustite (FeO) magnetite (Fe_3O_4) and hematite (Fe_2O_3). The initail scale structure was wustite, easily pickled in 10% v/v HCl solution at temperature of 80 ^{o}C and good adherence (high adhesion energy). The strip was moved to pass through the finishing mill and coiling. The wustite phase was tranfromed to be magnetite at the middle part and hematite at both the edges and the top surface because of the interstitial of oxygen in the coil yard. Scale thickness of distance from both edge regions to 50 millimeter was always higher than the one at the middle area. Quantification of mechanical adhesion energy of oxide scales of different thicknesses isothermally grown at 900 ^{o}C on AISI 441 ferritic stainless steel was calculated with the Galerie-Dupeux model, increasing of scale thickness resulted to low mechanical adhesion (Chandra-ambhorn, Roussel-Dherbey, Toscan, Wouters, Galerie, & Dupeux, 2007).

The effect of entry temperature on the scale adhesion at the middle part of the strip, Fe-0.148C-0.425Mn-0.019Si produced using the entry temperature of 990 ^{o}C and Fe-0.152C-0.453 Mn-0.02Si produced using the entry temperature of 1011 ^{o}C were studied (Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012; Ngamkham, 2014) in previous works. It was found that the scale consisted of hematite and magnetite with the thicknesses of 7.14 μm and 8.26 μm for the steel was produced using the entry temperatures of 1011 and 990 ^{o}C respectively. However, it was often found in practice that the surface problem of the hot-rolled steel was at the edge area of the strip. Therefore, the objective of this work was firstly to investigate the pickling behaviour of the scale at the edge area of the hot-rolled steel. Furthermore, our previous work has developed the tensile test to assess the scale adhesion and applied it to evaluate the adhesion of the scale to the hot-rolled steel produced using different entry temperatures. However, the quantification of the adhesion energy was not treated in our previous paper (Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012). The present work thus aimed at advancing such quantification, followed by the discussion of the relationship between pickling ability and scale adhesion.

Methodology

The same steel grades descibed in the previous paragraph was used. The sample with 50 mm from the edge of the strip was taken for the study. In the pickling test, the sample was cut into 20×25×3.2 mm dimension. In step one, the sample was cleaned in acetone and dried in forced air. The sample was then weighed before pickling, recorded in (Table 1) and immersed

for 5 seconds in 10% v/v HCl solution both with and without 10% v/v industrial inhibitor at 80 ^{o}C. The sample was cleaned with acetone acid and then dried using a blower, weighed after pickling and recorded in (Table 1). Weight loss (μg/mm^2) after the first time was calculated as shown in (Table 1). In step two, the same sample had to undergo step one again for 10 times using different periods of time up to 45 sec. The standard deviation of Weight loss (μg/mm^2) as a function of time (sec) from three to five samples in the (Figure 2), exemplifies the pickling test. From this figure, it can be seen that the curve consisted of two zones. The zone in the early period corresponds mainly to the dissolution rate of the oxide (slope line), while the zone in the latter period corresponds to the corrosion of metal substrate (horizontal line). The dissolution rate of the oxide scale was a slope of the curve in the early period. In order to find out the time for complete pickling, the tangent line of the curve in each zone was drawn. The time at the intersection between these lines, that is the slope line and the horizontal line in the early and latter period, is the time for complete pickling.

Table 1 Data of pickling test of the oxide scale.

number of immerse (times)	pickling time (sec) X-axial	(1) weight sample before pickling (μg)	(2) weight sample after pickling (μg)	weight loss (1)-(2) (μg)	(3) surface area (20*25*2) (mm^2)	weight loss [(1)-(2)/(3)] (μg/mm^2) Y- axial
1 to 10	5	3.50	3.40	0.09	1000	0.00

To study the behavior of mechanical adhesion of the oxide scale on hot-rolled low carbon steel at the edge of strip, the samples for tensile testing were prepared according to ASTM E8M. The surface of the sample was cleaned with ethanol and marked at the middle with red ink to help detect the oxide failure during straining. The sample was placed in the tensile machine (600 kN force type) (Ngamkham, 2014).

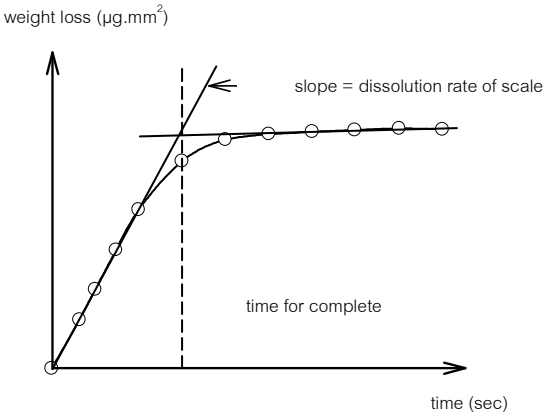


Figure 2 Example of the pickling result.

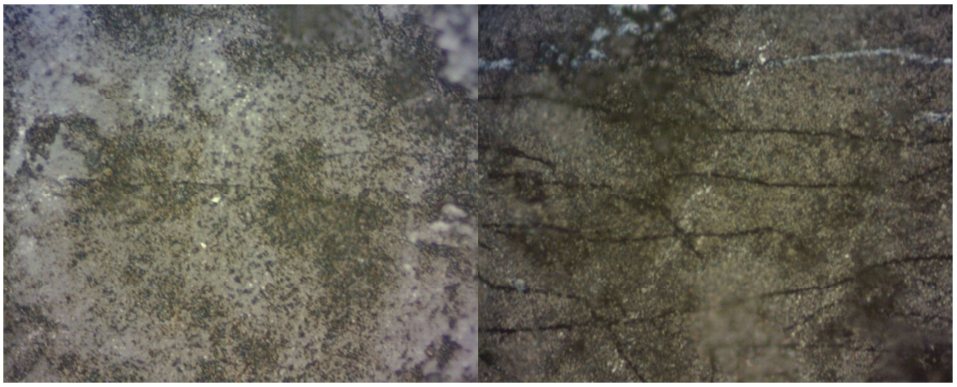
Results and discussion

(Figure 3) shows the surface of the oxide scale before pickling and after pickling for 15 sec. The mass loss of each sample was shown in (Figure 4). From this figure, we can determin the dissolution rate of the oxide scale as shown in (Figure 5), and also the time for complete pickling as shown in (Figure 6). From these results, it can be seen that when the inhibitor was added in the solution, the dissolution rate of the oxide would be lower than that of the oxide immersed in the solution without the inhibitor. This result showed the active role of the inhibitor in reducing corrosion rate. Furthermore, for the sample immersed in the solution with inhibitor, lowering the entry temperature from 1011 to 990 ^{o}C also reduced the dissolution rate of the oxide from 2.17 to 1.64 $\mu g.mm^{-2}.s^{-1}$. This indicated the lower effeciency of the inhibitor to dissolve the scale formed by lower entry temperature.

As for time to complete pickling, it was found that the higher the entry temperature, the longer the time to complete picklin. This should be from the thicker scale on the steel produced using higher entry temperature. As described in the Introductory part, our previous work found that the scale was thicker with the increased entry temperature, i.e. 7.14 and 8.26 μm for the steel was produced using the entry temperatures of 1011 and 990 ^{o}C respectively (Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012; Ngamkham, 2014). The increased scale thickness with the increased temperature was because increasing the oxidising temperature increased the diffusion of point defects responsible for the growth of the oxide, thus giving the faster oxidation rate and thicker scale at a given time (Kofstag, 1988).

Comparing the literature (Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012), it was found that the dissolution rate of the edge scale immersed in the solution without and with inhibitor was faster than that of scale at the middle part of the strip. For example, the dissolution rates were 2.11 $\mu g.mm^{-2}.s^{-1}$ (Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012) and 3.10 $\mu g.mm^{-2}.s^{-1}$ for the edge and centre scale of the steel immersed in the solution without inhibitor, respectively. On the other hand, the dissolution rates were 2.11 $\mu g.mm^{-2}.s^{-1}$ (Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012) and 3.10 $\mu g.mm^{-2}.s^{-1}$ respectively, for the edge and center scale of the steel immersed in the solution with inhibitor. As a matter of discussion, it was reported that the edge scale tended to have the higher ratio of hematite to the total oxide scale (Chen, & Yuen, 2003). Since hematite is an oxide phase which is relatively hard to be pickled, the increased ratio of hematite to the total oxide could thus

retard the oxide dissolution rate as found in this comparison (Chattophadyay, & Chanda, 2008). However, the present result showed otherwise, i.e. edge scale have higher pickling ability. The present result indicated that the pickling ability could be affected not only by the types of the oxide phase but also by other factors such as the morphology of the oxide. It seemed that the edge scale in the present work might have the oxide morphology which was vulnerable to be pickled than the centre scale. However, this assumption needed to be proved in the future work.



(a) (b)

Figure 3 Surface of the oxide scale before pickling (a) and after pickling for 15 sec (b).

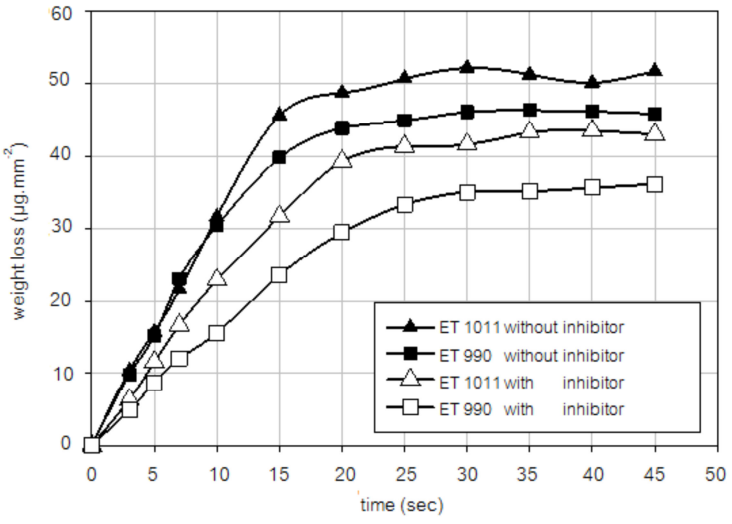


Figure 4 Pickling results of the studied steels.

For further discussion on the relationship between the pickling ability and scale adhesion, the tensile test has been applied to evaluate the scale adhesion (Chandra-ambhorn, Roussel-Dherbey, Toscan, Wouters, Galerie, & Dupeux, 2007; Ngamkham, Klubvihok, Tungtrongpairoj, & Chandra-ambhorn, 2012; Chandra-ambhorn, & Klubvohik, 2016). In the conventional method, the strain initiating the first spallation was measured to represent the degree of spallation. It was reported (Chandra-ambhorn, 2006) that we can also use the strain that provokes the maximum spallation to assess the scale adhesion. For scale at the center of the strip, the spallation ratios as a function of strain of the hot-rolled steels produced using the entry temperatures of 990 and 1011 °C were reported (Ngamkham, 2014). In this work, we proposed to produce the derivatives of those spallation ratios with respect to the imposed strain, giving the results in (Figure 7). From this figure, we can see that, for each sample, the spallation tended to increase up to the maximum ratio and then reduce to the lower one. From this curve, we define the strain at the maximum scale spallation as the strain provoking the maximum spallation. From this figure, we can see that increasing the entry temperature did not considerably change the strain provoking the maximum spallation. These critical strains were 4.2 ± 0.28 and 3.78 ± 0.13 % for the samples produced using the entry temperature of 990 and 1011 °C, respectively.

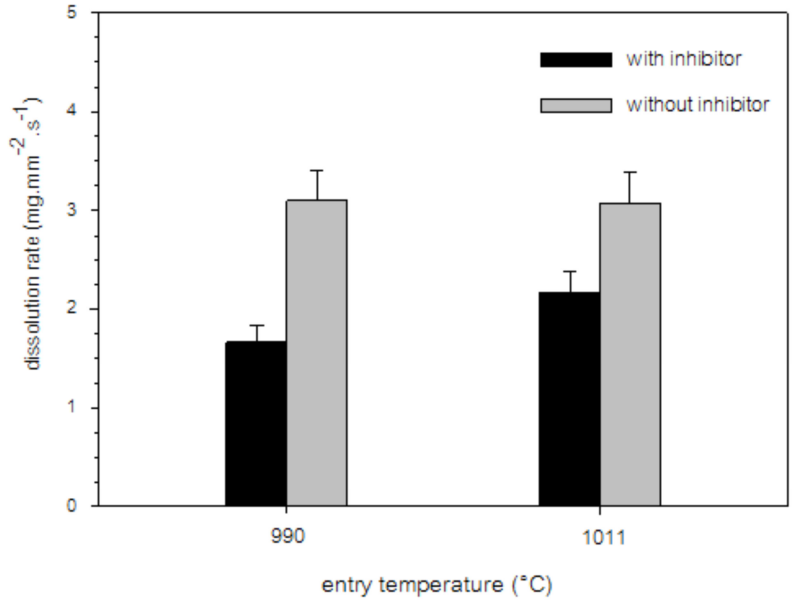


Figure 5 Dissolution rate of the oxide scale.

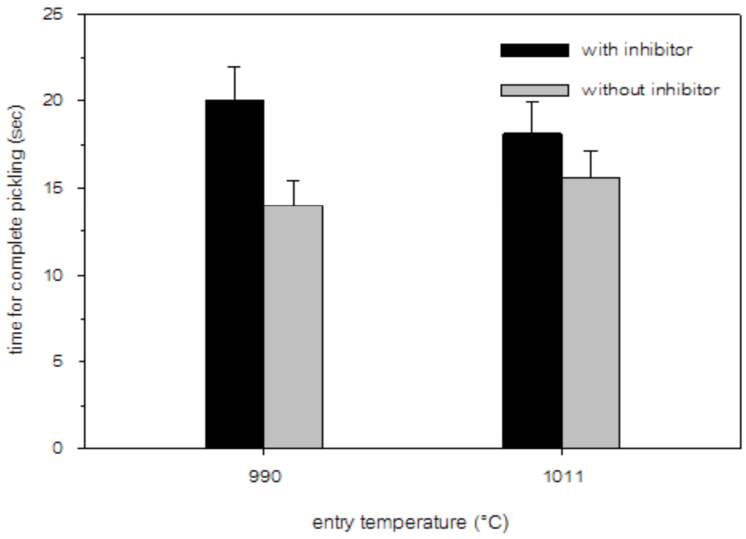


Figure 6 Time to complete pickling of the studied hot-rolled steels.

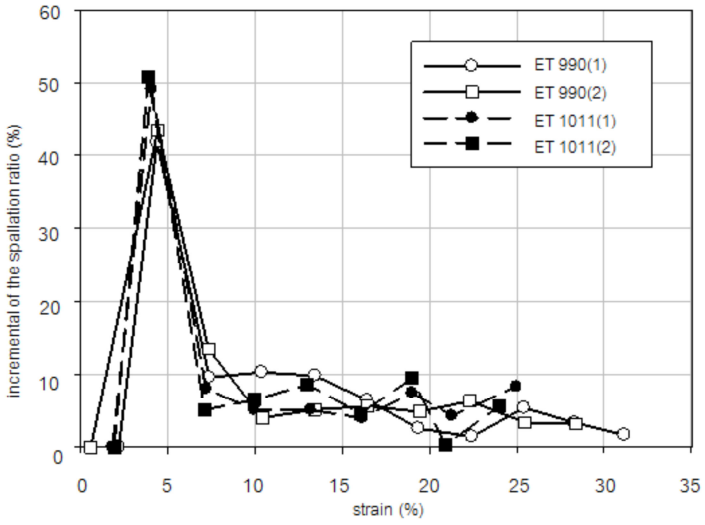


Figure 7 Increment of scale spallation ratio as a function of the imposed strain plotted using data from Ngamkham (2014).

Conclusion

(1) For the edge scale pickling, the addition of 10% v/v industrial inhibitor to the 10% v/v HCl solution at 80 $^{\circ}$C retarded the scale dissolution. In the solution with inhibitor, the dissolution rate of the scale formed on the steel

produced using the entry of 990 °C, was lower than that of the scale formed on the steel produced using the entry temperature of 1011 °C.

(2) For the adhesion test using the tensile method, the strain provoking the maximum spallation ratio was determined for the studied hot-rolled steels. The adhesion energies at this critical strain were calculated using Galerie-Dupeux model. These energies were 137 ± 0.64 and 110.86 ± 2.48 $J.m^{-2}$ for the hot-rolled steel produced using 990 and 1011 °C, respectively.

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