Adhesive Strength of Oxide Scale Formed on Low-Carbon Steel

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The scale formed on the surface of steel during hot rolling causes surface defects. Thus, controlling scale adhesion is important; however, there have been few reports on quantitative investigations of scale adhesion at high temperature. In this study, a method of measuring scale adhesive strength at high temperature is investigated. The effect of the external stress on the oxide scale is also investigated. The adhesive strength is on the order of 1 MPa. The adhesive strength increases proportionally as axial stress applies on the scale. The scale/steel interface analysis shows that there are many voids at the interface after the oxidation, however, the number of voids decreases and a rough interface is formed after axial compression stress is applied on. It is found that the adhesive strength of the scale depends on the contact area and the rough interface between the scale and the steel.

Key words: rolling, steel, scale, adhesive strength, measurement, axial compression, interface.

1. Introduction

In steel production, the surface of steel is oxidized and scale forms on it at high temperature; this oxide scale may cause surface defects. Typically, scale is removed by high pressure water before hot rolling because the roll may push a thick scale into the steel surface. Surface defects may be caused by the spontaneous detachment of blistered scale; blistering occurs when oxide scale swells during oxidation. Thus, the adhesion property of scale at high temperature is an important characteristic, and understanding and controlling scale adhesion properties would contribute to the quality of steel produced.

Some methods of measuring scale adhesive strength qualitatively have been reported. These methods can evaluate the amount of detached scale or the detaching behavior when compressive¹⁾ or tensile^{2), 3)} force applies on the sample material. A method of measuring such force when scale has been mechanically detached after a bolt is buried in scale has been reported.⁴⁾ However, it is difficult to use the method to evaluate adhesion quantitatively in the case of a thin oxide scale (tens of micrometers in thickness). A method of obtaining adhesive energy by in situ observation when a tensile test is conducted on an oxidized steel sample has been reported.⁵⁾ It is possible to apply this method at high temperature. Kushida et al.⁶⁾ proposed a method of estimating scale exfoliation stress. After generating scale on two pillar specimens at high temperature, the scales formed on the two samples were joined by pressurizing at high temperature. Next, by the tensile test of the joined specimen, the force and stroke curve was obtained. The scale exfoliation stress is estimated from the curve. The exfoliation stress between a

scale and a material was affected by the joining stress.⁶⁾

The authors improved the method of Kushida et al.⁶⁾ Scale merging is performed by oxidation with small compression stress rather than with high compression stress.⁷⁾ Scale adhesive strength was measured with low compressive stress at high temperature quantitatively⁷⁾. Here, a detailed study of the effect of external axial strength on the scale adhesion is conducted.

2. Experimental

Steel materials with the chemical components shown in **Table 1** were used for the experiment. Cylindrical samples with a diameter of 10 mm were used. An additional cylindrical specimen made of electrolytic iron was used for the sample with which the scaled sample is merged. This is why the iron has higher scale adhesive strength than the steel used for the experiment.

Table 1 Chemical compositions of the samples (mass%)

	C	Si	Mn	P	S	Al
A	0.094	0.028	0.45	0.004	0.003	0.016
B	0.092	0.014	0.43	0.004	0.003	0.014

Tensile test equipment with a furnace was used for the experiments. The atmosphere in the furnace could be controlled. The experimental method used to measure scale adhesive strength is shown in **Fig. 1**. Two cylindrical samples were placed 10 mm apart at room temperature. The electrolytic iron sample was placed on the top and the sample used to measure scale adhesive strength was placed at the bottom. The samples were heated up to 950 °C and oxidized for 360 s. Oxide scales formed on both samples. Then, the electrolytic iron sample was moved downward and positioned on the lower sample with constant compressive stress in the axial direction. The scale formed on

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the electrolytic iron sample merged with the scale formed on the lower sample. The axial compressive stress was varied from 6.4 kPa to 12.7 MPa during the scale merging. After the scales merged, the atmosphere was changed from oxidizing nitrogen to non-oxidizing nitrogen. Then, the electrolytic iron sample was pulled up and the load was recorded during scale detachment from the lower sample. The scale merging and separation processes were conducted at the oxidation temperature (950 °C). The maximum stress was considered to be the scale adhesive strength when the scale on the lower sample was completely detached.

Fig. 1 Method of measuring scale adhesive strength

3. Results

A photograph of typical samples after the separation for adhesive strength measurement is shown in Fig. 2. The scale of the lower sample is completely detached and the metal surface appears [Fig. 2 (b)]. Fig. 3 shows an example of the cross section of the detached scale. The scale formed on the lower sample is well merged with the scale formed on the electrolytic iron sample. Fig. 4 shows the stress change during the measurement when an axial compression stress of 6.4 kPa is applied on. The stress increases almost linearly when the electrolytic iron sample is pulled up. The sample and tools are elastically deformed in this region. The stress decreases rapidly after the scale has detached. In this case, the adhesive strength for the maximum stress obtained is 0.65 MPa. After the stress reaches its maximum, the stress decreases to zero. It is considered that the fraction of detaching oxide scale increases during this stress decrease.

Fig. 2 Surface appearance after tests.

(a) Opposite (upper) side sample. (b) Lower sample (scale is completely detached)

Fig. 3 Optical microscopy image of a cross section of the scale after adhesive strength measurement.⁷⁾ Surface appearance after tests

Fig. 4 Load change during experiment. The stress was recorded every 10 ms

The axial compressive stress applied on the scale during scale merging changes from 6.4 kPa to 12.7 MPa using material A, as shown in Table 1. The measured scale adhesive strengths are shown in Fig. 5. Although scale adhesive strength has a certain range when the axial compressive stress is small, above approximately 0.1 MPa, the adhesive strength becomes higher in proportion to the axial compressive stress. When the compressive stress was 12.7 MPa, the measured stress became very high and reached 12.8 MPa and the attached part of the specimen was damaged. It is assured that under these conditions the scale adhesive strength is more than 12.8 MPa.

The above results indicate that scale adhesive strength is proportional to compressive stress above a certain value and that it can be measured below that value. The scale adhesive strength of material A is on the order of 1 MPa.

4. Discussion

The method used to measure scale adhesive strength in this study is similar to that used by Kushida et al.⁶⁾ The scale exfoliation stress obtained by Kushida et al. ranges from 15 to 50 MPa and is much higher than the adhesive strength we obtained, which may be attributed to the high compressive stress applied on during scale joining. The result shown in Fig. 5 indicates that measured scale adhesive strength is affected by compression stress during scale merging above 0.04 MPa. A sufficiently low compressive stress during scale merging is required to measure scale adhesive strength.

Fig. 5 Effect of axial compressive stress during scale merging on scale adhesive strength

We should realize that the scale adhesive strength measured in this method may differ from the adhesive strength of the scale formed on an actual steel surface. The scale on the actual steel surface has three layers: hematite, magnetite, and wustite. In this method, the scale structure changes from a three-layer structure to a wustite mono layer structure during merging. The scale structure affects growth stress in oxide scale. Growth stress may be a factor for scale adhesion.

Using material B shown in Table 1, two experiments were conducted to examine the effect of outer force on scale adhesive strength. In the first experiment, the axial compressive stress was 25 kPa. After scale merging, the samples were cooled down and the axial compressive stress was maintained at 25 kPa. After cooling, a cross section of the sample was obtained using the focused ion beam (FIB) technique to avoid damaging the structure of the scale/steel interface. **Fig. 6** shows a scanning electron microscopy (SEM) image of the cross section of the scale/steel interface. Voids between the scale and the steel are observed. It is suggested that the scale makes partial contact with the steel. The void formation at the scale/steel interface is associated with the outward diffusion of iron ions in the oxide via ion vacancies, which condense and form voids near the scale/steel interface.

In the second experiment, quite a high axial compressive stress (12.7 MPa) was applied on. As in the first experiment, the axial compressive stress was held at 12.7 MPa while the samples were being cooled. A cross section of the sample was made by the FIB technique; the SEM image obtained at the cross sections is shown in **Fig. 7**. There are few voids between the scale and the steel. It appears that both the scale and the steel intruded on each other by plastic deformation at the interface, resulting in a rough interface. It is reported that the oxide of wustite deforms plastically above 700°C⁸⁾. It is considered that the increased contact area and rough interface result in pronounced scale adhesion.

Fig. 6 SEM image of scale/steel interface when axial compression of 25 kPa is applied on during scale merging⁷⁾

Fig. 7 SEM image of scale/steel interface when axial compression of 12.7 MPa is applied on during scale merging

The scale/steel interface when oxide scale is formed is shown schematically in **Fig. 8(a)**. There are some voids at the interface and the contact area is small. The interface where compressive stress is applied on the scale is shown schematically in **Fig. 8(b)**. There are few voids at the interface. The contact area is larger and a rough interface is also formed.

Fig. 8 Schematic representations of scale/steel interface.
(a) Oxidized. (b) Axial compression is applied on

It is considered that the improvement of scale adhesion by external force is due to the increase in the contact area and roughness at the scale/steel interface. It is supposed that, during hot rolling, scale is compressed under high pressure in roll bites and has very high adhesive strength. In this study, we suggest

that external force applied on oxide scale causes the scale/steel interface to deform plastically, thereby increasing the contact area between the scale and the steel; both the deformation and the increased contact area affect scale adhesive strength. However, the mechanism of scale adhesion on steel is not yet understood. Further research is necessary.

5. Conclusion

A method of measuring scale adhesive strength by oxidizing, scale-merging and separating two samples has been proposed. The application of this method revealed that the scale adhesive strength is on the order of 1 MPa and is increased by the application of compressive stress. The scale/steel interface is deformed by external force, thereby increasing the contact area between the scale and the steel. It is suggested that the contact area and rough interface between an actual scale and steel greatly affects scale adhesive strength.

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