

Reduction of Oxide Scale with Hydrogen

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Abstract: During hot rolling process metals will inevitably oxidize because of high temperature and air condition. In order to guarantee the surface quality, acid pickling is applied to remove the oxide scale while waste acid will do harm to the environment. Faced with the problem, by means of reduction process of hot-rolled plates, the oxide scale will be reduced to iron, so that acid pickling is unnecessary. One pass cold rolling procedure was applied. The compression ratios of hot-rolled plates with oxide scale were 10%, 18%, 26% and 31%, respectively. After that, samples mentioned above including a sample without deformation were separately reduced under hydrogen atmosphere condition (5% H₂ + 95% Ar in volume percent) at 600–1000 °C. The thermal gravimetric apparatus (TGA) was used to establish accurate experimental condition and obtain complete mass loss data. Field emission electron probe microanalysis (EPMA) was applied to analyze scale morphology change and composition distribution through the oxide scale. It was found that the sample with 26% compression ratio could be reduced completely at 900 °C which was favorable to galvanization.

Key words: oxide scale; pickling free; reduction; hydrogen; cold rolling

In the production process of hot-rolled steels, oxidation is unavoidable because of the high temperature and oxygen containing atmosphere. Oxide scales must be removed to ensure the steel surface quality and pickling process is applied. However, plate surface after the pickling process is always not satisfactory because of surface defect caused by residual oxide. Moreover, the pollution produced during pickling process is also serious^[1]. Reduction of oxide scale is probably one of the most studied technologies for solving the problem mentioned above. Both H₂ and CO can be used as reducing medium. H₂ is more favorable because the generated gases consist of H₂O and H₂, avoiding the release of CO and CO₂ which will lead to air pollution. Mechanism and reaction kinetics of reduction of Fe oxide under H₂/CO condition have been studied^[2–4]. And reduction process of Fe₂O₃ is fully discussed^[5,6]. Pineau et al.^[7,8] have given an comprehensive review on kinetics of reduction of magnetite and hematite. Hayes^[9] proved that during reduction of Fe oxide structures and reaction rates could be explained through the occurrence of four principal mechanisms: continu-

ous gas pore formation, dense metal layer growth, discontinuous metal layer breakdown and continuous coupled growth of metal and pores. Mann et al.^[10] believed that iron formed in the early stages was epitaxial with the host magnetite; at later stages the epitaxial iron was vanished and fissures frequently formed in the metal. Reduction of oxide scale of hot-rolled plates is seldom discussed. Because of the formation of the Fe layer during the reduction process, the reductive reaction will be hindered. The present study will discuss reduction process at 900 °C after cold rolling and the phenomenon is explained from the point of view of hydrogen diffusion.

1 Experimental

Four plates in size of 600 mm × 50 mm × 2.1 mm were supplied by a hot rolling mill and chemical composition was shown in Table 1. By one pass cold rolling, the plates were compressed to 10%, 18%, 26% and 31%, respectively. Before cold rolling process, plate surface was covered with lubricant in order to protect the oxide scale. Coupon specimens in size of 15 mm × 10 mm × 2.1 mm were machined out

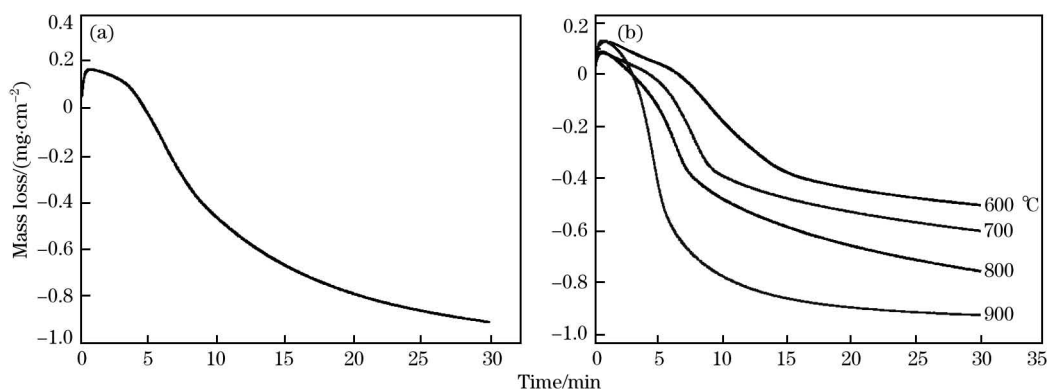
Table 1 Chemical composition of the specimens
(mass percent, %)

C	Si	Mn	P	S	Nb
0.06	0.19	1.08	0.01	0.008	0.015

of these plates and a hole of 2 mm diameter was drilled near one end of each specimen for suspending in the furnace chamber of thermal gravimetric apparatus (TGA). Finally specimens were washed with acetone.

Isothermal reduction experiments were carried out in TGA. The mass change during the period of reduction was monitored continuously. Specimens were suspended in furnace chamber of TGA. After evacuation, it was filled with argon until the pressure of furnace chamber returned to 10^5 Pa. In each test, a sample was heated in an argon atmosphere at a rate of 25 °C/min until it reached experimental temperature. As the temperature reached the pre-set value, dry 5% $H_2 + 95\%$ Ar (volume percent) with a flow rate of 50 mL/min was introduced into the TGA chamber to conduct the 30 min reduction experiment. Then the TGA chamber was cooled to room temperature at a rate of 25 °C/min.

The metallographic preparation including mounting, grinding, and polishing, was performed in the usual sequence^[11]. Electron probe microanalysis (EPMA) was applied to observe scale morphology and analyze composition distribution through the scale.



(a) Specimen without cold rolling reduced at 1000 °C; (b) Specimen with compression ratio of 26% reduced at 600 – 900 °C.

Fig. 1 Mass loss curves of specimens under 5% H_2 /Ar condition

As shown in Fig. 3, the compression ratios of the specimens with oxide scale were 10% , 18% and 26% , respectively. As demonstrated in Fig. 3 (a), the oxide scale was intact and slight crack could be observed in the scale. It was mainly composed of eutectoid structure ($Fe + Fe_3O_4$), and the thickness of the scale was about 8 μ m. By means of cold rolling,

2 Results and Discussion

As shown in Fig. 1 (a), the specimen with oxide scale was reduced at 1000 °C under 5% $H_2 + 95\%$ Ar (volume percent) condition for 30 min. The oxide scale was relatively intact compared with other rolled specimens. Although the temperature was very high, there was no obvious steady stage of the mass loss curve, and it means that after 30 min reductive reaction the oxide scale has not been totally reduced to iron layer. So it was not possible that the oxide scale could be reduced completely without a number of cracks in the scale, even if the reduction temperature was high. As shown in Fig. 1 (b), specimens compressed 26% were reduced under 5% $H_2 + 95\%$ Ar (volume percent) condition at 600 – 900 °C for 30 min. There was no steady stage could be found in the other three curves except at 900 °C. Residual oxides still existed in the oxide scale after the 30 min reductive process at 600 – 800 °C. It could be concluded that both high temperature and cracks were necessary for the complete removal of oxide scale with hydrogen. If the reduction temperature was lower than 900 °C, maybe greater compression ratio was needed to provide enough cracks for complete reductive reaction. But if the compression ratio was above 31% as shown in Fig. 2, parts of oxide scale fell off and the integrity of the oxide scale was damaged.

as shown in Figs. 3 (b) to 3 (d), with the compression ratio increasing, more cracks could be found in the scale. At room temperature oxide scale was harder compared with the substrate. During the cold rolling process, because the stress produced in the oxide scale by deformation could not be released through plastic deformation, cracks were observed and

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