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Abstract

Oxidation characteristics of a microalloyed low carbon steel were investigated by a hot rolling mill combined with acceleration cooling system over the cooling rate range from 20 to 70°C/s. The effects of cooling rate after hot rolling on microstructure and phase composition of oxide scale were examined. The results showed that the increase of the cooling rate has a significant influence on the decrease of the grain size and surface roughness of oxide scale. A higher cooling rate promotes the formation of retain wustite and primary magnetite precipitation while suppression of eutectoid α -iron precipitates. This provides the possibility to enhance potential contribution of magnetite precipitates with preferable ductility, and hence fabricates a desired oxide-scale structure under continuous post cooling conditions considering a suitable cooling rate.

Keywords

microalloyed, steel, behaviour, effect, oxidation, rate, cooling

Disciplines

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Effect of cooling rate on oxidation behaviour of microalloyed steel

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Keywords: Oxide scale, Magnetite precipitation, Cooling rate, Wustite decomposition

Abstract. Oxidation characteristics of a microalloyed low carbon steel were investigated by a hot rolling mill combined with acceleration cooling system over the cooling rate range from 20 to 70°C/s. The effects of cooling rate after hot rolling on microstructure and phase composition of oxide scale were examined. The results showed that the increase of the cooling rate has a significant influence on the decrease of the grain size and surface roughness of oxide scale. A higher cooling rate promotes the formation of retain wustite and primary magnetite precipitation while suppression of eutectoid α -iron precipitates. This provides the possibility to enhance potential contribution of magnetite precipitates with preferable ductility, and hence fabricates a desired oxide-scale structure under continuous post cooling conditions considering a suitable cooling rate.

Introduction

During hot rolling, oxidation occurs inevitably on the surface of steel strips, and hence forms the oxide scale which is a complex mixture of three iron oxide phases: hematite (Fe_2O_3), magnetite (Fe_3O_4) and wustite (Fe_{1-x}O , $x=0.84-0.95$) [1, 2]. The mechanical properties and subsequent descalability intimately depend on phase distribution and microstructure of these oxides. Correspondingly, oxidation behaviour of steel strips is well known to be highly sensitive to both steel composition and oxidation conditions, in particular the post cooling conditions at the exit of the last finishing stand of the rolling mill [3]. Throughout the run-out laminar cooling, the three-layer oxides structure is maintain until an eutectoid point of Fe-O phase equilibrium diagram is reached if oxygen is available at the temperature of around 570°C [4, 5]. The microstructure of oxide scale is complicated further by the instability of wustite below 570°C, which results in wustite decomposition to magnetite or eutectoid structure of magnetite and proeutectoid iron [6, 7].

Under various cooling regimes, different reaction paths taking place in the oxide layer lead to particular phase compositions of the oxide scale. In order to achieve desired phase constitution of the final oxide scale, some elegant studies have been done to investigate the morphological development of wustite in oxides scale on hot-rolled steel strips under various simulated coiling and cooling conditions [2, 8-11]. It has been reported that increasing the cooling rate and lowering the coiling temperature can obtain the oxide structure which could improve descalability [8]. Several research results have indicated that the thinner oxide scale was produced when cooled in acceleration air cooling [9]. The relative amount of oxide scale formed on forced-air-cooled wire rod of low carbon steel can be decreased by 15% using modifying the cooling rate [10]. Although the effect of forced air cooling on the transformation of wustite to magnetite through the laboratory route is known [2, 8-10], the literature on industrial route and acceleration water cooling is very limited, due to inevitably involving the effect of water vapour. In previous studies, oxidation behaviour of microalloyed and stainless steel was investigated under the 19.5% H_2O moisture air [12, 13]. In addition, a previous study has been made to explore the influence of the coiling temperature, ranging from 550 to 570°C, on the morphology and the phase composition of the oxide scale [11]. It was found that the water

content and coiling temperature have significant influence on the thickness of oxide scale and morphologies of magnetite precipitation. Therefore, it is necessary to address the effect of cooling rate on the oxide scale for completeness.

The aim of the present investigation is to characterise the influence of different cooling rates on the oxidation behaviour of a microalloyed low carbon steel in a wide range of temperatures and oxidation time. A newly developed acceleration water-cooling system is employed to simulate cooling conditions after hot rolling, with emphasis on a cooling strategy to achieve desired phase compositions of oxide scale.

Experimental

Material and sample preparation. The material used was a commercial microalloyed low-carbon steel. The chemical compositions are listed in Table 1. The steel was supplied as hot-rolled strips by a steel plant of China. The steel samples were cut to be a flat plate with section size of $300 \times 100 \times 3.5 \text{ mm}^3$. Each specimen has a lead as illustrated in Fig. 1a, A is the point where specimen starts to contact the roll when it enters the roll gap, and B is the point where the specimen undertakes full set reduction. Prior to the experiments, the specimens were ground using SiC papers of 2400 mesh to the surface finish of $0.5 \mu\text{m}$, and cleaned in acetone solution.

Table 1 Chemical compositions of the studied microalloyed steel.

C	Si	Mn	Cr	P	Al	V	Nb	Ti	S	N
0.1	0.15	1.61	0.21	0.014	0.034	0.041	0.041	0.016	0.002	0.003

Apparatus and oxidation procedure. The controlled rolling process followed by controlled cooling was performed on a pilot rolling mill combined with a newly developed acceleration water-cooling system, as shown in Fig. 1b. Hot rolling experiments were carried out on 2-high Hille 100 experimental rolling mill with 225mm diameter rolls and rolling speeds of 0.12-0.72 m/s. Reheating was conducted using a high temperature electric resistance furnace with chamber size $350(\text{W}) \times 330(\text{H}) \times 870(\text{D})$, power of 30,000 watts and current of 40 A.

Fig. 1 a) Lead of rolling specimens; b) Photograph of a 2-high Hille 100 experimental rolling mill combined with an acceleration water-cooling system.

An acceleration cooling technology [14] was employed to simulate the ROT (Run Out Table) cooling process after hot rolling. The technology has larger flow rates than conventional cooling methods (such as spray or water column cooling), as well consists of top and bottom double banks of water spray nozzles. Total water flow is well known as 17,000 L/min/m of cooling length,

corresponds to 9,200 L/min-m² assuming a 1.8m width, which is more than double the maximum flow rate for the conventional ROT cooling [15]. Two Raytek GPs noncontact infrared pyrometers were located above the roll gap entry and exit of the cooling equipment to simultaneously monitor and control the sample temperature and cooling rates.

The following procedure was conducted for each of the cooling rate experiment. Each specimen was reheated at 1100°C in the furnace under nitrogen atmosphere and soaked for 10 min to ensure uniform temperature. Upon removal from the furnace, each specimen was placed on the entry roll platform of the rolling mill and allowed to air cool to a predetermined temperature at which each individual strip sample was given a single rolling reduction of approximately 15% and a rolling speed of 0.2 m/s. The rolled specimens were cooled by an acceleration cooling device above, from accelerated cooling start temperatures of 730, 760°C down to coiling temperature (600-350°C) at three different cooling rates of 20, 40, 70°C/s to observe the effect of cooling rate on the oxidation behaviour of steel, as shown in Fig. 2.

Fig. 2 Schematic diagram of laboratory processing schedules used to simulate industrial hot rolling. RT-Reheating temperature (1100°C), RT2-temperature at exit of the rough mill, FT-finishing temperature, accelerated cooling start temperature (730-760°C), CT-Coiling temperature (600-350°C).

Analysis methodology. During the controlled cooling process, the real-time temperature profile was recorded from Raytek GPs noncontact infrared pyrometers. After oxidation, Atomic Force Microscope (AFM) was employed to investigate the surface morphology and roughness of the oxide scale formed on the sample surface. The oxidised samples were immediately examined in a Nanoscope IIIA AFM from Digital Instruments, in order to prevent contamination of the sample surface after each oxidation experiment. The surface topography and roughness after oxidation were analysed in the contact mode with a lateral resolution of 1-5 nm and a vertical resolution of 0.08 nm. The nanoprobe cantilevers were made of silicon nitride (Si₃N₄) with normal spring constant of 0.06 N/m and with a tip radius of 5-10 nm. Images consisted of 512×512 pixels, and the scanning frequency was 1Hz. The Digital Nanoscope software was used to analyse the surface roughness profiles of the samples. For all samples, several images were recorded at different locations to verify the reproducibility of the observed features. An X-ray diffraction (XRD) using a GBC MMA diffractometer with mono-chromated Cu-K α radiation was used to detect the phase composition of the samples before and after oxidation. The operating voltage and current of X-ray beam were set as 35kV and 28.6mA, respectively.

Results and Discussion

Temperature distribution. Fig.3a shows cooling rates during the acceleration cooling process, depending on the start temperature and the exit temperature. It can be seen that initial temperature of

the strips is around 760°C and then the final cooling temperature can be obtained ranging from 600 to 350°C. Fig. 3b shows an example for the recording of the temperature distribution during the laboratory hot rolling trials. As expected, the acceleration cooling device can achieve a relatively higher cooling rate [15]. It can be seen that the temperature difference of the steel strip at entry and exit of cooling device can reach up approximately 150 °C. The temperature gradient at either entry or exit of the cooling system is approximately uniform, which is preferred to generate uniform oxide scale. It is noted that the temperature increases suddenly at end of air cooling as observed in Fig.3b. It can be explained through accelerating cooling the temperature gradient within the solid is not equalised with the gradient at the surface, so there is an accumulation of heat flux at the surface.

Fig. 3a) Variation of temperature at different cooling rate during acceleration cooling, b) example for the recording of the temperature distribution during the laboratory hot rolling trials

Microstructure of oxide scale. Fig. 4 shows AFM images of the surface topography of the oxidised specimens cooled with cooling rates of 20 and 70°C/s. It can be seen that different morphologies and grain sizes of oxide scale are exhibited according to the cooling rate. As shown in Fig. 4a, slow cooled microstructure of oxide scale shows progressively larger grain size (mostly 10µm) coexisting with minor precipitates at its grain boundaries. A comparison of morphology of oxide scale through higher cooling rate (70°C/s) is shown in Fig. 4b. It has been observed that the grain size of oxide scale decreases with an increase of cooling rate. Furthermore, surface roughness achieves more smooth and homogeneous to grain surface at higher cooling rate. In addition, Fig. 5 provides an example of 3D image of an oxidised sample cooled at cooling rate of 70°C/s. Also it can be noted that as for slow cooling rate, surface roughness cannot be measured using AFM due to the height of overflow 3µm. It indicates that very low surface roughness value is obtained where the oxide scale formed high temperature through higher cooling rate.

The above observations can be explained as follows: by increasing the cooling rate, shorter time is available for atomic diffusion and larger amount of wustite is available for phase transformation to magnetite precipitation. That results in a morphology that is characterised by relatively fine oxide scale formed on the steel surface in accelerated cooling condition. These results of microstructures are in good agreement with literatures [6, 8, 16-18].

Phase composition. Fig. 6 shows the X-ray patterns of the oxide scales formed on the micralloyed low carbon steel at 750°C followed the post cooling with 20, 40, 70°C/s. It is clear from Fig. 6 that oxide phases are dominated by retain wustite and magnetite containing sparse hematite. It has also been suggested that the variation of cooling rate has significantly influence on the phase composition of oxide scale. The previous achievement is maintained when higher cooling rate is applied, particularly promotes the formation of the retained wustite. Nonetheless, the magnetite intensities gradually increase concurrently with wustite drastically reducing with decreasing cooling rate. Also, it can be noted that there is not detected α -iron phase as a product of complete eutectoid reaction,

which indicates that the fraction volume of these phases is below 3%. It was possible to suppress α -iron precipitation by rapid cooling rate. Wustite could transform to very fine magnetite crystals without a noticeable composition change. This is due to that, during the transition $\text{Fe}_{1-x}\text{O} \rightarrow \text{Fe}_3\text{O}_4$, the number of oxygen ions per crystal volume remains constant, only iron being lost [6].

Fig. 4 Morphology of the specimens cooled with cooling rates of a) 20°C /s, b) 70°C/s.

Fig. 5 AFM images of surface roughness of the oxidised sample cooled with cooling rate of 70°C/s.

Fig. 6 XRD pattern at different cooling rate conditions.

The above results are in good agreement with earlier studies results [2, 6, 8, 18]. However, it has been reported in literature [8] that even when cooling rate is faster than 60°C/min, some magnetite precipitates were hardly suppressed and observed inside the wustite layer. Thus, it is possible to enhance potential contribution of magnetite precipitation with preferable ductility, and hence produce a desired eutectoid oxide-scale structure under continuous post cooling conditions considering a suitable cooling rate [13].

Summary

The current investigation effort was made to study the effect of cooling rate during accelerated cooling after hot rolling on the oxidation behaviour of microalloyed low carbon steels. The following conclusions can be summarised in the present work. The cooling rate has significant influence on the microstructural features and phase composition of final oxide scale. The increase of the cooling rate leads to a formation of oxides phase with relatively small grain size and low surface roughness. A higher cooling rate promotes the formation of retained wustite and primary magnetite precipitates at the expense of α -iron precipitation. This provides the possibility to design a desired oxide-scale structure under a suitable cooling rate using accelerated cooling system.

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References

- [1] M. Krzyzanowski, J.H. Beynon and D.C.J. Farrugia: *Oxide Scale Behavior in High Temperature Metal Processing* (Wiley-VCH, Darmstadt, 2010).
- [2] P.H. Bolt: *Res. Int.*, Vol. 75 (2004), p. 399.
- [3] R.Y. Chen, W.Y.D. Yuen: *Oxid. Met.*, Vol. 59 (2003), p. 433.
- [4] J. Paidassi: *Acta Metall.*, Vol. 6 (1958), p. 184.
- [5] H.A. Wriedt: in *Binary Alloy Phase Diagrams*, 2nd edn., edited by T.B. Massalski, H. Okamoto, P.R. Subramanian and L. Kacprzak, Vol. 2, ASM Intern., Materials Park (1990), p. 1739.
- [6] G.D. West, S. Biroscas, R.L. Higginson: *J. Microsc.*, Vol. 217 (2005), p. 122.
- [7] X.L. Yu, Z.Y. Jiang, X.D. Wang, D.B. Wei and Q. Yang: *Adv. Mat. Res.*, Vols. 415-417 (2012), p. 853.
- [8] R.Y. Chen and W.Y.D. Yuen: *Oxid. Met.*, Vol. 53 (2000), p. 539.
- [9] J. Tominaga, K. Wakimoto, T. Mori, M. Murakami, T. Yoshimura: *ISIJ*, Vol. 22 (1982), p. 646.
- [10] R. Bhattacharya, G. Jha, S. Kundu, R. Shankar, N. Gope: *Surf. Coat. Tech.*, Vol. 201 (2006), p. 526.
- [11] X.L. Yu, Z.Y. Jiang, X.D. Wang, D.B. Wei, Q. Yang: *Adv. Mat. Res.*, Vols. 415-417 (2012), p. 853.
- [12] D.B. Wei, J.X. Huang, A.W. Zhang, Z.Y. Jiang, A.K. Tieu, X. Shi and S.H. Jiao: *Wear*, Vol. 271 (2011), p. 2417.
- [13] X.L. Yu, Z.Y. Jiang, D.B. Wei, C.L. Zhou, Q.X. Huang, D.J. Yang: *Wear*, (2013), in press.
- [14] F. Akio, I. Sadanori, H. Yoshimichi, M. Toru, M. Yoichi and I. Shozo: EP patent application, 2002, EP1210993A1.
- [15] J.C. Herman: *Ironmak. Steelmak*, Vol. 28 (2001), p.159.
- [16] B. Gleeson, S.M. Hadavi and D.J. Young: *Mater. High Temp.*, Vol. 17 (2000), p. 11.
- [17] D.P. Burke, R.L. Higginson: *Scripta Mater.*, Vol. 42 (2000), p. 277.
- [18] S. Biroscas, D. Dingley, R.L. Higginson: *J. Microsc.*, Vol. 213 (2004), p. 235.