



Oxidation of simulated recycled steels with 0.23 and 1.03 wt.% Si in Ar–20% H_2O at 900 °C



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ARTICLE INFO

Article history:

Received 12 March 2014

Accepted 4 June 2014

Available online 13 June 2014

Keywords:

A. Iron

A. Steel

C. High temperature corrosion

C. Oxidation

ABSTRACT

Simulated recycled steels containing 0.002, 0.23 and 1.03 wt.% Si were oxidised in Ar–20% H_2O at 900 °C. The oxidation kinetics of the steels was linear-parabolic. Higher Si content in the steel resulted in prolonging the period of linear oxidation and increasing Si content from 0.23 to 1.03 wt.% enhanced the parabolic oxidation rate constant. The integrated framework using thermodynamic analysis in static and dynamic modes together with the stoichiometric one was proposed for analysing the global reaction mechanisms. The role of hydroxyl ion on the oxidation behaviour of the steels was discussed.

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1. Introduction

This article addresses the investigation results that form part of a studying programme in the high-temperature corrosion behaviour of recycled steel and its properties vital to a hot rolling process. The present paper focuses on the role of Si with the content relative to the one in the recycled steel on its oxidation kinetics and structure of thermal oxide scale formed in the simulated hot-rolling atmosphere containing water vapour. The industrial context of the work is as follows.

The production of low carbon hot-rolled steel can be carried out by two different routes: blast-furnace and electric-arc-furnace [1,2]. A plain low carbon steel slab made via the first route has Si content up to 0.005–0.01 wt.% [1–4]. The Si content can be increased, for instance, to 0.1 wt.% or higher in the case that higher strength of plain carbon steel is required [5]. For the electric-arc-furnace route, which is the main one applied in Thailand, scraps are used as main raw materials. In this case, Si content in the slab is allowed to be 0.25–0.3 wt.% in practice [3], and even 0.35 wt.% for steel production in mini steel plants elsewhere [6]. After the steelmaking process, the slab is further delivered to hot rolling. In this process, the steel is exposed to an atmosphere containing water vapour in the temperature range from 1200–1300 °C

at the upstream reheat furnace to around 600 °C at the downstream coiling unit [7,8]. In this condition, thermal oxide scale forms on the hot-rolled steel. In the case that steel contains Si or water vapour is mixed in the hot gas, the oxidation behaviour of the steel can be significantly changed as reviewed in the following.

Fukumoto et al. [9] studied the oxidation of Fe–1.5Si alloy in air with 10.5% H_2O at 800 °C. It was reported that oxidation kinetics was likely a sigmoidal curve i.e. relatively slow in the early period, but after around 5.5 h the oxidation rate increased rapidly and then slowly again. Zhang et al. [10] studied the oxidation of iron with silicon content up to 1.5 wt.% in the interstitial-free steel in Ar–5% H_2 –0.652% H_2O at 843 °C. It was reported that increasing silicon in steel promoted the oxidation rate. The oxidation kinetics seems to be more parabolic than that found in the work of Fukumoto et al. [9]. However, the role of Si on the evolution of the scale formed by the external oxidation was not reported [10]. Kusabiraki et al. [11] studied oxidation of iron with higher Si content up to 3 wt.% in Ar– H_2O . Parabolic growth of the inner and internal oxidation layers was found. Furthermore, many works have been conducted to study the oxidation of iron with Si content up to 3–5 wt.% in various atmospheres, for example Fe–3Si in ambient air with various pressures [12], Fe–Si alloys with Si up to around 3 wt.% in humidified hydrogen [13,14], Fe–(1,2,3)Si and Fe–6Al–3Si in dry and humidified Ar–20% CO_2 [15,16] and Fe–(1,2,3,5)Si in 68% CO –26% H_2 –6% H_2O [17]. However, the Si contents in those works were far beyond that typically found in recycled steel which is our present interest. The former works of our group recently reported [18,19] were the study of the oxidation behaviour of

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Fe–Si alloys in N₂–20%O₂–15%H₂O at 900 °C for a short period of time of 15 min. This was to focus on the passivity and passivity breakdown mechanism of Si-rich oxide formed during the short-term oxidation [19]. However, its oxidation behaviour at longer period of time has not yet been investigated.

The objective of this work was then to investigate the oxidation behaviour of the steels with 0.23 wt.% Si comparing to those with 0.002 and 1.03 wt.% Si in the atmosphere containing water vapour during 16 h. The corrosion kinetics and evolution of the oxide scale structure when Si content in the steel was varied are discussed. This study will fulfill the understanding of the role of Si content particularly on the formation of external oxide scale in water vapour which has never been reported.

2. Experimental

The chemical compositions of the studied steels are shown in Table 1. Here, the steel with 0.002 wt.% Si is called “reference steel”. Steel with 0.23 wt.% Si has a Si content close to the maximum allowed in industry, whilst steel with 1.03 wt.% Si has a Si content higher than the maximum limit. The latter steel was included in the present study in order to help understand what would happen, in terms of oxidation behaviour and oxide scale structure in water vapour, if the Si content in the recycled steel was significantly more than the typical maximum limit. The initial shape of the samples was cylindrical with the diameter of 5 mm. For an oxidation test, they were cut to have the length of 5 mm. The samples were ground on SiC papers to 1200 grit, ultrasonically cleaned by ethanol, dried in air, and immediately taken to the oxidation test.

The oxidation tests were conducted by a thermobalance (SETARAM B 24) using a vitreous silica tube with the inner diameter of 2 cm. The studied gas was prepared by flowing Ar into the first flask containing boiling water for saturation. The gas was further delivered to the second flask containing water heated to the temperature of 64 °C. Using thermodynamic data from Ref. [20], at this temperature the partial pressure of the water vapour in equilibrium with water was 0.2 bar. The mixed gas was further fed via a transfer pipe covered by the heating tape at the temperature of 110 °C to prevent the undesired condensation into the furnace. The applied flow rate was 6 litre per hour, corresponding to the linear velocity of 0.53 cm s⁻¹. From the plot of mass gain as a function of time, the linear oxidation behaviour with the substantial increase in mass gain of the order of 1–10 μg cm⁻² s⁻¹ was clearly observed in the early period of oxidation. Extrapolation of this line to the abscissa was carried out to determine the onset value of the mass gain used in the study. The studied oxidation temperature was 900 °C. At this temperature, the partial pressure of oxygen was calculated to be 8.45 × 10⁻⁷ bar using thermodynamic data from Ref. [21]. After oxidation for 16 h, Ar was flowed instead of Ar–20%H₂O and the sample was cooled down in the furnace to room temperature.

For physico-chemical characterisation, cross-section of the samples was observed using a scanning electron microscope (SEM) model LEO S440 equipped with an energy-dispersive spectroscope for elemental analysis. An X-ray diffractometer

(Shimadzu-6100) using the Cu Kα line (λ = 1.5406 Å) was applied to identify the oxide phase of the scale. A Raman spectroscope (Renishaw RM 1000) was employed to determine the nature of the oxides at different locations within the scale.

3. Results

3.1. Oxidation kinetics

Fig. 1 shows mass gain of the studied samples as a function of time. It is noted that the experiment was performed twice for each sample as illustrated by overlaid normal and bold lines. It is primarily seen that oxidation kinetics of all studied steels follows the linear regime in the initial period and then tends to approach the parabolic law in the successive stage. The overall mass gain of the reference steel is higher than those of the steels with 0.23 and 1.03 wt.% Si. For the latter two steels, the mass gains are similar during the first 5 h, and afterwards a significant increase in mass gain of the steels with higher Si content is observed.

To help identify the linear from parabolic oxidation regimes, a plot of the derivative of mass gain with respect to time as a function of time has been proposed [18]. In this study, the thermobalance measured mass of the sample every 5.3 s. The increment of mass over each time interval was obtained and is presented in Fig. 2. In the figure, the increment of mass at the time *t* is defined as the mass measured after oxidation for *t* seconds subtracted by the mass measured after the oxidation for (*t* – 5.3) seconds. The increment of mass, not the increment of mass per unit area, is presented here because it will be further used for stoichiometric analysis in Section 4.2.

From this figure, the plot is the horizontal line in the early period when the oxidation is linear. The ordinate value in this region is proportional to the linear rate constant and the width of this line corresponds to the period of time when the oxidation is linear. The linear rate constants of the steels were measured and plotted in an Arrhenius form as shown in Fig. 3(a) where literature values are also present. The linear rate constant of the reference steel is 2.53 ± 0.08 μg cm⁻² s⁻¹. This value is higher than those of the steels with 0.23 and 1.03 wt.% which are 1.06 ± 0.19 and 1.25 ± 0.08 μg cm⁻² s⁻¹, respectively. The linear rate constants of the steels with 0.23 and 1.03 wt.% Si are not significantly different. Furthermore, the time period of the linear oxidation stage is plotted in Fig. 3(b). This value is 1.41 h ± 17 min for the reference steel. It lasts longer to 2.17 h ± 9 min for the steel with 0.23 wt.% Si, and even longer to 5.87 h ± 27 min for the steels with 1.03 wt.% Si.

From Figs. 1 and 2, we can further see that after the linear oxidation regime the increment of mass tends to be less with the time elapsed indicating the protectiveness of the formed oxide scale. Furthermore, in the period following the linear stage, the square of mass gain is plotted as a function of time as shown in Fig. 4. The linear relationship between these two variables is observed, demonstrating the parabolic oxidation behaviour of the steels. From the figure, it is noted for the reference steel and the steel with 1.03 wt.% Si that a linear-to-parabolic transition period is initially observed before the plot follows the parabolic law. Fig. 5 shows the parabolic rate constants of the studied steels compared to

Table 1
Chemical compositions of the studied steels (wt.%).

Steel	Chemical composition (wt.%)					
	C	Si	Cu	S	P	Fe
Reference steel	0.00610	0.00198	0.03294	0.00782	0.01193	Bal.
Steel with 0.23 wt.% Si	0.00980	0.23213	0.03154	0.00752	0.01138	Bal.
Steel with 1.03 wt.% Si	0.01269	1.02854	0.03335	0.01017	0.01409	Bal.

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