

## Article

# Effect of Oxidation Temperature on the Oxidation Process of Silicon-Containing Steel

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**Abstract:** The oxidation behavior of silicon-containing steel was studied by applying segmented heating routes similar to the atmosphere and heating process in an industrial reheating furnace. The oxidation tests were carried out on a simultaneous thermal analyzer at heating temperatures of 1150 °C–1300 °C. The morphologies of  $\text{Fe}_2\text{SiO}_4$  were observed by SEM, and the penetration depths of the  $\text{Fe}_2\text{SiO}_4$  layer at different oxidation temperatures were determined by using the Image-Pro Plus 6.0 software. The results show that at heating temperatures  $\geq 1235$  °C, the oxidation rate and total oxidation mass gain have no relation with the heating temperature; the mass gain versus time follows a linear law after about 1164 °C (lower than the eutectic temperature of fayalite). In addition, the oxidation rate first decreases slowly and then drops from 1190 °C to 1210 °C during the isothermal holding stage. With the increase in temperature, the oxidation rate and mass gain also increase gradually; the relationship between the mass gain and time is close to a parabolic law. Moreover, at a heating temperature of 1150 °C, the oxidation rate decreases rapidly during the isothermal holding stage, and the mass gain versus time follows a parabolic law.

**Keywords:** oxidation temperature; oxide scale; fayalite; oxidation kinetics

## 1. Introduction

Silicon (Si) is an important alloying element for improving the corrosion resistance and mechanical performance of conventional alloys [1–3]. However, the addition of Si often leads to red scale, a surface defect of hot rolled steels [4]. In Si-containing steels, red scale is related to the presence of fayalite ( $\text{Fe}_2\text{SiO}_4$ ) which forms by the combination of  $\text{SiO}_2$  and FeO, firmly bonding steel substrate and iron scale [5,6]. The melting point of  $\text{Fe}_2\text{SiO}_4$  is about 1173 °C and liquid  $\text{Fe}_2\text{SiO}_4$  irregularly penetrates into FeO and the matrix. It is difficult to completely wipe off the FeO layer after descaling due to the very high strength of the eutectic compound  $\text{Fe}_2\text{SiO}_4/\text{FeO}$ . The remaining FeO scale is oxidized into red  $\text{Fe}_2\text{O}_3$  during following cooling process. The oxidation temperature influences the oxidation process and  $\text{Fe}_2\text{SiO}_4$  morphology during the reheating process [7–9]. So far, some studies on the effects of the oxidation temperature on the oxidation mass gain and  $\text{Fe}_2\text{SiO}_4$  have been reported.

Suarez *et al.* [10] found that oxidation treatments in air at temperatures higher than 570 °C lead to three different iron oxide layers: wustite (FeO), magnetite ( $\text{Fe}_3\text{O}_4$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ), in increasing order of oxygen content, going from substrate to free surface. Below 570 °C, only the last two layers are thermodynamically stable. Cao *et al.* [11] investigated the isothermal oxidation kinetics of low carbon steel at temperatures ranging between 500 °C and 900 °C. They reported that the oxidation mass gain per unit area with time has a parabolic relation and that the oxidation rate decreases with time. Mouayd *et al.* [12] studied the oxidation of silicon-containing steels in the temperature range of 900 °C–1200 °C. They found that a passivation period occurs during the oxidation of silicon steels

and that a high silicon concentration leads to a long passivation of oxidation at temperatures lower than 1173 °C. The passivation period is due to the formation of a thin silica layer as Si is more prone to oxidation than iron. This layer dramatically slows down the diffusion of  $\text{Fe}^{2+}$  towards the surface in contact with the oxidizing atmosphere. Fukagawa *et al.* [13] and Okada *et al.* [5] reported that when the temperature reaches 1173 °C (the eutectic temperature of fayalite), the liquid  $\text{FeO}/\text{Fe}_2\text{SiO}_4$  flows into the external scale and develops a net-like distribution, considerably increasing the scale thickness of Fe-Si alloy.

In previous studies, the oxidation atmosphere was usually introduced into the chamber of the simultaneous thermal analyzer (STA) during the isothermal oxidation stage. Few studies have adopted heating routes similar to those in the reheating technology used in the industrial production of hot strips; *i.e.*, steel slabs are kept in an oxidation atmosphere at temperatures ranging from low to high temperature during the whole reheating process. In addition, the heating temperatures applied in most studies were below 1200 °C, whereas the heating temperature in industrial production is higher than 1200 °C in most cases. Therefore, it is necessary to investigate the oxidation process of silicon-containing steels under an oxidation atmosphere and temperature similar to those used in industrial production. In the present study, the oxidation kinetics of silicon-containing steel was investigated by using oxidation atmosphere and temperatures similar to those applied in the industrial reheating process. The results provide theoretical reference for controlling oxide scale defects and preventing red scale on the surface of silicon-containing steels.

## 2. Materials and Methods

Silicon-containing steel composed of Fe-0.07C-1.11Si-1.2Mn-0.035Al-0.016Cr (wt. %) was obtained from a hot strip plant. The oxidation tests were carried out on a Setaram Setsys Evo STA (Setaram, Lyon, France). The dimensions of the samples were 15 mm × 10 mm × 3 mm. A hole with a diameter of 4 mm was drilled near the edge center in each sample for suspension in the oxidation chamber. The surfaces of all samples were polished on a series of SiC polishing paper up to 2000 to remove the scale before the tests. The experimental procedure was designed according to the industrial reheating process, as shown in Figure 1. The samples were first heated to 850 °C at a rate of 20 °C/min and then heated to predetermined temperatures (1150, 1190, 1210, 1235, 1260 and 1300 °C) at a slower heating rate of 6.6 °C/min; they were kept at these temperatures for different periods to ensure the same total heating time for all samples. After the isothermal holding, the samples were cooled to room temperature at a rate of 50 °C/min. The accuracy of temperature measurement is  $\pm 0.5$  °C, so that the effect of thermal drifts of thermobalance setup on mass gain could be ignored. The atmosphere for slabs in industrial reheating furnace contains 4.0 vol. % oxygen. A binary gas mixture of oxygen and nitrogen with an oxygen concentration of 4.0 vol. % was introduced into the STA chamber during the heating and holding stages to simulate the oxidizing atmosphere of reheating furnace in the industrial production process; the oxygen concentration was changed to 21.0 vol. % during the cooling stage to simulate the oxygen concentration in the air. The mass gain of the samples and the temperature were digitally recorded during the oxidation processes.

After the oxidation tests, the samples were molded in resins at room temperature to protect the integrity of the oxide scale. The cross-sections of mounted samples were ground and polished. The microstructures of the oxide scale were observed on a Nova 400 Nano scanning electron microscope (SEM) (FEI, Hillsboro, OR, USA) operated at an accelerating voltage of 20 kV. In addition, the penetration depths of the  $\text{Fe}_2\text{SiO}_4$  layer at different oxidation temperatures were determined by using the Image-Pro Plus 6.0 software (Media Cybernetics, Rockville, MD, USA).

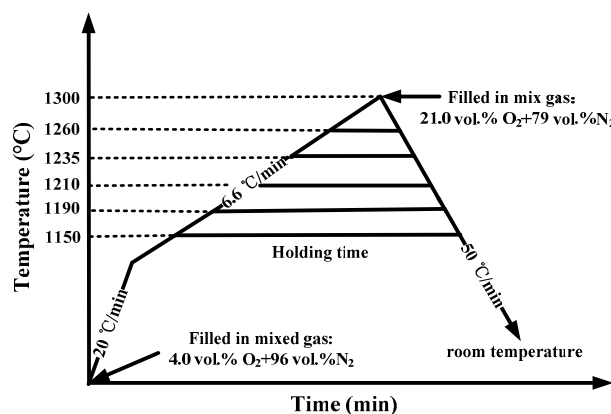


Figure 1. Oxidation experiment route.

### 3. Results and Discussions

#### 3.1. Morphology of $\text{Fe}_2\text{SiO}_4$

Figure 2 shows the morphologic images of  $\text{Fe}_2\text{SiO}_4$  at different oxidation temperatures. Table 1 presents the main atomic percentages of the inner layer at temperature 1260 °C (Figure 2e); the inner layer is a composite of eutectic compounds  $\text{Fe}_2\text{SiO}_4/\text{FeO}$ . The bright area (Spectrum 1) is FeO, while the dark area (Spectrum 2) is mainly  $\text{Fe}_2\text{SiO}_4$ . In addition, Mn is detected in the bright and dark area. The constitution of the inner layer is consistent with the results in the other studies [14,15]. Figure 2 shows that the granular  $\text{FeO} + \text{Fe}_2\text{SiO}_4$  layer is dispersed close to the substrate at 1150 °C. However, a continuous net-like distribution layer of  $\text{FeO} + \text{Fe}_2\text{SiO}_4$  is observed at 1190 to 1300 °C. At temperatures higher than the melting point of  $\text{Fe}_2\text{SiO}_4$  (1173 °C), the liquid  $\text{Fe}_2\text{SiO}_4$  is forced to penetrate at the grain boundaries of the steel substrate and oxide scale [16–18]. The penetration depths of  $\text{Fe}_2\text{SiO}_4$  at different oxidation temperatures were measured by using the Image-Pro Plus 6.0 software (Figure 3). Several images were used to improve the accuracy of  $\text{Fe}_2\text{SiO}_4$  measurement. With the increase in temperature from 1150 to 1190 °C, the depth of  $\text{Fe}_2\text{SiO}_4$  increases significantly. However, at temperatures ranging from 1190 to 1235 °C, the depth of  $\text{Fe}_2\text{SiO}_4$  increases slowly. As the temperature increases further, the depth of  $\text{Fe}_2\text{SiO}_4$  shows no significant change.

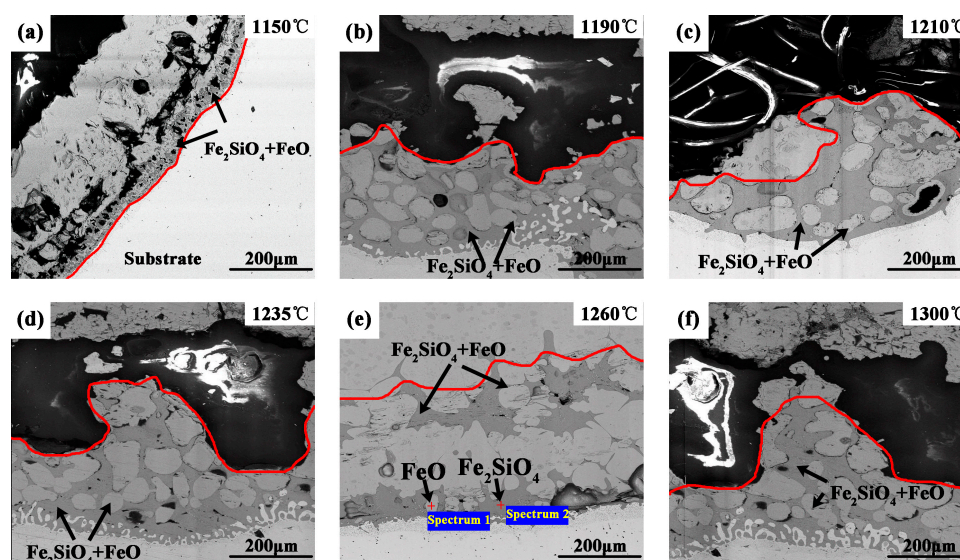
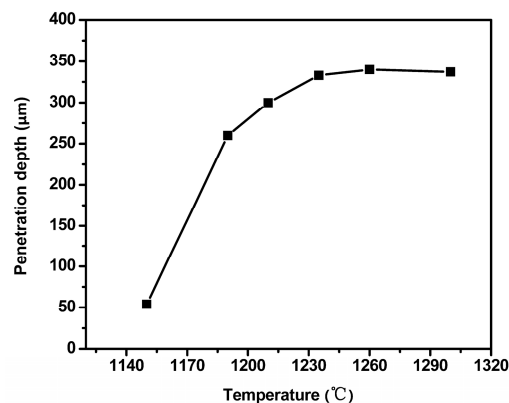


Figure 2. SEM cross-section micrographs showing the distribution of  $\text{Fe}_2\text{SiO}_4$  at different oxidation temperatures: (a) 1150 °C; (b) 1190 °C; (c) 1210 °C; (d) 1235 °C; (e) 1260 °C; (f) 1300 °C.

**Table 1.** Main atomic percentages of different layers (at. %).

Chemical Elements	O	Si	Fe	Mn
Bright area (Spectrum 1)	53.71	-	45.62	0.67
Dark area (Spectrum 2)	59.66	12.16	27.29	0.89

**Figure 3.** Penetration depths of  $\text{Fe}_2\text{SiO}_4$ .

### 3.2. Oxidation Analysis

Figure 4 shows the mass gain per surface square centimeter of samples and the oxidation rate as functions of time at different oxidation temperatures. The whole oxidation process includes three stages; slow oxidation, intense oxidation, and final stop oxidation. At a heating temperature  $\geq 1235^\circ\text{C}$ , during the intense oxidation stage, the oxidation rate remains constant, and the mass gain per surface unit with time follows a linear law. At temperatures ranging from 1190 to 1235  $^\circ\text{C}$ , during the intense oxidation stage, the oxidation rate first decreases slowly and then drops obviously. The mass gain per surface square centimeter with time is close to a parabolic law. At 1150  $^\circ\text{C}$  (lower than the eutectic temperature of fayalite-wustite), during the intense oxidation stage, the mass gain versus time shows a parabolic relationship. There are three critical temperatures: the starting oxidation temperature (SOT, point A), the intense oxidation temperature (IOT, point B), and the finishing oxidation temperature (FOT, point C). Before the SOT, the rates of diffusion and combination among ions are slow due to the lower temperature; thus, the sample is almost not oxidized. Between the SOT and the IOT, the adhesion of Si and  $\text{O}_2$  is stronger than that of Fe and  $\text{O}_2$  at low temperatures [19,20], leading to the appearance of  $\text{SiO}_2$  before the formation of the FeO. Because  $\text{SiO}_2$  retards the diffusion of iron ions and oxygen atoms, the oxidation reaction is slow as the temperature increases. The jump in oxidation rate is due to a buoyancy effect, caused by turbulent gas flows at about 43 min in Figure 4b. At the intense oxidation temperature, the oxidation rate increases dramatically. This is due to the considerable increase in the diffusion rate at higher temperatures [21–24], resulting in a sharp increase in the oxidation rate. After the FOT, the oxidation reaction stops rapidly. According to the mass gain curves, the three critical temperatures at different oxidation temperatures are determined (Table 2). Because the oxygen concentration increases to 21.0 vol. %, the oxidation rate increases dramatically at the beginning of the cooling process.

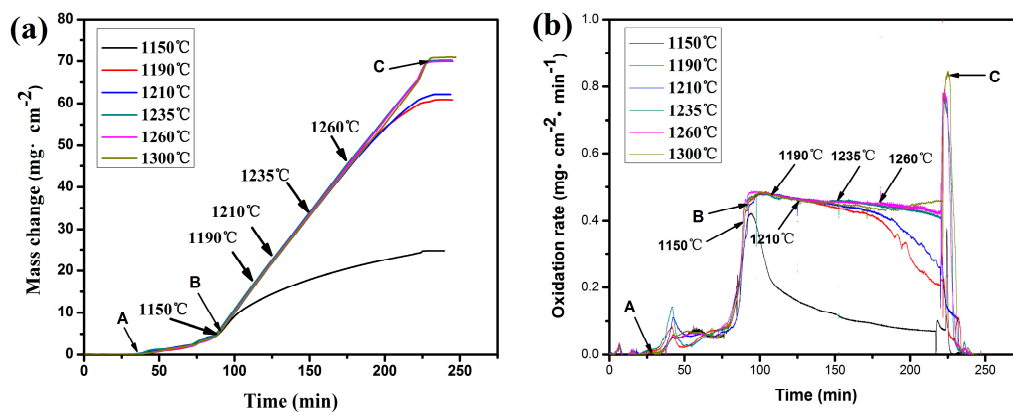


Figure 4. (a) Mass gain versus time and (b) oxidation rate versus time at 1150 to 1300 °C.

Table 2. Transition temperatures of the oxidation reaction.

Oxidation Temperature/°C	Starting Oxidation Temperature (A)/°C	Intense Oxidation Temperature (B)/°C	Finishing Oxidation Temperature (C)/°C
1150	480	1150	803
1190	483	1161	1158
1210	506	1167	1160
1235	491	1164	1162
1260	487	1162	1161
1300	494	1167	1166

Regarding to the oxidation kinetics at constant heating temperature, it is often reported that the mass gain and heating time follows parabolic rule [12,15]. The results in the present study indicate that the mass gain versus time follows the parabolic law at the temperature below 1210 °C. However, mass gain changes linearly with time at the heating temperature higher than 1235 °C. This point is useful for the determination of heating routes of silicon-containing steels in industrial production of hot strip because the heating temperature is often higher than 1235 °C. The results demonstrate that the total mass gains of oxidation are the same at the temperature above 1235 °C.

The oxidation kinetics during the isothermal holding period at temperatures below 1235 °C can be described by a parabolic formula proposed by Kofstad *et al.* [25]:

$$\Delta W^n = K_p \cdot t \quad (n > 1) \quad (1)$$

The oxidation kinetics during the isothermal holding period at temperatures higher than 1235 °C can be represented by a linear equation:

$$\Delta W = K_l \cdot t \quad (2)$$

where,  $\Delta W$  is the mass gain of the oxide scale in  $\text{mg}/\text{cm}^2$ ;  $K_l$  and  $K_p$  are the oxidation rate constants at a constant temperature  $T$ ; and  $t$  is the oxidation time. The value of  $n$  in Equation (1) varies from 1 to 2 from a linear relationship to a parabolic law. Therefore, Equation (2) can be integrated into Equation (1).

The oxidation rate constants for different heating temperatures can be calculated based on the experimental data, as shown in Table 3. The kinetic constants increase at 1150 to 1210 °C but remain similar at temperatures higher than 1235 °C.

As shown in Figure 4 and Table 2, there is no significant change in the intense oxidation temperatures at a heating temperature  $\geq 1190$  °C, and the average IOT is 1164 °C. The IOT is a temperature range of about 1060 °C–1164 °C, where the end of temperature of the interval is selected as IOT in this paper. It is interesting to note that the intense oxidation temperatures are lower than



the eutectic temperature of fayalite (1173 °C), indicating that the intense oxidation has no relationship with the melting of fayalite.

**Table 3.** Oxidation kinetics parameters.

Temperature (°C)	$K_l$ (mg·cm <sup>-2</sup> ·min <sup>-1</sup> )	$K_p$ (mg <sup>2</sup> ·cm <sup>-4</sup> ·min <sup>-1</sup> )
1150	-	0.075
1190	-	0.141
1210	-	0.318
1235	0.452	-
1260	0.459	-
1300	0.454	-

At temperatures higher than 1164 °C, during the heating stage, the oxidation rate shows no significant change (Figure 4b). Regarding to the oxidation of steels, five main factors should be considered. Firstly, the radius of the oxygen ion is much larger than that of the iron ion; thus, it is difficult for the oxygen ion to diffuse into the reaction interface [26]. As a result, the oxidation rate depending on the diffusion of iron ions increases with the temperature. The liquid Fe<sub>2</sub>SiO<sub>4</sub> provides diffusion passages for diffusion of iron ions. Thus, the mass gain increases with the temperature. Secondly, manganese (Mn) in the steel substrate can be oxidized [6]. Manganese oxides which are detected in Table 1 increase the oxidation mass gain. Thirdly, the oxidation reaction of carbon (C) causes decarburization in the steel. Simultaneous scaling and decarburization has been studied [27,28], and the general consensus is that during steel oxidation, carbon reacts with the scale via the following reaction [28]:



where [C] denotes carbon in solution in steel. Further reaction between CO and the scale may produce CO<sub>2</sub> via the following reaction:

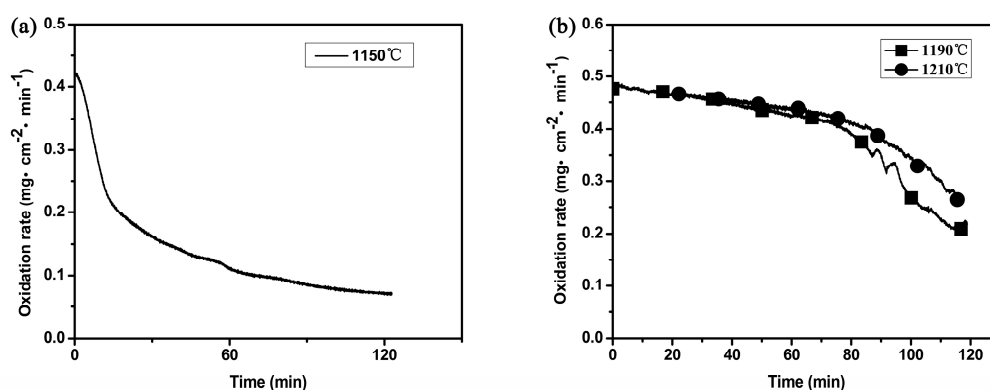


From the reactions, mass loss due to decarburization overlaps with the mass gain from the oxidation. Fourthly, wustite occurs with a broad composition range that can be described as Fe<sub>1-x</sub>O, with  $x$  varying from 0.04 to 0.17 [11]. The ion transport through oxides is dominated by the stoichiometry of the oxide (wustite) that is p-type [26,29]. The fact that  $x$  is always positive implies that wustite is cation-deficient and that the interaction between cations and vacancies is the predominant mechanism of mass transport. The amount of iron ions increases and the vacancies of iron ions decrease with the increasing of temperature, leading to a reduction in diffusion passages. Therefore, the oxidation rate decreases with the increase of temperature. Fifthly, the thickness of oxide scale hinders the diffusion of iron ion, decreasing the oxidation rate. The fact that oxidation rate at temperatures higher than 1164 °C has no significant change in Figure 4b indicates the dynamic equilibrium of above five factors affecting oxidation rate. In addition, linear mass gain could be caused by a constant gradient in chemical potential that drives oxygen/iron diffusion.

In addition, as shown in Figure 4, at heating temperatures ranging from 1235 to 1300 °C, the oxidation rates of the samples are basically the same, and the mass gain versus time follows an almost linear relationship, indicating that the oxidation mass gain is not sensitive to temperature. The liquid Fe<sub>2</sub>SiO<sub>4</sub> is forced to penetrate at the grain boundaries of the steel substrate and oxide scale, which plays a role as passage for the ions transfer process and provides fast diffusion passages to accelerate the oxidation rate [14]. In the isothermal holding process at 1235 °C, the oxidation rates of Fe, Mn and C can reach dynamic equilibrium during the isothermal holding period. Therefore, the oxidation rate remains constant. At temperatures higher than 1235 °C, the oxidation rates of Fe, Mn and C can reach dynamic equilibrium, resulting in the same oxidation rate. Therefore, the oxidation rate is not sensitive to temperature.

Figure 5a shows the oxidation rate as a function of time in the isothermal holding period at 1150 °C. As indicated in the figure, the oxidation rate decreases rapidly; Figure 4a shows that the mass gain versus time has a parabolic relationship. Because the oxygen concentration is constant, the oxidation reaction is mainly determined by the iron ion concentration in the interface reaction during the isothermal holding period at 1150 °C. The thickness of the oxide scale, the amount of solid-phase particles of  $\text{SiO}_2$  and  $\text{Fe}_2\text{SiO}_4$  increase during the isothermal holding period at 1150 °C, which is unfavorable for ion diffusion. Therefore, the iron ion concentration in the reaction interface decreases quickly, resulting in a rapid decrease in the oxidation rate.

Moreover, as shown in Figure 5b, the oxidation rate first decreases slowly and then reduces obviously during the isothermal holding period at 1190 °C and 1210 °C. In the isothermal holding period, the amount of liquid  $\text{Fe}_2\text{SiO}_4$  increases with the oxidation time, promoting the diffusion of ions. However, at the same time, the thickness of the oxide scale increases, which is unfavorable for ion diffusion. At the beginning of the isothermal holding period, the promotion and inhibition effect on the ion diffusion of the oxidation reaction basically reach dynamic equilibrium. Therefore, the oxidation rate decreases slowly. With the progress of the isothermal holding time, the thickness of the oxide scale increases such that the inhibition effect on the ion diffusion becomes greater than the promotion effect. Therefore, the oxidation rate decreases obviously with time. In addition, the promotion effect of temperature on the ion diffusion increases with the temperature, resulting in a slightly higher oxidation rate at high heating temperatures. Because  $\text{Fe}_2\text{SiO}_4$  is in solid phase at 1150 °C, which hinders the diffusion of ions, the decrease in oxidation rate at 1150 °C is significantly greater than that at 1190 °C and 1210 °C during the isothermal holding period.



**Figure 5.** Oxidation rate versus time during the isothermal holding period: (a) 1150 °C; (b) 1190 °C and 1210 °C.

Figure 6 presents the total mass gain as a function of the oxidation temperature for the same total heating time (about 111 min). The total mass gain increases significantly at temperatures ranging from 1150 to 1190 °C. At 1190 °C, the liquid  $\text{Fe}_2\text{SiO}_4$  that provides fast diffusion passages promotes the oxidation process, leading to a significant increase in mass gain compared with that at 1150 °C where the  $\text{Fe}_2\text{SiO}_4$  is a solid phase. The total mass gain increases slowly at temperatures ranging from 1190 to 1235 °C due to the gradual increase in the oxidation rate, as previously mentioned. At temperatures  $\geq 1235$  °C, the total mass gain shows no significant change relative to 1235 °C. At temperatures above 1235 °C, the oxidation rate is higher, as shown in Figure 4b, resulting in a larger total mass gain than that at low temperatures. The mass gains are basically the same at 1235 to 1300 °C because the oxidation rates are similar, indicating that the total mass gain has no relationship with the heating temperatures higher than 1235 °C.

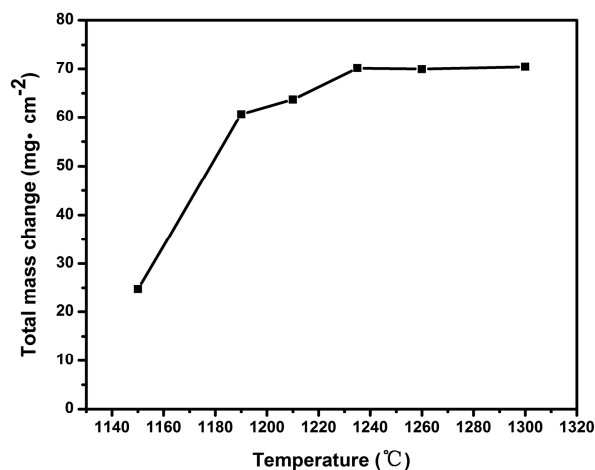


Figure 6. Total mass gain at different oxidation temperatures.

#### 4. Conclusions

The oxidation behavior of silicon-containing steel was studied by using an atmosphere and heating route similar to those applied in the industrial reheating process. The oxidation experiments were carried out on STA at different oxidation temperatures. The effect of oxidation temperature on the oxidation process of silicon-containing steels was investigated. The results show that at temperatures higher than 1235 °C, the mass gain versus time follows a linear law after the intense oxidation temperature (lower than the eutectic temperature of fayalite). The oxidation rate and oxidation total mass gain have no relationship with the heating temperature. However, the oxidation rate first decreases slowly and then drops markedly during the isothermal holding stage at 1190 °C and 1210 °C. With the increase in the heating temperature, the oxidation rate and mass gain increase gradually, and the mass gain with time is close to a parabolic law. Only at the temperatures below 1210 °C does the mass gain versus time follow a parabolic law. Moreover, the penetration depth of  $\text{Fe}_2\text{SiO}_4$  and the total mass gain increase quickly and then go up slowly before finally becoming basically unchanged at temperatures ranging from 1150 to 1300 °C.

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