

A study on the metal loss of spring steel 40S2 heated for rolling and peculiarities of the structure, chemical and phase composition of its scale

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Abstract. The article presents the results of study on kinetics of high-temperature oxidation of steel 40S2. For the implementation of predictive calculations, an equation is obtained that allows metal loss to be determined depending on the temperature and the heating time. The structural features, chemical and phase composition of the scale, formed during high-temperature heating of 40S2 steel are studied. It is established that the hematite layer bordering on the furnace atmosphere has a minimum thickness of about 40 – 60 μm, the magnetite layer has a thickness of about 250 – 280 μm, the largest thickness 800 – 900 μm has a wuestite layer with secondary magnetite appeared upon cooling. The chemical and phase compositions of hematite and magnetite layers are relatively uniform in thickness and perimeter. The scale layer adjacent to the steel surface is characterized by a much greater macrostructural, chemical, and phase inhomogeneity. In addition to the wustite-magnetite mixture, iron silicates with an increased silicon content are present.

1. Introduction

Medium- and high-carbon siliceous steels are widely used for the manufacture of springs of various purposes and rod griffons – elastic elements of rail fastenings of rail joints with assembled rails and sleepers [1 - 3]. The rolled metal for their production is obtained by hot rolling of continuous castings, which are heated in the continuous furnaces. High-temperature heating is accompanied by scaling, leading to losses in the useful metal mass and the formation of a scale layer on the surface of the workpieces, which can lead to a decrease in the quality of round products surface [4, 5].

The structure and properties of the scale formed play an essential [5, 6]. When developing the heating technology and assigning temperature modes that provide metal-saving heating [7 – 9], information is needed on the kinetics of the steel oxidation. Since precise analytical solutions for finding the dependence of the oxidation intensity of steels of different chemical composition are missing, the only way to obtain such dependences is empirical.

The analysis of special technical literature [4, 5, 9 - 11] shows that the oxidation kinetics of 40S2 silicon steel has been little studied, and there are no quantitative data on the influence of the temperature-time factor on its loss. Thus, obtaining new information on the kinetics of high-temperature oxidation of spring steel 40S2 and the features of its scale structure is of great practical importance.



2. Methods of research

In this paper a study on the metal loss of steel 40S2 (chemical composition, %: 0.377 C, 1.604 Si, 0.65 Mn, 0.204 Cr) and the structural features, phase and chemical composition of its scale is presented. The oxidation kinetics is studied using a gravimetric method on cylindrical samples with a diameter 11 mm, length 20 – 30 mm. The samples were cut from sections of round bars. Before cutting out the samples, the rods were softened and turned on a turning workstation with in order to completely remove the decarburized layer.

In the laboratory experiments the samples heating up to temperatures 850 – 1200 °C (with 50 °C interval) and holding at constant surface temperature for 5, 15 and 25 min was carried out in the electric resistance oven SUOL-0.25.1/12.5-II with silicon carbide heaters in the air atmosphere.

Before and after the experiments the geometrical dimensions of the samples were measured with a caliper with 0.1 mm accuracy, the mass was determined on Vibra AF-220CE weighing-machine with 0.1 mg accuracy. To remove scale from the surface, the samples were etched in the warmed up to 40-60 °C 10% sulfuric acid solution with addition of 0.1 g/l thiourea.

When processing the experimental data, it was considered that the dependence of the loss (or the thickness of the oxidized layer) on time obeys the square root law, which reflects the diffusion mechanism of scale formation, the temperature of the metal surface is constant in time, and the dependence of the metal loss on the temperature-time factor obeys the following equation [4]:

$$L = k\sqrt{\tau}, \quad (1)$$

where k – the oxidation rate constant, $\text{g}/(\text{cm}^2 \cdot \text{min}^{0.5})$; τ – metal holding time at constant temperature, min.

For relation (1), it is assumed that the oxidation rate constant obeys the Arrhenius equation, which can be represented in the form [4]

$$k = A \exp\left(-\frac{Q}{RT}\right), \quad (2)$$

where A – the empirical coefficient, which depends on the chemical composition of steel, the atmosphere composition and other factors, $\text{g}/(\text{cm}^2 \cdot \text{min}^{0.5})$; Q – energy of activation or loosening of the oxide lattice, J/kmol ; $R = 8.3143$ – universal gas constant, $\text{J}/(\text{kmol} \cdot \text{K})$; T – the temperature, K.

Assuming $B = Q/R$, relation (2) is written in the form

$$k = A \exp\left(-\frac{B}{T}\right), \quad (3)$$

where the coefficient B has the dimension K.

3. Analysis of the results of experimental studies

Figure 1 shows the dependence of the logarithm of oxidation rate constant on temperature for steel 40S2.

For predictive calculations the dependence was obtained that makes it possible to determine the metal loss L as a function of the temperature and the heating time:

$$L = 22371.9 \cdot \exp(-18435/T) \cdot \sqrt{\tau} \quad (4)$$

The analysis of the obtained results shows that the temperature-time dependence of the metal loss of steel 40S2 is similar to the dependences in chemical composition to steels 60S2 and 60C2KhA [4,

10, 11]. In the temperature range 1050 – 1100 °C there is sharp intensification of metal loss. For steel 40S2 the temperature has a stronger effect on the loss compared with steel 60S2KhA. In particular with a holding time 25 min, an increase in temperature from 850 to 1175 °C for steel 40S2 is accompanied by an increase in the loss from 0.016 to 0.367 g/cm², i.e. almost in 23 times, and for steel grade 60S2KhA from 0.019 to 0.270 g/cm², i.e. in 14 times.

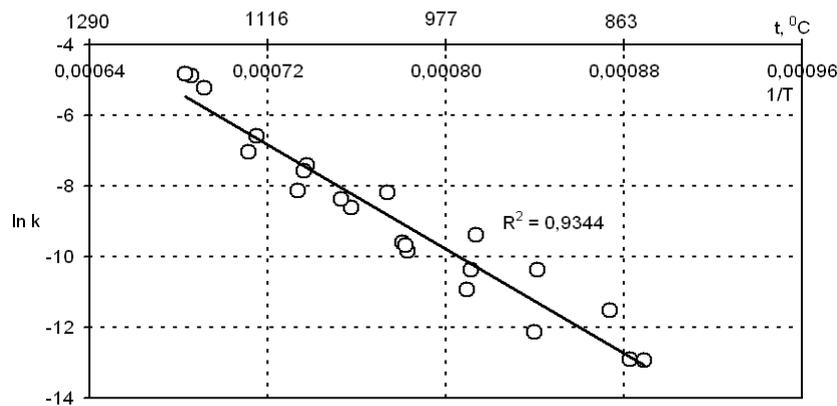


Figure 1. Dependence of the logarithm of oxidation rate constant on temperature for steel 40S2.

For steel grade 40S2 in the temperature range 850 – 950 °C, a less intense scale formation is observed in comparison with the steel grade 60S2KhA, and for temperatures over 1150 °C on the contrary.

A study of the structure, chemical and phase composition of the scale was carried out at the NUST MISIS on the electron scanning microscope TESCAN VEGA 3 with OXFORD AZtec energy dispersive attachment for micro-X-ray analysis. Samples for analysis were taken from scale plates with thickness 1 – 1.5 mm heated to 1175 °C, and the holding time was 25 minutes. After heating the samples with scale were cooled in atmospheric air in the conditions of natural convection.

Figure 2 shows a general view of the scale microstructure. The hematite layer bordering on the furnace atmosphere has a minimum thickness – approximately 40 – 60 μm, the magnetite layer has a thickness approximately 250 – 280 μm, the largest thickness 800 – 900 μm has a wuestite layer appearing upon cooling with secondary magnetite bordering on the steel surface.

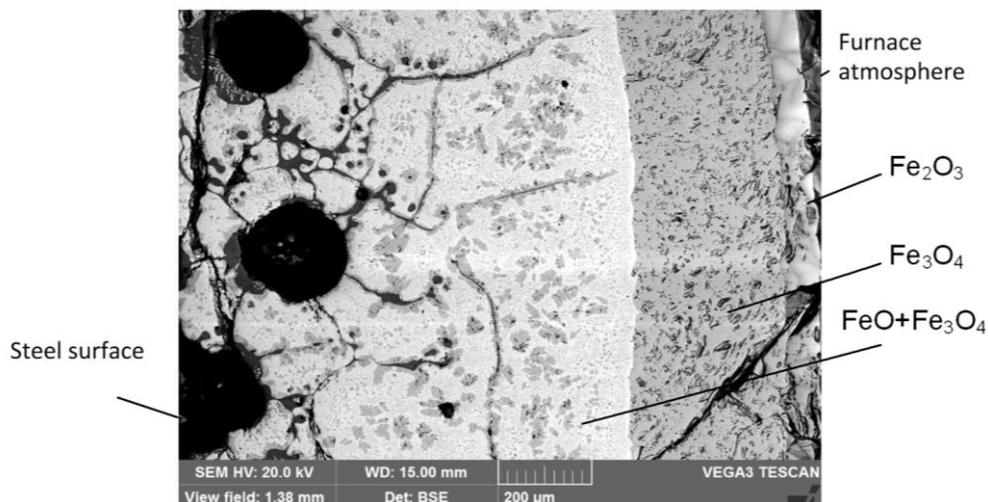


Figure 2. Scale microstructure.

The chemical and phase compositions of the hematite and magnetite layers are relatively uniform in thickness and perimeter (figure 3). Table 1 shows their compositions at different points in the layers.

The scale layer adjacent to the steel surface (figure 4) is characterized by a much greater macrostructural, chemical and phase inhomogeneity (table 2). In addition to the wustite-magnetite mixture, iron silicates are also present, characterized by an elevated (up to 13 wt.%) silicon content.

As the distance to the steel surface gets smaller, the number of sites enriched with silicon increases.

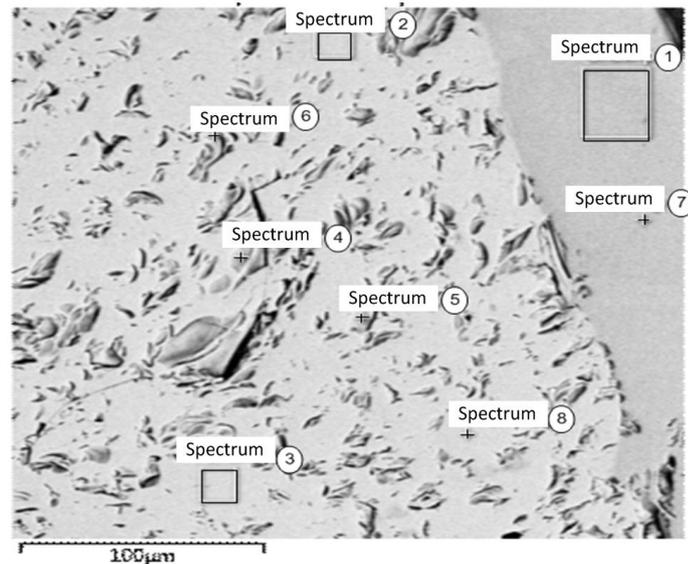


Figure 3. Microstructure of hematite and magnetite layers.

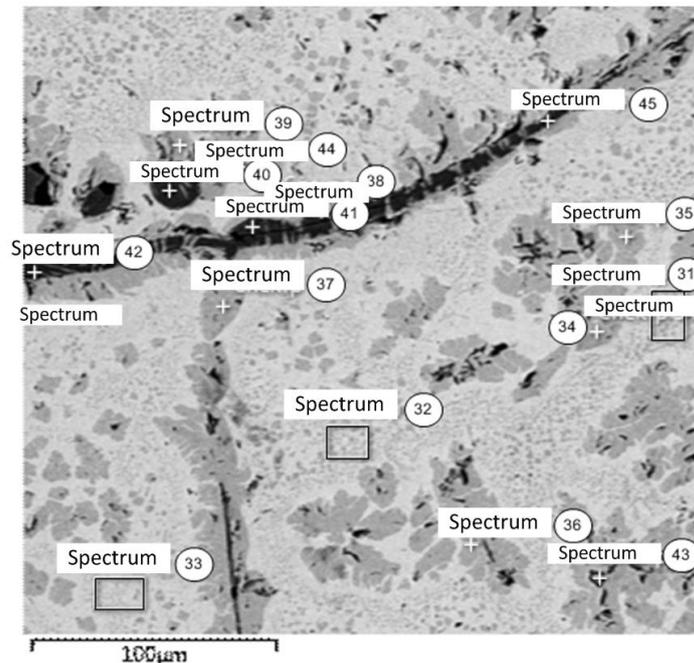


Figure 4. Microstructure of a wustite layer with secondary magnetite released during cooling.

Table 1. Chemical composition (wt.%) of hematite and magnetite layers.

Spectrum No.	O	Si	P	S	Mn	Fe	Total
1 hematite	33.52	0.09	0	0	0.08	66.31	100
7 hematite	34.11	0.06	0.03	0	0.1	65.7	100
2 magnetite	31.37	0	0.02	0.03	0.44	68.14	100
3 magnetite	31.17	0.06	0	0.02	0.52	68.23	100
4 magnetite	31.52	0	0	0.03	0.42	68.02	100
5 magnetite	31.21	0.02	0	0.01	0.45	68.3	100
6 magnetite	31.86	0.02	0	0.01	0.37	67.74	100
8 magnetite	30.19	0.12	0.03	0.03	0.48	69.16	100

Table 2. Chemical composition (wt.%) of the wuestite layer with secondary magnetite released during cooling.

Spectrum No.	O	Si	P	S	Mn	Fe	Cu	Mo	Total
31 wuestite + magnetite	27.58	0.4	0	0.03	0.55	71.44	–	–	100
32 wuestite + magnetite	27.97	0.06	0.02	0.05	0.42	71.49	–	–	100
33 wuestite + magnetite	27.37	0.04	0.01	0.03	0.43	72.12	–	–	100
34 wuestite + magnetite	31.02	0.02	0	0.05	0.42	68.5	–	–	100
35 wuestite + magnetite	31.77	0.44	0.02	0	0.35	67.43	–	–	100
36 wuestite + magnetite	31.44	0.01	0.01	0.02	0.4	68.12	–	–	100
37 wuestite + magnetite	31.24	0.3	0.02	0	0.31	68.12	–	–	100
38 wuestite + magnetite	31.1	0.33	0	0.04	0.35	68.17	–	–	100
39 wuestite + magnetite	31.67	0.22	0	0	0.36	67.75	–	–	100
40 iron silicates	36.74	13.58	0.08	0.03	1.61	47.96	–	–	100
41 iron silicates	36.26	13.48	0.14	0	1.65	48.47	–	–	100
42 iron silicates	36.04	13.34	0.14	0	1.35	49.14	–	–	100
45 iron silicates	31.87	7.39	0.01	0.02	1.09	59.62	–	–	100
43 iron silicates	26.72	2.12	0	0.05	0.3	70.24	0.22	0.35	100
44 iron silicates	31.77	1.61	0.04	0.04	0.45	66.09	–	–	100

4. Conclusion

The effect of the temperature-time factor on the oxidation kinetics of siliceous spring steel 40S2 is studied. For the implementation of predictive calculations, an equation is obtained that allows the loss to be determined, depending on the temperature and the heating time. It is established that in the temperature range 1050 – 1100 °C a sharp intensification of the metal loss is observed, for steel 40S2 in the temperature range 850 – 950 °C, less intense scale formation is characteristic in comparison with 60S2KhA steel, and for temperatures over 1150 °C, on the contrary.

With the use of electronic scanning microscopy, the structure of scale was investigated. It is established that the hematite layer bordering on the furnace atmosphere has a minimum thickness of about 40 – 60 µm, the magnetite layer has a thickness of about 250 – 280 µm, the largest thickness 800 – 900 µm has a wuestite layer with secondary magnetite precipitated upon cooling. The chemical and phase compositions of hematite and magnetite layers are relatively uniform in thickness and perimeter.

The scale layer adjacent to the steel surface is characterized by a much greater macrostructural, chemical, and phase inhomogeneity. In addition to the wustite-magnetite mixture, iron silicates with an increased silicon content are present.

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