THE STUDY OF SCALE FORMATION ON HOT ROLLED INGOTS AND BILLETS

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***Abstract**: During the heating for rolling, ingots and billets are affected by the chemical action of the heating atmosphere. Between the heating environment elements and steel components there are taking place chemical reactions initiated at the separation surface that may affect a layer in metal of about a few microns to a few millimeters. The thickness of the affected layer is a function of the environment nature, heated material composition, chemical reaction's type and kinetics, heating rate and temperature. The aim of this paper is to correlate the oxidation and the decarburization processes during the heating of ingots and billets for rolling. The study was made on ingots and billets rolled at the breakdown mill of the Special Steel Complex, Physics Laboratories from MECHEL Targoviste, Romania. The scope of the study is the analysis of the scale obtained from the rolling mill train in order to obtain specific information that will be used in adjusting the rolling parameters for diminishing the oxidation and decarburization and for recycling the scale in electric furnaces.*

***Keywords:** steel, ingot, billets, oxidation, decarburization, XRD analysis*

1. INTRODUCTION

The treatment furnances are heated by burning natural gas (>94% CH_4). The reducing or oxidizing character of the furnance atmosphere depends on its composition. The composition of the gases resulting from the natural gas burning depends on the quantity of consumed air. If α is the air consumption factor, the general equation of burning is:

$$CH_4+2\alpha(O+3.7N_2)=xCO+yCO_2+zH_2+tH_2O+mCH_4+nC+7.52\alpha N_2$$

where x, y, z, t and m are concentrations in m^3/m^3, and n is the quantity of carbon deposed as soot (in Kg/m^3). These coefficients have non-zero values for $0.25<\alpha<1$.
The general relations for the oxidation process are:

$$nMe+H_2O\leftrightarrow mMe_{n/m}O_{1/m}+H_2$$
$$nMe+CO_2\leftrightarrow mMe_{n/m}O_{1/m}+CO$$

If the values of CO/CO_2 and H_2/H_2O rations are greater than the values corresponding to the equilibrium $CO\text{-}CO_2$ and $H_2\text{-}H_2O$, the heating gas a non-oxidizing character. For the temperatures used for heating carbon and low alloy steels (1000°C) the ratio CO/CO_2 have to be greater than 2.5. This condition is fulfilled for $x/y\geq 2.5$ or $4(1-\alpha)/(4\alpha-1)\geq 2.5$, that leads to $\alpha\leq 0.47$ [2].

Thus, the equilibrium atmosphere produced by partially burning of the natural gas with $\alpha\leq 0.5$ is non-oxidizing for temperatures between 650°C and 1000°C. For carbon and most low and medium alloyed steels, the oxidized quantity has a parabolic variation in time:

$$\Delta m = k\sqrt{t}\ ,$$

where k is a constant depending on the oxidized material.

The oxidizing process is strongly enhanced by the temperature increasing:

$$\Delta m = Ae^{-\frac{Q}{RT}}$$

where Q is the activation energy (kcal/atom/g), A is a temperature independent constant, R is the universal constant of ideal gases and T is the temperature (K).

The decarburization process is characterized by the reactions between carbon (free, bounded in cementite, diluted in ferrite or austenite) and some component of the burned gasses:

$$C(\gamma) + CO2 \leftrightarrows 2CO$$
$$C(\gamma) + H_2O \leftrightarrows CO + H_2$$
$$C(\gamma) + 2H_2 \leftrightarrows CH_4$$

The way in which reactions take place is determined by the values of ratios CO/CO_2, H_2/H_2O and $CH_4/(H_2)^2$ [3].

Experiments show that the non-decarburising process is possible only for steels with carbon content less than 0.2% and for $\alpha < 0.5$-0.3, but for industrial furnaces α can take values only between 0.7 and 1 [4].

Also, the thickness of the decarburized layer of finished products depends on the decarburized layer thickness of semifinished ones [5].

2. EXPERIMENT AND RESULTS

Samples of scale were taken from the 950mm, 850mm, and 750mm rolling stands. The samples were prepared for structural analysis by XRD. Table 1 presents the technological parameters for low and medium alloyed steel ingots rolling. The ingots have a mass of 5.4 tons.

Table 1. Technological parameters for low and medium alloyed steel ingots rolling.

Ingot's temperature before heating [°C]	Air comsumption coefficient α	Heating temperature [°C]	Maintaining time [h]	Rolling temperature [°C]		
				950 stand	850 stand	750 stand
700-800	0.8-1	1000-1100	6-7	970-1000	900	850

The highest scale quantity is obtained at the 950 rolling stand. During the rooling process the billet is in direct contact with air. The thickness of the decarburized layer was measured on samples by 80 plotting the micro hardness curve $HV_{0,3}$ versus thickness d. The scale samples were visually inspected for visual aspect, color, layer structure. The thickness of the samples was measured. Thus, the thickness of the scale varies among 6 and 12mm for the 950 stand, 0.25-0.5mm for the 850 stand and is less than 0.3mm for the 750 stand. The decarburized layers have dimensions of 3 to 4mm. The sample preparation procedure consist of grinding and palletizing with borax (1/2 w/w). The procedure was applied also to three reference material powders: Fe_2O_3, Fe_3O_4 and FeO. The analyzed samples are presented in Table 2.

Table 2. **Analyzed samples description**

Sample number	0	1	2	3	4	5	6	7
Sample description	Fe_2O_3 reference material	950 stand, superficial layer	950 stand, deep layer	850 stand	750 stand	950 stand, mixed layers	Fe_3O_4 reference material	FeO reference material

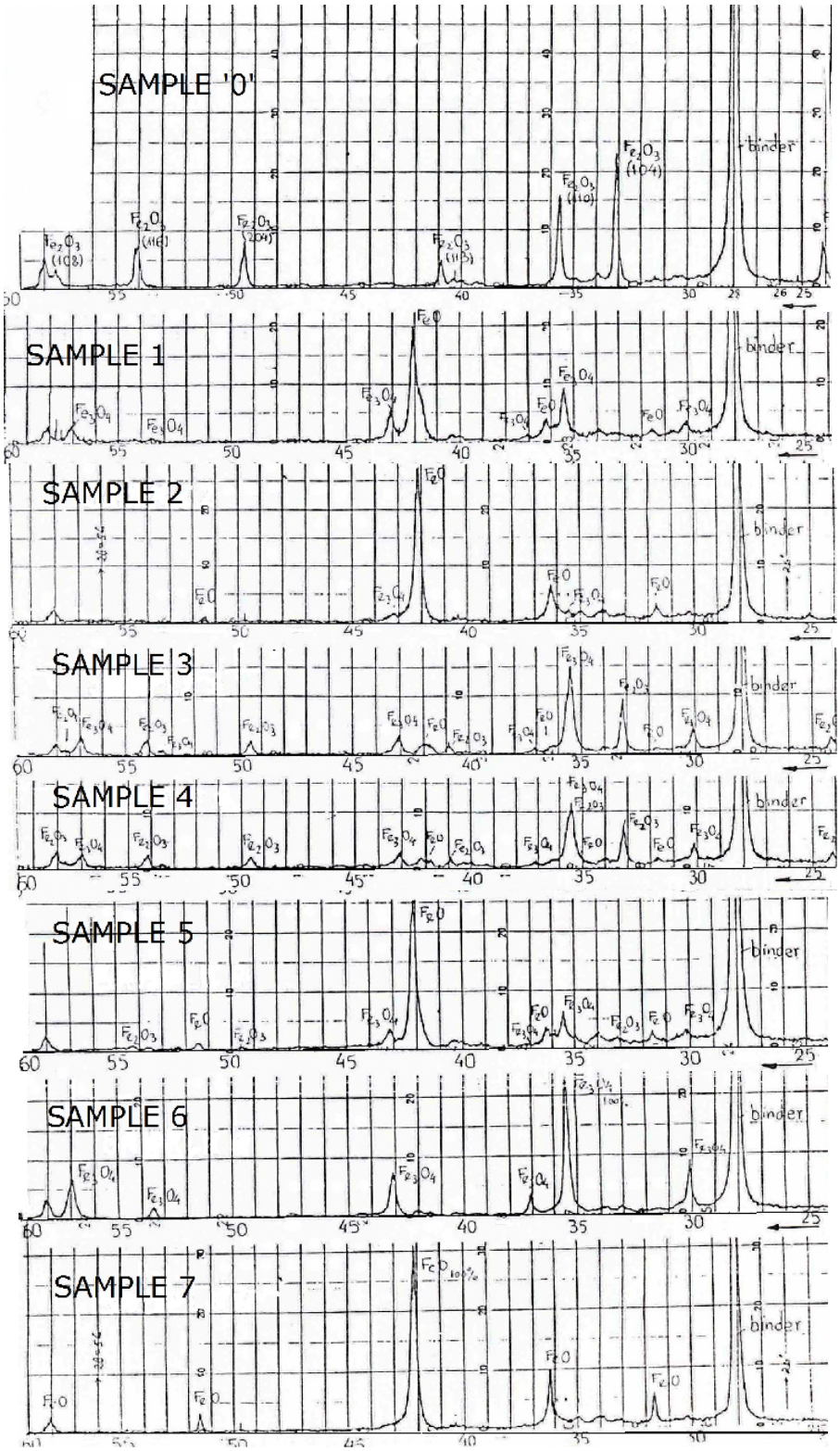

Fig. 1. Structural analysis by XRD

The qualitative XRD analysis was performed by the normalization method and comparison with ASTM files. The diffractograms for eight analysed samples was shown in Fig. 1. The XRD quantitative analysis was performed by using concentration versus intensity calibration curves. The standard error was less than 2%. The obtained concentrations was presented in Table 3.

Table 3. The results of XRD quantitative analysis.

Sample number	FeO [%]	Fe_3O_4 [%]	Fe_2O_3[%]	Impurities [%]
1	54-55	40	5	<1
2	82	12-14	<2	<1
3	5-7	55	38-40	<1
4	5-6	25-30	60-65	<1

3. CONCLUSIONS

From the results shown in Table 2 it can be concluded that the heating atmosphere from the deep furnace has an oxidant character that leads to the forming on the ingot's surface of a 6-12mm, rich in FeO scale layer. This type of scale is released at the 950mm stand. After this rolling step the billets are oxidized in contact with the environment atmosphere, a fact evidenced by the increasing of the Fe_2O_3 content. Because the total rolling time has a magnitude of tenths of minutes, the oxide quantity formed in this time is very small. The billet's oxidation is not significant by quantity, but is important from the decarburization point of view. The layer scale obstructs the decarburization process on the rolling train. Thus, the observed decarburized layers arise only from the heating in the deep furnace.

We propose a procedure to obtain a smaller decarburized layer by using a more oxidant atmosphere, because at 1100^{o}C the oxidizing rate is higher than the decarburizing rate. A more oxidizing atmosphere can be obtained by increasing the air consumption coefficient α. The oxidation process can be controlled by increasing the temperature rate in the 800-1200^{o}C interval and by reducing the furnace temperature to 850-1000^{o}C in the case of the rolling steps. The scale resulting from the rolling process is usually recycled in blast furnaces. Also it is possible to recycle the scale in electric furnaces. For this reason it was proposed the following preparation of the scale: milling and heating in an inductive furnace at 1000-1100^{o}C with coke and limestone.

At 1000^{o}C the coke reacts with oxygen:

$$2C + O_2 \rightarrow 2CO + 52.8kcal$$

and the iron oxides will react with CO:

$$Fe_3O_4 + CO \leftrightarrows 3FeO + CO_2$$
$$FeO + CO \leftrightarrows Fe + CO_2$$

After that, the temperature is increased to 1100-1200^{o}C was obtained iron which can be used in electric furnaces.

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