Characterization and Microstructural of Hot Rolling Mill Scale

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Abstract— **In the IMETAL-El Hadjar complex, during the steel manufacturing process, a significant amount of scale is produced on the surface of slabs and billets of hot-rolled steel. In the various rolling mills, the quantity of scale produced is estimated to be about 0.1% of the annual production of the steel complex. The quality of the thin steel sheet during the rolling process is affected by the behavior of the iron oxide layers formed on their surfaces (scale).**

This amount of scale which is a fatal by-product of the forging and rolling processes can be reused in certain areas and applied by appropriate recycling techniques such as agglomeration of iron ores in the blast furnace as a raw material. This aspect of recycling is taken into consideration.

The objective of this study is to identify the microstructural properties of the scale, using different analytical methods such as X-ray diffraction and scanning microscopy.

Several samples were used for characterization of the locally produced scale.

The analyzes of the results given by SEM showed that hematite and magnetite, the main phases present in the scale, are stacked in thin layers of a magnitude of the order of a micron meter. These phases of iron oxides are confirmed by the analysis of the spectra given by the X-ray diffractometer.

The presence of silicon in the scale is due to the covering powder used on the wall layers of the ladles.

Key words: **Mill scale, Oxidation, Hot rolling, Microstructural properties, SEM, X-ray.**

I. Introduction

Until the last decade, the scale, slag, dust and sludge generated by integrated steel plants was called waste, but now this term has been replaced with by-product and sometimes product due to intensive re-utilization of these materials. The management of all these substances generated in steel plants has become an important issue due to ever-tightening environmental regulations.

Mill scale is one of these materials produced in the processing of steel during continuous casting, reheating and hot rolling operations. The scale formed during these operations is removed by water sprays and then mill scale is accumulated as a by-product in all iron and steel companies, either integrated iron and steel companies or mini steel mills and small mill shops.

Many researchers have found the structure and the thermal properties of scale formed during manufacturing of carbon steels were realized for a long time [1].

However, the research in this field was always actively studied by numerous specialists, in particular those of the steel industry [2].

In the steel-making of IMETAL El-Hadjar complex, the flat products are hot-rolled. The quality of the thin sheet steel during the process of rolling is allocated by the formation of the layers of scale on the surface of the sheet steel to the cost all the operation of deformation along the hot rolling mill process. To minimize his growth inside the furnace we have to respect certain necessary conditions among which the content in smokes which has to be between 1,5- 2,5 %.

In the high-temperature heating of steel in pusher furnaces operating under traditional conditions with excess oxidant (usually air with n = 1,1-1,3), three layers of oxides are formed on the surface of the steel.

The iron content is around 67%, a large part of the scale is under magnetite and hematite, namely the oxygen content in the furnace. A certain amount of magnetite crystals will be present in the wüstite.

The degree of oxidation of the steel depends on the temperature, the chemical composition of the metal and the gas composition [1].

The steel where our scrap is collected is an A9 type extra-soft steel (internal nomination) according to the french standard AFNOR NFA 36-150 for packaging called black iron, tinplate, the grade reached after cold rolling and tinning is T57.

II. Materials and Method

The steel intended for the called packaging tin plate is defined by the standard NFA 36-150.the grade after cold rolling and tin plating is T57. The chemical composition of hot rolled low-carbon steel A9 is represented in Table 1 below.

TABLE I. Chemical analysis of A9 steel

Eléments	**C**	**Mn**	**Si**	**S**	**P**	**Al**	**Fe**
%	0,09	0,35	0,04	0,025	0,025	0,05	Balance

The mill scale picked up by the hot rolling mill has a grey color. Its chemical composition is identified by by SIEMENS SRS 3000 FRX analyzer. The obtained results are represented in the table 2.

TABLE II. Chemical analysis of the scale by FRX

Eléments	**Fe**	**FeO**	**Fe_2O_3**	**Fe_3O_4**	**MnO**	TiO_2	SiO_2	CaO	Al_2O_3
%	7,04	9,09	20,22	29,31	0,36	0,05	0,34	0,05	0,03

The optical microscopy and scanning electron microscopy analyzes are carried out on calamine scrap collected from the hot rolling mill and on polished surfaces of the same sample using ZEISS EVO-MA 25 scanning electron microscopy. Driven by integrated software where one can acquire micrographic results and chemical analysis by EDS.

The cooling system ensures proper functioning of the components so as to ensure a secondary vacuum, a 99.99% pure nitrogen bottle is connected to the equipment.

The principle of scanning electron microscopy is based on electron - matter interaction. The incident electron will interact with the nucleus, the more bound electrons of matter and the electrons of the outer layers [2, 3].

Secondary electrons result from an inelastic interaction of the electrons incident with the material. They come from a depth between 5 and 10 nm and are easily detected.

From the surface layers of the sample, secondary electrons are very sensitive to surface variations, as each variation will change the amount of electrons collected. These electrons give us topographical information of the sample analyzed.

Backscattered electrons are electrons resulting from the interaction of primary beam electrons with nuclei of atoms in the sample.

III. Results and discussion

The micrographs taken by scanning microscopy and for different magnifications show the microstructural appearance of laminated iron oxides in layers that are more or less large according to the phases and their distribution over the layer of scale formed.

The faster oxygen scattering by the thin iron surface layer than the rate of oxygen supply by the interface reaction was assumed to be crude will result in a lower oxygen potential at the interface of magnetite-external hematite fig 2 and fig 4. Thus providing a motor element for the iron to spread through the hematite layer. The inner wüstite -iron interface has become corrugated initially; then with the rapid advance of some protruding sections, some parts of the wüstite layer were reduced by first and ultimately the wüstite holding of the islands was reduced to complete the reduction process. Porosities were produced when Wüstite islands were reduced due to localized volume shrinkage. Higher oxygen concentrations in the underlying areas of the near-edge samples were assumed to be responsible for their slower reduction rates than those at the center [7,8].

The pure iron scale is formed by three layers of superimposed oxides, corresponding to increasing levels of oxidation from the metal to the atmosphere, the layer of FeO protoxide against the metal, then the layer of magnetite Fe_3O_4, then a very thin layer of Fe_2O_3 hematite on the surface, these phases were confirmed by X-ray diffraction Fig 1.

The protoxide is stable only above 570 ° C. Below this temperature it decomposes into magnetite and iron. If the cooling is very fast, it does not decompose. If the cooling is very slow, it is completely decomposed. If the cooling is done at medium speed, only the part of the protoxide layer which is on the magnetite side is decomposed.

The mill scale of ordinary or low alloy steels have the same constitution but they may contain additional phases due to the presence of the elements of addition or impurities.

The micrographs taken by scanning electron microscopy and for different magnifications show well which are synonymous with a removal of unstable oxides in small quantities, in contrast to those of iron oxides layered in laminations [6].

The pores and bubbles are also seen in the scale, especially on an uneven surface.

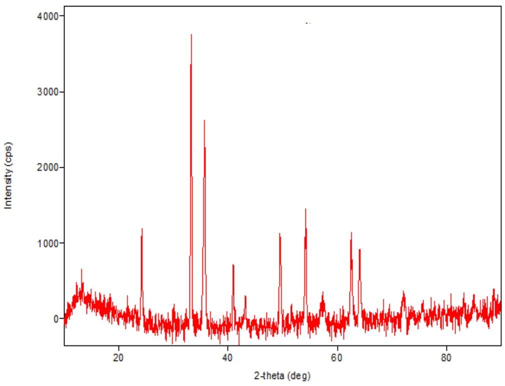

Fig. 1 X-Ray diffraction results

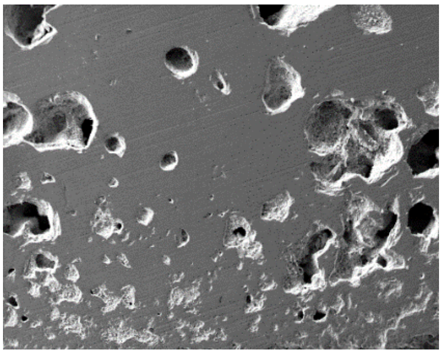

Fig. 2 SEM micrographs showing obviously cluster's areas which magnetite and hematite are present.

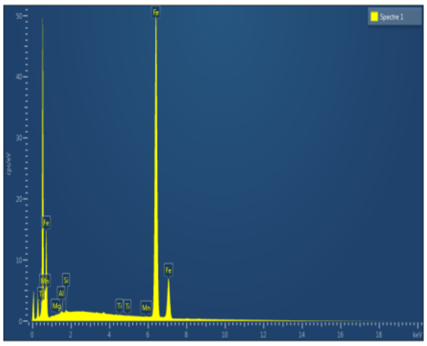

Fig. 3 EDS of crude mill scale, shows the presence of silicon in large quantities in preferential areas.

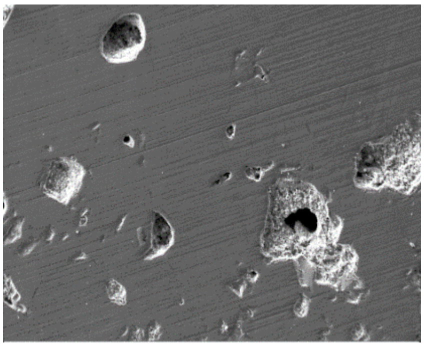

Fig. 4 SEM micrographs showing obviously cluster's aereas witness the presence of silicon.

IV. CONCLUSION

These oxides are confirmed with X-ray technique which their shape and dispersion were been obviously seen in SEM with different magnificant.

The chemical analysis shows a total iron of the order of 67% with other oxides in trace.

The presence of silicon was confirmed by X-ray analysis and the most phases common are magnetite and hematite and the SEM micrograhs corroborate their allocation.

The presence of silicon which confirmed by EDS analysis.

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