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Influence of the Si content in steels on their mechanical and chemical behaviors. Part 1: Properties in compression, hardness and high temperature oxidation

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ABSTRACT

Most of steel products are available as sheets, therefore shaped by cold- or hot-rolling. Such processes induce plastic deformation. On the one hand this is more or less difficult to achieve, this depending on the microstructure or simply on the chemical composition of the steel, notably concerning the more common elements present in steels: carbon and silicon. On the other hand the same elements also act on the behaviour of the hardened steels in high temperature oxidation in the case of hot working, and on room temperature corrosion in aqueous milieu in both families of fabrication routes. In this work the effects of the silicon contained in a simple ternary steel on its formability and on its high temperature and room temperature chemical properties will be characterized. In this first part, four ternary steels with different silicon contents (0.1 to 0.25w%) were elaborated by foundry, plastically deformed in compression, then subjected to high temperature oxidation as this can be encountered on the steel sheet surface during the last cooling fabrication stage.

KEYWORDS

Steels; hot-rolling;
Plastic deformation;
High temperature oxidation;
Silicon content.

INTRODUCTION

In most cases steels are available as semi-finished products, for example as flat products obtained by plastic deformation[1,2], an operation inducing not only changes of shape but changes of morphology for the initial grains resulting from solidification (outer columnar and inner equiaxed crystals[3]). Indeed the grains become elongated and much richer in dislocations (this resulting in hardening). The laminated products may result from shaping at ambient temperature by cold-rolling, or at more or less high temperature by hot-rolling[4]. At each shaping step cooling and lubrication are ensured by a mix of water and oil. Since metallic alloys are generally threatened by oxidation especially at high temperature[5-7], staying at rather high temperature in wet air necessarily induces oxidation of the surfaces of the flat steels which goes on during cooling and coiling, influenced by the presence of water vapor[8,9]. The oxides formed on surface and eventually in the sub-surface may be sources of defects for the final products[10,11]. This is one of the numerous reasons why many studies have been carried out on the mechanisms and kinetics of oxidation of low-alloyed steels in this con-

text ([12] for example), either in plant on industrial products or in laboratory by reproducing phenomena. The high temperature oxidation of such low-alloyed steels is generally rather fast since these ones are the most often totally chromium-free and moreover present simple chemical compositions (with at least carbon and silicon). In addition the rate of hot oxidation of these steels maybe influenced by the rate of plastic deformation previously endured by the steel products.

This work deals with the oxidation phenomenon of flat carbon steels during the cooling when already coiled. If the special surface cleaning has removed all oxidation/corrosion products just before coiling (often by hydro-mechanical descaling) oxidation continues a lot during the cooling of the coil. This oxidation is due to the wet air trapped between two successive sheets until temperature becomes low enough or until the partial pressures in oxygen and in water vapor have sufficiently decreased. This new oxidation may generate surface oxides which can be possibly detrimental for the properties of the sheets.

For a given complex atmosphere (hot air + water vapor coming from the previous hydro-mechanical descaling step) the amount of the resulting oxidation products may depend on the chemical composition of the carbon steel (notably silicon) and maybe on the compression deformation rate.

In this study it was wished to better know the influence of the silicon content (at fixed carbon content), for steel samples after plastic deformation, on the high temperature oxidation kinetic of such steels in a given atmosphere typical of industrial ones.

EXPERIMENTAL DETAILS

Elaboration

Steels destined to be manufactured as coils are often extra low carbon ones, and the contents in some of the other elements possibly present in the chemical composition are rated to achieve specific mechanical properties: this is for example the case of silicon. Thus, the steels considered for this study are very simple ones – ternary Fe-C-Si – with 0.01 weight percent of carbon and containing silicon with different several contents. Four steels were elaborated by foundry way by targeting the following chemical compositions: Fe-1Si-0.01C, Fe-2Si-0.01C, Fe-2.2Si-0.01C and Fe-2.5Si-0.01C (in weight percents) and formed as ingots of about 40g. They were synthesized from pure elements: Fe (Alfa Aesar, purity: more than 99.97%), Si (Alfa Aesar, 99.999%) and graphite. Fusion and solidification were both achieved in the water-cooled copper crucible of a high frequency induction furnace (CELES) and under 0.3bars of pure Argon (<3ppm O_2), with an intermediate 3 minutes – long dwell. The operating parameters of the furnace were 115 kHz and 4kV.

They were cut in order to obtain:

- in the one hand parts to prepare metallographic samples destined to control the metallographic health and microstructure by optical microscopy as well as X-Ray analysis,
- in the second hand parallelepipeds having the following approximative dimensions: 8mm × 8mm × 4mm and destined to the oxidation tests with preliminary deformation in compression.

Compression runs

Before becoming coils the steels are several times plastically deformed at temperatures of several hundreds degrees, this resulting in final very high ratios of thickness. Such deformation ratios were not envisaged in the present study but the possible effect of plastic deformation, what was wished to be examined here, was considered for much lower deformation with comparison of results with the same ternary steels characterized in both hardness and high temperature oxidation.

A part of the previous samples were uni-axially plastically deformed at room temperature. As previously done on a Fe-0.4wt.%C steel[13] before corrosion testing[14] this was done using a MTS RF/150 universal testing machine equipped with a force-measuring cell (capacity: 150kN) and of compression platens. Additional parts with parallel faces made of hard metals were placed between platens and samples in order to prevent any damage for the first ones. In each case the maximal stress to reach was chosen high enough to be sure to exceed the yield stress of the sample and to obtain a significant rate of plastic deformation (typically at least -20% of permanent deformation). Because of irregular lateral deformation the compressed samples were cut again in order to rediscover a parallelepiped allowing an easy calculation of surface and also, more

important, guaranteeing to have a whole lateral surface which can be considered as plastically deformed homogeneously in terms of rate and orientation.

Hardness measurements

The four alloys were subjected to Vickers indentation (load: 10kg), in their as-cast condition as well as in their compressed state. Three indentations were done on each sample (deformed or not) and the results led to the calculation of an average value and a standard deviation one.

High temperature oxidation tests

The oxidation runs were performed using a Setaram Setsys Ev 1750 TGA thermobalance equipped with a WETSYS vapor generator, according to the following thermal program:

- Heating from room temperature up to 700°C at +20°C min-1 under an inert atmosphere of pure argon
- Heating from 700°C to 750°C at +5°C min-1 under an inert atmosphere of pure argon
- Isothermal dwell for 10 minutes at 750°C in order to thermally stabilize the apparatus (still under argon)
- Switch to humidified air (14.5 g H_2O per kg of dry air) and isothermal oxidation during three minutes
- Switch to pure argon again and slow cooling at -5°C min-1 down to ambient temperature.

These oxidation tests were carried out on one sample of each alloy in the compressed state.

The samples were subjected to X-Ray Diffraction to characterize their oxidized surfaces to specify the oxides formed during the preceding oxidation tests. This was done using a Philips X'Pert Pro diffractometer (Cu K_α, λ = 1.5406 Angströms).

They were thereafter first coated by a thin layer of gold deposited by cathodic pulverization to provide a conductive surface necessary for the subsequent electrolytic nickel deposition. The later one was realized by cathodic reduction of Ni^{2+} ions in a Watt's bath heated at 50°C (during at least two hours for a current density of 16 mA/dm²). The nickel thickness obtained with these parameters generally allows a sufficient protection of the oxidized surface for keeping the external oxides during the cutting which is necessary for cross-section examination.

Metallography

The as-cast and compressed steel samples for the metallography examinations were imbedded in a cold resin mixture (manufacturer ESCIL: resin CY230 + hardener HY956). After that, the mounted samples were polished with SiC papers (from 80 to 1200 grits) and with 1µm diamond sprayed textile disk, until reaching a mirror state.

The metallography samples were investigated using X-Ray Diffraction (XRD) and Electron Probe Microanalysis (EPMA).

RESULTS AND DISCUSSION

Compression runs and hardness

The compression curves are presented in Figure 1 all together in the same graph. It appears first that the curves obtained for the two steels containing the two medium silicon contents are close to one another, what is rather logical since they contain almost the same silicon content. The compression curve obtained for the steel with the lowest Si content is significantly under the two former curves while, in contrast the compression curve corresponding to the highest Si content is sensibly above the ones obtained for the two steels with 2.0 and 2.2wt.%Si.

On the same curve one can also see that the loss of linearity seems occurring for a stress increasing with the silicon content in the ternary steel. This can be more

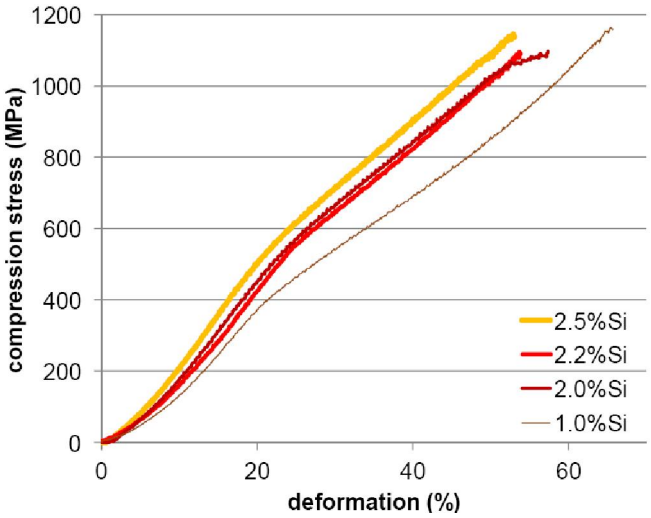

Figure 1 : Strain-stress curves acquired during the compression tests applied to the four steels

efficiency evidenced in Figure 2 in which the yield stress determined on these curves are directly plotted versus the silicon content. One can see that yield strength obviously increases with the silicon content, from about 380MPa up to about 490MPa.

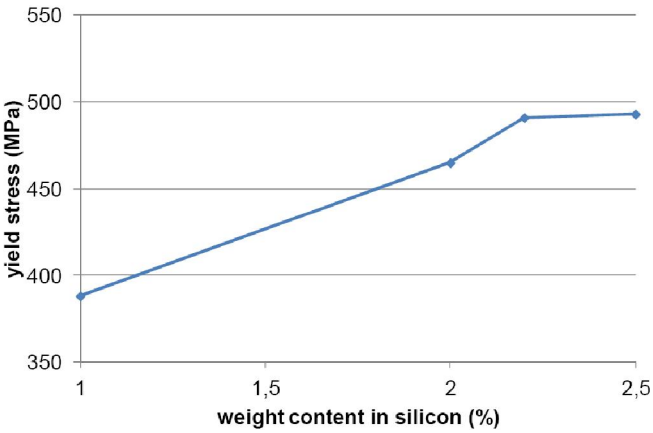

Figure 2 : Evolution of the yield stress versus the silicon content in the steel

The thickness losses were not red on the former compression curves since the precision was not high enough (and even not really known) since no extensometer was available during these compression tests. The deformations were simply measured after test on the compressed samples using a numerical caliper. Thus, only the plastic deformation was determined (no data about the maximal elastic deformation). The values of these permanent deformations are listed in TABLE 1. No comparison can be done by considering the silicon content since the compression tests were not all finished in the same conditions (notably in terms of maximal stress applied). These values are only informative about the hardening of the four samples. Since the four deformations are not really far from one another it will be thereafter considered that the obtained hardening is sensibly the same for the four steels, even if the plastic deformation rates vary between 22 and 34%.

These compression runs induced for the four steels a hardening which can be revealed by the induced increase in hardness. The results of Vickers indentation are graphically presented in Figure 3. On the blue "as-cast" curve one find again the strengthening of the Si-containing Fe-0.01C steels when their silicon contents increase (from about 120Hv10kg for 1 wt.%Si to about 180Hv10kg for 2.5 wt.%Si), confirming the effect on yield strength observed above. Compression induced also an increase in hardness for the four steels, of about 70-80Hv10kg for all. This allows considering that the compression runs brought similar hardenings to the four steels.

TABLE 1 : Amount of plastic deformation obtained by compression on the four ternary Fe-0.01C-xSi steels

Si content in steel (x)	1 wt.%	2 wt.%	2.2 wt.%	2.5 wt.%
Plastic deformation	- 33.63%	- 24.28%	- 29.09%	- 22.55%

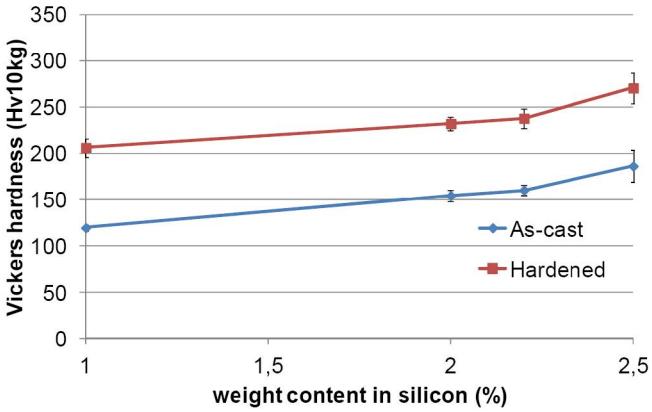

Figure 3 : Curves of hardness variation illustrating the strengthening and hardening effects of the silicon content and of the compression tests

High temperature oxidation runs

The mass gain curves obtained for the four steels are plotted together in Figure 4 (full scale) and in Figure 5 (zoomed on their first parts). It is obvious that the global oxidation rate strongly depends on

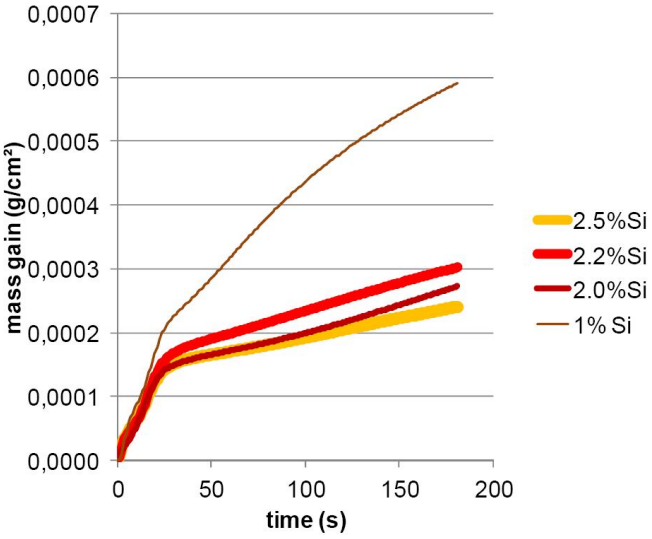

Figure 4 : Mass gain curves obtained for the four steels (full scale)

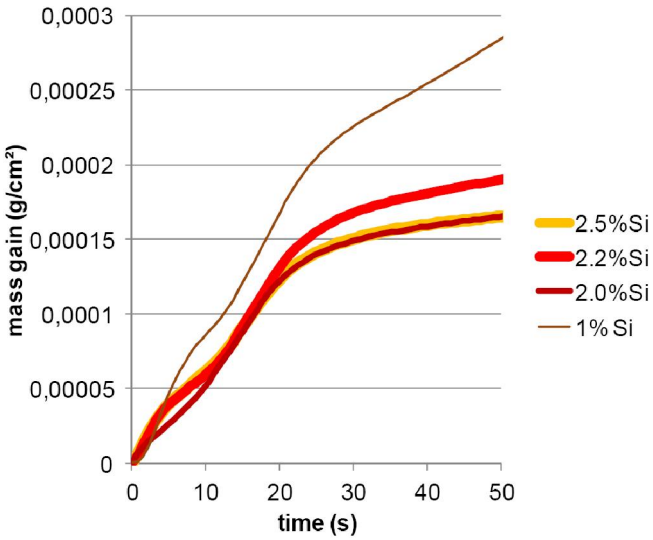

Figure 5 : Mass gain curves obtained for the four steels (zoomed on the linear first part)

the silicon content in the steel: the steel with the lowest silicon content oxidized much faster than the three other ones the Si content of which are twice and more (Figure 4). This is already also true for the first tens of seconds (Figure 5) when oxidation is particularly fast (and more or less linear) before that it decelerates. One can notice in addition that this first linear fast mass gain finishes about five seconds later (at near t=25s) for the lowest Si steel than for the three high Si steels (near t=20s). As natural consequences, the mass gain observed at the end of the linear first part and the total one achieved at the end of the isothermal stage are both significantly higher for the steel with the lowest silicon content than for the three other steels, as evidenced on the graphs presented in Figure 6 and Figure 7 respectively.

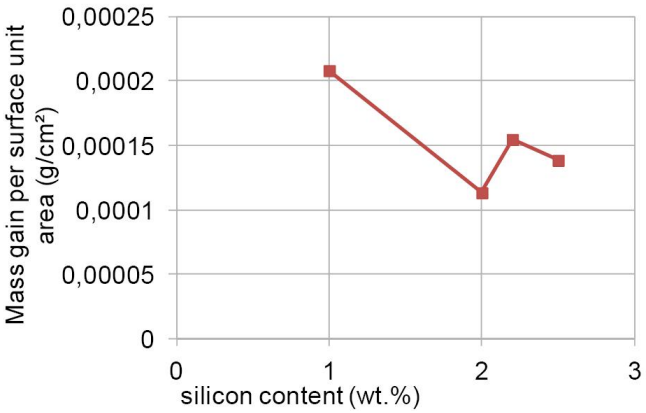

Figure 6 : Evolution of the mass gain observed at the end of the first linear oxidation part

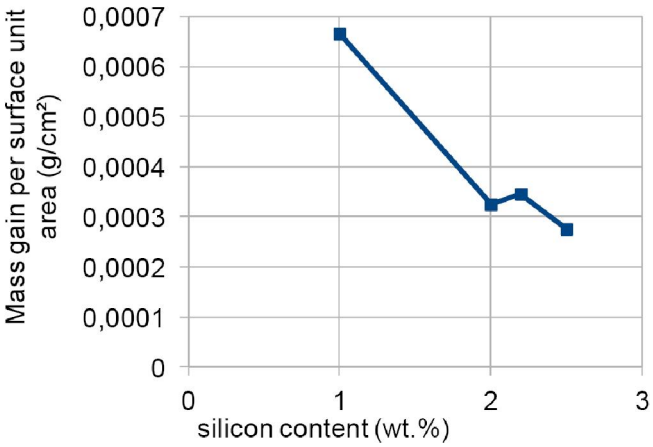

Figure 7 : Evolution of the total mass gain achieved at the end of the isothermal stage

The oxidized surfaces of the four samples were subjected to X-Ray Diffraction analysis. The obtained results are illustrated by two examples corresponding to the steels containing the two Si extreme silicon contents studied here: 1wt.%Si (Figure 8) and 2.5wt.%Si (Figure 9). Only one oxide was obviously present, i.e. present in quantities high enough to be really detected by XRD: hematite Fe_2O_3. Indeed no oxide involving Si was revealed. The other peaks are to be attributed to the steel itself (ferritic iron), which was logically also solicited by the radiation because of the too thin oxide scale formed dutring the rather short exposure in hot air during the whole oxidation test.

General commentaries

In this first part of the work carried out for ternary carbon steels which differ from one another about their Si contents, the increasing applied stresses recorded during compression evidenced the strengthening effect of silicon for such simple steels, as this is commonly observed for more complex industrial carbon steels. Hardness measurements brought furthermore additional data about this increase in mechanical resistance with silicon. These two properties modifications are the results of the hardening induced for the four steels by plastic compression, dislocation's structure modification which may influence the chemical reactivity of these steels. Here only the behavior in oxidation at high temperature was examined, in a combination of conditions allowing more or less reproducing the usual industrial context of hot-rolling followed by packaging. The test performed on the hardened steels using a thermo-balance led to

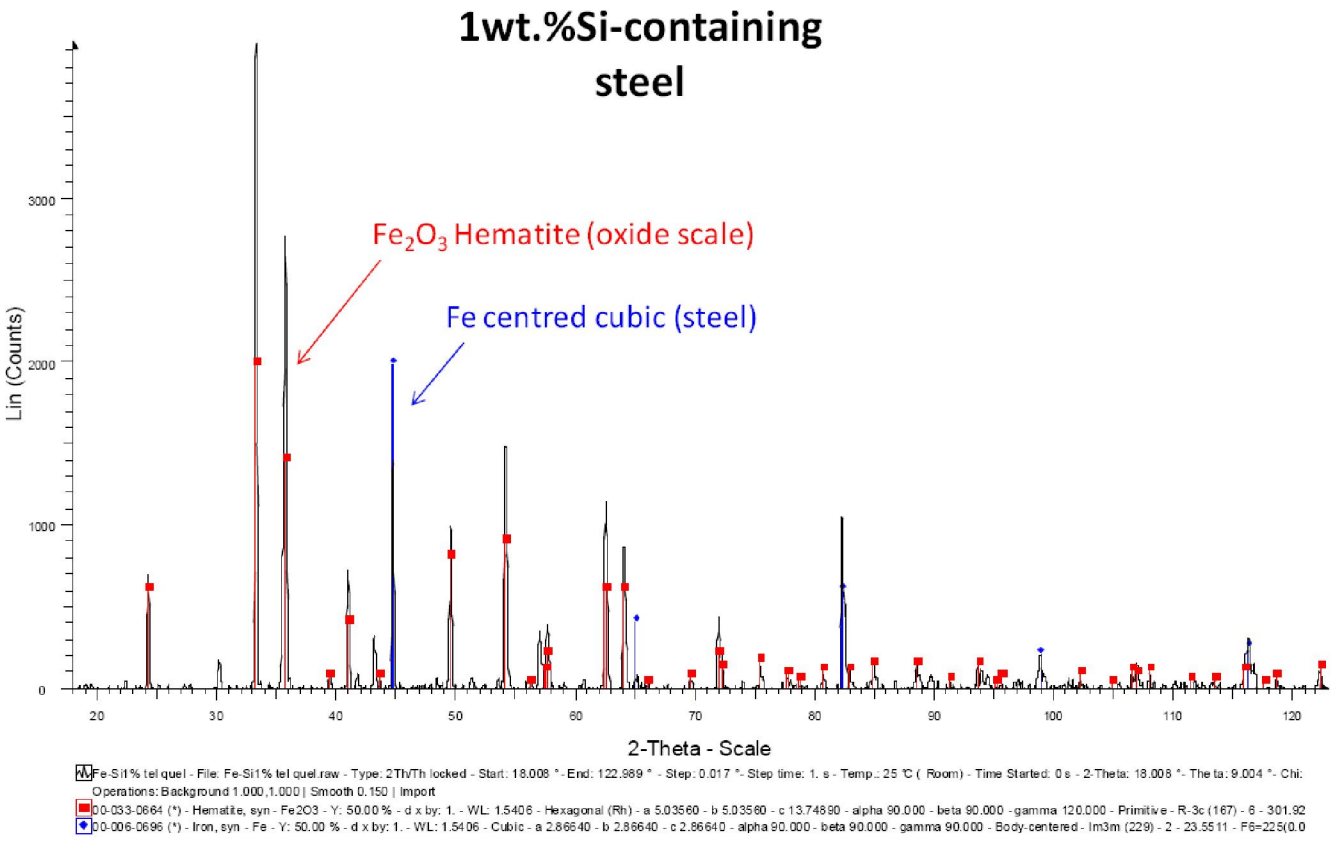

Figure 8 : The XRD Spectra acquired on the oxidized surface of the 1wt.%Si-containing steel

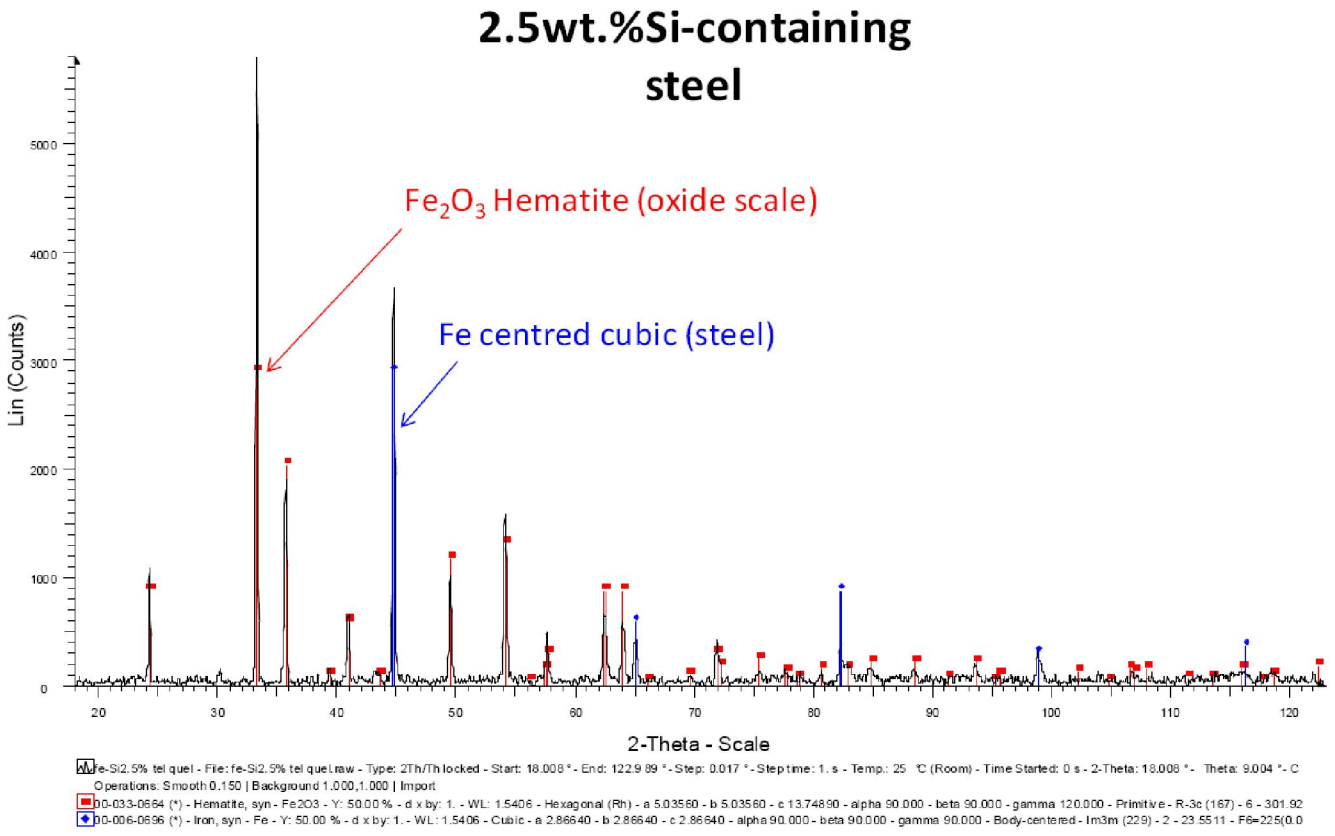

Figure 9 : The XRD Spectra acquired on the oxidized surface of the 2.5wt.%Si-containing steel

first interesting observations about the kinetic of mass gain, with the evidencing of two steps, a first one of fast oxidation and a second one of slowed oxidation. It appeared that the passing from the first stage to the second stage seems occurring a little earlier for the high Si steels by comparison with the steel with the lowest silicon content, while the mass gain achieved during the first stage as well as during the second stage were both lower for the {2.0 to 2.5 wt.%Si}-containing steels than for the {1.0wt.%Si}-containing one. Silicon appears as a beneficial element for limiting the surface oxidation of the sheet steel rolled during the cooling of the coil, then to diminish the defects risk, this although no participation of Si to the surface oxides was revealed by XRD.

CONCLUSIONS

The four steels studied here, displaying carbon and silicon contents typical of the chemical composition of sheets of carbon steels shaped by hot-rolling, clearly showed a beneficial effect of the presence of silicon - or of a higher content in this element – for the surface or sub-surface defects of sheets. Although that the hardening preliminarily brought by room temperature compression to the simple steels studied here was far lower than the one really achieved in industry, one can think that this tendency will be probably still true for much higher deformation rates achieved in hot conditions. Knowing that corrosion by condensed water enriched by other substances keeps on deteriorating steels after completed cooling, these steels were also subjected to corrosion study in aqueous milieu using different electrochemical techniques. The results and comments of the additional room temperature corrosion tests will be presented in the second part of this work[15].

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