

Identification of undeformed/deformed FeSi oxides scales by EBSD

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Summary

Oxide scales growing during hot rolling of steel represent an industrial and environmental problem. They affect the final surface quality and the response to downstream processing. Characterizing scale layers and the scale/steel interface in terms of phase morphology, texture, grain structure, chemical composition and the effect of thermomechanical cycles on the material response to further processing is fundamental for a better understanding of their behaviour.

The structure and composition of oxide scale in 2.4 m.-%Si electrical steel has been investigated in the present work. The oxidation temperature region of 1100°C up to 1250 °C for different oxidation times was particularly focused on.

Results showed that EBSD appears to be a powerful tool for differentiating the different phases of classical oxide scale layer. Measurements allowed an accurate identification of the different oxides phases. It was observed that a very rapid build-up of oxide occurs when a liquid phase, due to the wustite-fayalite eutectic, is present in the surface of the steel.

Introduction

The influence of silicon on the scale structure and behaviour depends on the temperature range under consideration (see Fig. 1). Below 1177°C, FeO reacts with SiO₂ to form fayalite (Fe₂SiO₄) at the metal/scale interface, and like the spinels, this decreases the overall oxidation rate. At 1177°C, a eutectic reaction takes place between FeO and Fe₂SiO₄ leading to the formation of a liquid phase, associated with catastrophic oxidation kinetics. In addition, the liquid phase preferentially attacks the metal grain boundaries, making the scale highly adherent. This can lead to the creation of defects known as "embedded scale" during hot rolling [1, 2, 3].

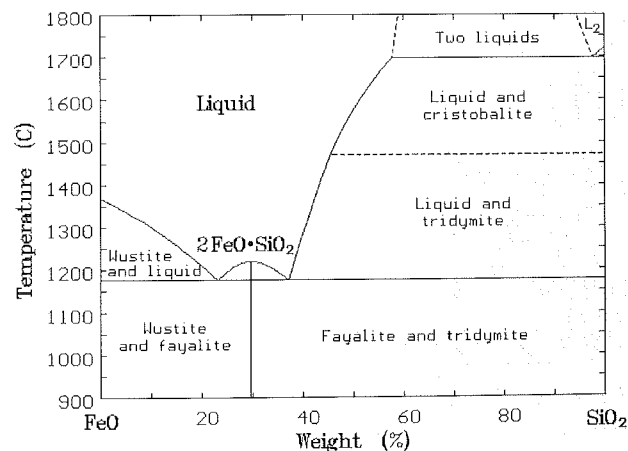


Figure 1. FeO-SiO₂ binary system [1].

The oxides involved include SiO₂ which forms a silicate, Fe₂SiO₄, and FeO, Fe₃O₄ and Fe₂O₃. At low silicon contents, SiO₂ is formed at the alloy surface, distributed very finely. Simultaneously, it reacts with FeO to form fayalite, Fe₂SiO₄, and, when the SiO₂ particles become larger, they are engulfed by the scale. These islands lie in the FeO layer as markers but do not give an accurate indication of the position of the original metal/scale interface. Fayalite particles are often aligned parallel to the surface [1, 4].

Many scientific papers about Si-containing steels have focused on the occurrence of red scale, ranging the Si content from 0.2 to 11.5 m.-% [1, 5, 6, 7]. It was found that the formation of red Fe₂O₃ is accelerated by incomplete de-scaling of FeO. In Si-containing steels, de-scaling loses efficiency due to the appearance of the eutectic compound FeO-Fe₂SiO₄ at the steel interface. After solidification it leads to a strong adhesion of a part of the scale to the steel. The oxide growth rate then depends on the oxidation temperature and in the amount of silicon of the steel, while being reduced by silicon diffusion and the formation of fayalite

Insufficient attention has been paid in research to one of the most important structural factors directly relating to the behaviour of steel surfaces as they oxidize, i.e., their crystallographic texture. The phase constitution, structure and texture of rolled surfaces largely determine the effectiveness of the descaling process in hot strip mills; they

influence the formation of defects during subsequent cold rolling and are in many cases directly related to specific properties of the steel [8]. First results on microstructural and microtextural characterization of oxide scale using EBSD have been recently reported [4, 8, 9, 10]. They succeeded in differentiating between different phases in a classical oxide scale layer. Because of some problems in optimization of the differentiation between wustite, magnetite and iron (cubic phases in all cases) they found sometimes quite low confidence index (CI) or high mean angular deviation (MAD) values in the raw data for some alpha iron grains but nevertheless clear layers containing alpha iron, wustite, magnetite and hematite can be distinguished.

In this work the structure of scale in 2.4 m.-%Si electrical steel has been investigated. Special attention was paid to the formation of fayalite. The temperature region of 1100°C up to 1250°C was particularly focused on. The goal of the present work is to study the oxide formation and its deformation behaviour under controlled laboratory conditions by means of the EBSD technique.

Experimental

Cylindrical specimens Fe 2.4 m.-%Si were heated up to the oxidizing temperatures of 1250 and 1100 °C for 10 and 60 s. In order to assess the deformation behaviour of the thermally-grown oxide scales, uniaxial compression tests were carried out in computer-controlled hot forging simulator. Deformation experiments were performed at 900 and 700°C and 1 s⁻¹ of true strain rate, to a true strain of 0.22, after deformation the samples were cooled in air atmosphere.

Metallographic samples were prepared following the procedure described elsewhere [4]. Microstructure, microtexture and phase identification of the oxide layer were performed with a Zeiss Ultra Plus, Field Emission electron microscope, equipped with an EDS-hkl[®] EBSD system. The samples were tilted 70°, and the accelerating voltage was 20kV. The chosen available phases (alpha iron, fayalite, wustite, magnetite and hematite) were detected using 32 reflectors, band edges, standard divergence, 5 minimum and 7 maximum bands and 60 for resolution of the Hough Space.

Results and discussion

EBSD measurements allow an accurate identification of the different oxides phases. Nevertheless, it is not possible to statistically evaluate their texture: magnetite phase displays similar texture components than the wustite parent grain.

The EBSD Band Contrast maps, presented in Figure 2, reveal the low Kikuchi pattern quality of the fayalite, which is not always possible to be indexed, in comparison with the other oxides. It is not clear if the cracks that appear along the fayalite phase (parallel and perpendicular to the iron surface) are caused by the stresses developed during oxides growth or are produced while the samples are cooling down to room temperature.

Examination of samples oxidized at 1250°C shows the occurrence of various layers and structures (as expected for such range of temperatures where the wustite-fayalite eutectic temperature of 1177°C is superpassed): i) fayalite in contact with the iron substrate, followed by ii) a thick layer of wustite and iii) on top of it a thinner layer of magnetite, as shown in Figure 2 a. The layer of hematite might be lost due to the thermal contraction during cooling or while sample preparation. The thick layer of wustite shows particles of magnetite (formed during the solid state decomposition of wustite into magnetite and iron) and fayalite (located at the wustite grain boundaries).

Samples oxidized at 1250°C for 60 s shows a higher degree of wustite decomposition to magnetite and a much more rougher iron-oxide interface than the ones oxidized for 10 s, see Figure 2 d and e.: small rounded wustite and magnetite grains are uniformly distributed inside the fayalite layer, being the magnetite grains much more closer to the iron substrate (they show higher Band Contrast so their colour is lighter than the one of the fayalite).

Samples oxidized at 1100°C show a thinner layer of oxides made of fayalite and a mixture of iron oxides. The thickness for samples oxidized at 1100°C is approximately 20 µm, which is much lower than for samples oxidized at 1250°C (200 µm), due to the slower oxidation kinetics (growth of the oxide is strongly accelerated when the liquid phase of fayalite is present). Most of the formed oxides at 1100°C peel off, while it adhere for samples oxidized at 1250°C. The iron-oxide interfaces are fairly smooth, so the measurements only reveal the presence of the magnetite and a layer of hematite on top of it (see Figure 2 c). Fayalite layer is not adherent to the substrate (as the one formed at higher temperatures).

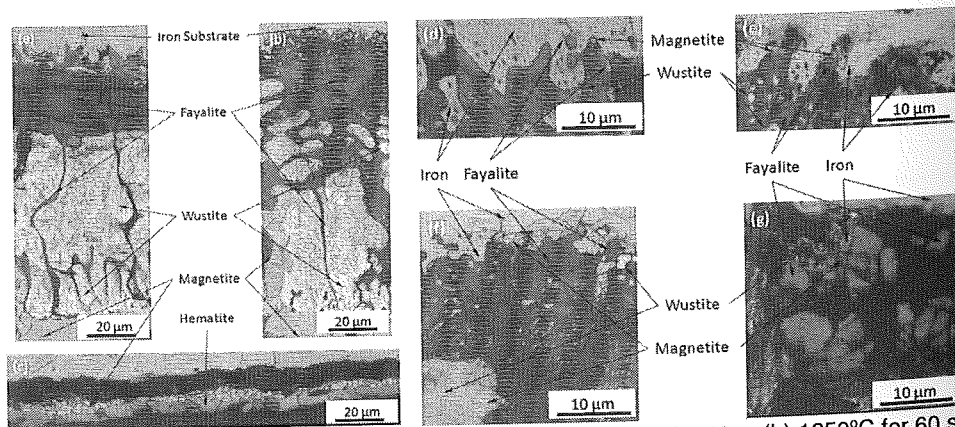


Figure 2. Band Contrast maps of oxidized samples, (a) 1250°C for 10 s, (b) 1250°C for 60 s, (c) 1100°C for 10 s, detailed view of the iron-oxide interface for oxidized samples (d) 1250°C for 10 s, (e) 1250°C for 60 s and samples oxidized at 1250°C for 10 s and deformed at (f) 900°C and (g) 700°C.

Samples oxidized at 1250°C during 10s and deformed at 900 and 700°C (see Figure 2 f and g) show subgrain microstructure at the substrate/ surface interface due to the recovery effect. Fayalite does not appear adherent to the substrate (as temperature of deformation decreases). It shows also more brittle behaviour.

Conclusions

The microstructural characterization of oxide scale was made using EBSD. This technique appears to be successful in differentiating between different phases in a classical oxide scale layer.

It was observed that a very rapid build-up of oxide occurs when a liquid phase, due to the wustite-fayalite eutectic, is present in the surface of the steel. The composition of the oxide scale in the steels treated at 1250°C was a fayalite layer adherent to the iron substrate, followed by a thick layer of wustite and on top of it a thinner layer of magnetite. The thick layer of wustite shows also particles of magnetite (due to wustite partial decomposition into magnetite and iron) and fayalite (located at the wustite grain boundaries). Samples oxidized at 1100°C showed a thinner remaining layer of oxides made of fayalite and a mixture of iron oxides due to the slower oxidation kinetics. In the last case the fayalite layer was not adherent to the substrate.

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