



## Dependence of texture development on the grain size of tertiary oxide scales formed on a microalloyed steel



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### ABSTRACT

Both orientational and geometrical characteristics of grains in tertiary oxide scales have been quantitatively investigated using the electron backscatter diffraction (EBSD) technique. Phase and orientation mappings demonstrate that the {001} planes of magnetite and the {0001} planes of hematite are parallel to the direction of oxide growth. Pole figures (PFs) as scattering plots clearly display the direct correlation between the orientations and microstructure sites of magnetite grains. Magnetite grains develop a strong rotated cube texture component that shifts to the {100}<210> component, whereas those with the grain size of 1–5 μm develop the {001}<100> cube texture component. The refined magnetite grains surrounding the abnormal ones can be a combined process, including magnetite pro-eutectoid during hot rolling and cooling and re-oxidation during air-cooling. Our findings offer insights toward further understanding of the link between the geometrical and orientation parameters of grains with respect to the deformation behaviour of oxide scales formed at elevated temperatures.

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### 1. Introduction

Most metals and alloys inevitably form surface oxide layers due to rapid oxidation during processing at high temperatures, generally at 500 °C and above [1]. Consequently, the formed metallic oxides (scales) have posed a serious obstacle to ensuring a defect-free surface of the steel product in an ecologically friendly way [2,3]. With this in mind, the nature of the surface layers produced on the metal plays a major role in the behaviour of the materials, particularly when exposed to high temperatures and oxidising atmospheres [4,5]. In the case of hot rolling, the surface properties include the surface energy, crystallographic orientation, grain boundaries, texturing of the surface and crystal structure [6]. It is therefore widely expected that tailoring the atomic structure of the oxide layers can enhance tribological properties during environmentally friendly nanoparticle lubrication and further improve the surface quality of the final products [7]. However, the evaluation of individual grain features, such as grain size and orientation in oxide layers has received less investigation.

In the particular case of hot rolling, oxide scales can generally be classified as primary, secondary and tertiary oxide scales. The three types of oxide scales normally correspond to the reheating stages, the roughing stages and the finishing passes of continuous mills, respectively [8]. As the final product on the hot-rolled steel strip, the tertiary oxide scale grows during the finishing rolling and the subsequent cooling down to ambient temperature. The oxide scale is therefore deformed while the steel is processed [9]. In most cases, the tertiary oxide scale consists of a thin outer layer of hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), an intermediate layer of magnetite (Fe<sub>3</sub>O<sub>4</sub>) and an inner layer of wustite (Fe<sub>1-x</sub>O, with 1 – x ranging from 0.83 to 0.95) just above the steel substrate [2,8,9]. These layers of oxide scales may evolve further and undergo structural changes if oxygen is available during air-cooling after coiling [7,8]. Hence, the distribution of these oxide phases depends largely on the heat treatment and atmospheric conditions during hot rolling and the alloying elements contained in the steel compositions [8,10]. In particular, phase distributions within oxide scales will be elusive when considering wustite decomposition below 570 °C and re-oxidation of magnetite in open-air storage of hot-coiled steel [11–13]. In addition, it is still unknown how to characterise the crystallographic texture evolution in tertiary oxide layers.

Measurements of grain size and phase identification in oxide scales have previously been performed using optical microscopy with conventional sample preparation [8,14]. Nevertheless, the main challenge is the complicated and time-consuming polishing procedures because the oxide phases and the steel substrate need to be polished at different

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rates [15]. Sometimes, one or two phases are still difficult to identify, even after etching in a 0.5–1% hydrochloric acid solution in ethanol [14]. With the electron backscatter diffraction (EBSD) technique combined with ion miller sample preparation, only crystallographic characterisation is relied on to determine a grain rather than etching and others [15]. Automated EBSD has therefore become the ideal technique for concurrent access to both the spatial distribution of the grain size and the crystallographic orientation in oxide scales [16], which in turn permits correlations to be made between the two. Texture measurements can hence also be linked to grain size, such as the effect of second-phase particles on the rate of grain refinement in an aluminium alloy [17]. However, the link between grain size and texture evolution in the tertiary oxide scale formed on hot-rolled steel is still unknown.

In this present study, four cases of different grain sizes and orientation distributions in tertiary oxide scales were analysed using EBSD in

an attempt to quantitatively evaluate the corresponding texture evolution. Based on our previous studies [18,19], what we can gain in the current work is a deeper understanding of the microstructural features and crystallographic texture associated with deformation behaviour of oxide scales formed at elevated temperatures.

## 2. Experimental and analytical procedures

### 2.1. Material and HR-AC tests

The material used in this study is a microalloyed low-carbon steel with chemical compositions (wt.%) as follows: C 0.1, Si 0.15, Mn 1.61, Cr 0.21, Al 0.034, P 0.014, S 0.002, N 0.003, Nb + V + Ti 0.016–0.041, and balance Fe. The steel samples were cut into  $400 \times 100 \times 3 \text{ mm}^3$  sheet, ground using SiC papers with 2400 mesh to a surface finish of

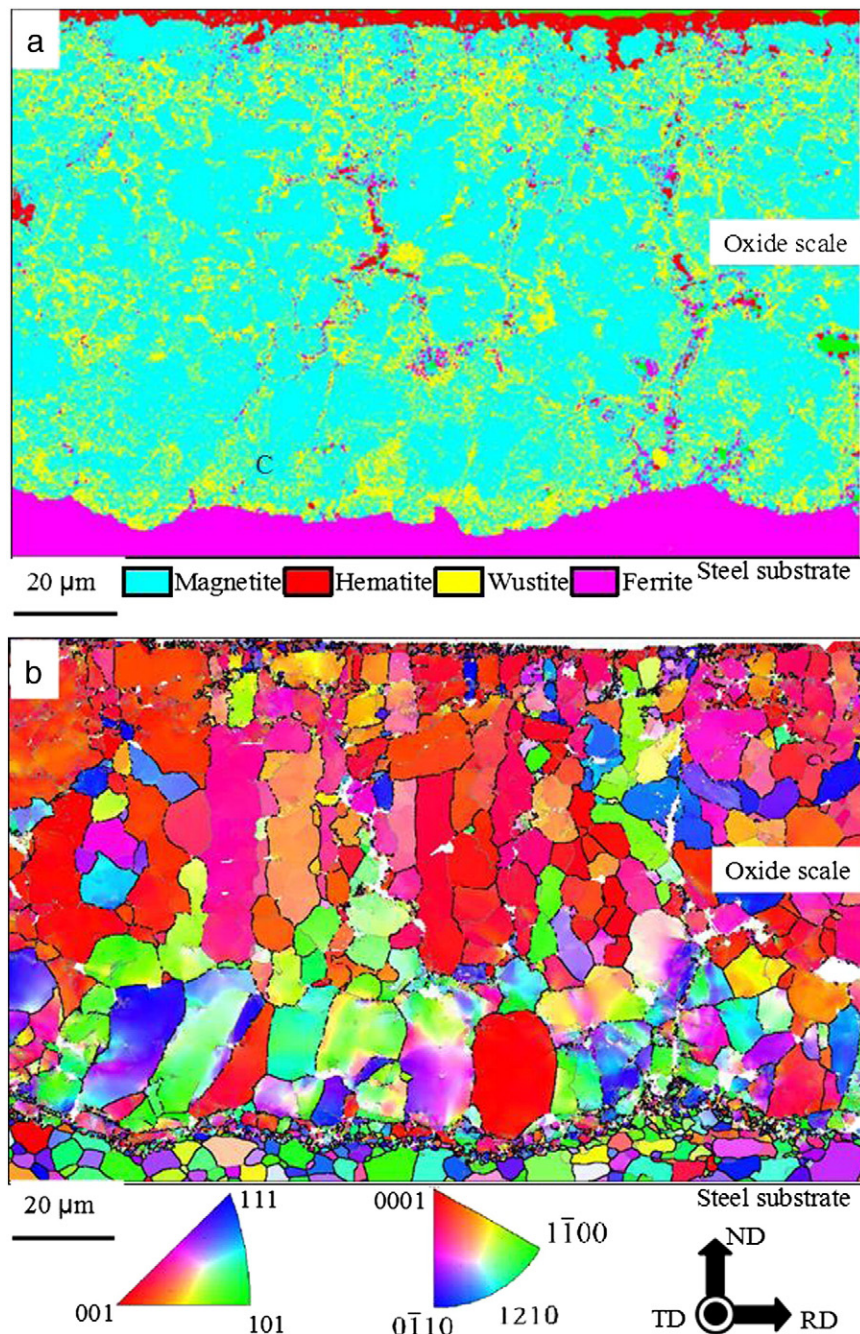


Fig. 1. EBSD (a) phase and (b) inverse pole figure (IPF) orientation maps of tertiary oxide scales formed on the steel substrate.

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