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Microstructure and microtexture evolutions of deformed oxide layers on a hot-rolled microalloyed steel

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ABSTRACT

Electron backscatter diffraction (EBSD) analysis has been presented to investigate the microstructure and microtexture evolutions of deformed oxide scale formed on a microalloyed steel during hot rolling and accelerated cooling. Magnetite and wustite in oxide layers share a strong {001} and a weak {110} fibres texture parallel to the oxide growth. Trigonal hematite develops the {0001} basal fibre parallel to the crystallographic plane {111} in magnetite. Taylor factor estimates have been conducted to elucidate the microtexture evolution. The fine-grained magnetite seam adjacent to the substrate is governed by stress relief and ions vacancy diffusion mechanism.

1. Introduction

Metallic oxide (scale) inevitably grows on the surface of hot-rolled strips due to thermal oxidation at elevated temperatures in corrosive environments, and causes the surface quality of final products to deteriorate [1]. Conventionally, the hot-rolled steel is pickled to remove oxide scale prior to subsequent processing such as cold rolling. On the other hand, the tight oxide scale on an as-hot-rolled microalloyed steel strip can be maintained without acid or mechanical pickling before downstream forming [2–4]. To perceive central point of pickle-free steel [5,6], therefore, becomes more important. This is usually associated with the deformation behaviour of oxide scale during hot rolling [7–9].

Iron-based materials generally evolve in excess of one oxide monolayer which consist of two (below 570 °C) or three (above 570 °C) oxide sub-layers having distinct mechanical properties and growing by different mechanisms [10,11]. Specifically, wustite ($Fe_{1-x}O$, with $1-x$ ranging from 0.83 to 0.95) dominates at high temperatures (normally >650 °C for steel alloys), whereas magnetite (Fe_3O_4) prevails at low temperatures, and hematite (Fe_2O_3) remains low in either case [12–16]. The proportion of the three phases within oxide scale at high-temperature oxidation depends greatly on the equilibrium transformation of wustite or magnetite precipitation which is related to heat treatment, atmospheric conditions, and alloying elements in the steel [17–19].

Substantial research has been conducted on electron backscatter diffraction (EBSD) to investigate the formation of oxide scales on steel products either for phase identification or to determine the grain orientation relationship (OR) and microtexture [20]. The oxide layers: wustite, magnetite and hematite, may be identified using EBSD, even though there is usually little Fe_2O_3 [16,21]. In some cases, the Fe_2O_3 layer may be too thin to be detected with EBSD [13,14]. The internal layer of wustite at high temperature may be either columnar [14,16,22] or equiaxed [13] depending on steel compositions and oxidation conditions such as temperature and atmosphere. It must be emphasised here that the oxide scale formed at high temperature is normally polycrystalline [23,24], whereas, the thermally grown oxide on single crystal substrates are normally epitaxial due to their coherency growth stresses in semiconductor field [25].

In addition to phase identification, analysis of the microtexture in the oxide phases and their OR is now being studied. The distinct oxidation behaviour of microalloyed steel is therefore expected to be associated with oxidation kinetics [26], particular internal stress states [22,27], and the consequent preferential orientation of oxide growth. A strong {001} texture may be found in wustite whatever the steel substrate [12,16], though this fibre texture also evolves in magnetite under low-temperature oxidation [22]. A cube–cube OR between wustite and magnetite may also prevail in undeformed oxide scale possibly due to the defective structure of the wustite [16,21]. Such phenomenon, however, is not always found [13]. In

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contrast, studies on the texture evolution in hematite with trigonal crystal symmetry are very limited due partly to the challenging in phase identification of small amount of hematite. Nonetheless, hematite texture is of practical interest because its high hardness is liable to damage the roll by abrasive wear during hot rolling [3,8]. Further, it is not yet known what kind of texture will be developed during hot rolling, and what its evolution will be during stress relaxation after cooling process.

Currently, the development of ion milling techniques in sample preparation [28,29] allows for the detection of the thin outer layer of hematite in the cross-section of oxide layers, and hence to explore the texture evolution of hematite with a trigonal crystal structure. It is also widely expected to improve our understanding of the texture development of deformed oxide scale, which is now depending primarily on data from the macrotexture [22] rather than microtexture, with little account taken of the fine microtexture or the local stress intensity due to grain orientation.

Even though extensive investigations have been conducted on the formation of oxide scale, only a few studies [30,31] have made any attempt at the deformation behaviour of oxide layers during hot rolling. Instead, they carried out simulations – using plane strain compression, without taking into account of hematite that forms at the steel/rolls contact surface during hot rolling as there is a temperature gradient between them. To date research on wustite decomposition with magnetite precipitation has also been limited to isothermal transformation behaviour [17,32]. Little is known about the dynamics continuous cooling and what effects it has on phase transformation or on the magnetite seam with respect to preferential grain growth. It is therefore a great need for thoroughly understanding phase transformation and adhesion of deformed oxide scale, associated with microstructural variations and microtexture evolution resulting from a coupled rolling–cooling process.

The present study is to characterise the development of microstructure and microtexture in deformed oxide scale subjected to hot rolling with various thickness reductions and accelerated cooling, using EBSD analysis combined with sample preparation in an ion milling system. An investigation has also been conducted to identify deformation microtexture of the trigonal hematite with a higher c/a ratio than the ideal one. By drawing parallels with the observations on the oxidation behaviour of microalloyed steel in previous studies [3,4,9,18,33], insights are gained to help understand the influence of crystallographic orientation on the phase evolution, and thus the final pickle-free steel with tertiary oxide scale through hot-rolling and subsequent accelerated cooling processes.

2. Experimental and analytical procedure

2.1. Material and oxidation procedure

The material used was a commercial Nb–V–Ti microalloyed low carbon steel for an automotive beam. Its chemical compositions are listed in Table 1. Prior to heat treatment, the rectangular samples with size of $400 \times 100 \times 3$ mm^3 were cut from the as-received hot-rolled steels. A tapered edge of each sample [9,33] was also machined in order to feed into the roll gap during the tests. The starting surfaces of all the samples were ground using SiC papers with 2400 mesh to a surface finish of 0.5 μm and cleaned in a solution of acetone. These aim to remove all the initial oxide scales and make sure the same surface state of samples before oxidation experiments.

Hot rolling tests were performed on a 2-high Hille 100 experimental rolling mill combined with a newly developed accelerated cooling system. Samples were reheated in a high temperature electric resistance furnace. Full details of the experimental instruments can be found elsewhere [9,33]. The following procedure was carried out for every oxidation test. The samples were put into the furnace at room temperature firstly, and then were reheated to the predetermined temperature (900 °C) at a heat rate of 1.5 °C/s under a nitrogen atmosphere. After the samples were soaked for 15 min to ensure a uniform temperature and homogenise the austenite grains, a 30 s short-time air oxidation was given before hot rolling. The selection of oxidation time was based on this grade of the steel in the industrial hot rolling process measured by a steel factory [4]. Each individual sheet was given a single rolling reduction at a rolling speed of 0.3 m/s, approximately around 25.5 revolutions per minute (RPM). The rolling direction was the same as the tapered edge without reversing and lubricant was applied on the rolls in hot rolling. The rolled sample was cooled by an accelerated cooling system down to the coiling temperature, and then air cooled to obtain the tertiary oxide scale at room temperature. The steel sheets were hot rolled at different deformation ratios. This heat-treatment schedule is illustrated schematically in Fig. 1. Table 2 shows details of experimental conditions during hot rolling and subsequent accelerated cooling process.

It is noted that the main objective was to maximise magnetite in oxide scale attached to the ferrite substrate. Therefore, the heat treatment sequence was designed to generate a steel substrate first with single austenite phase above 900 °C, and then with a ferrite plus small amount of pearlite at room temperature at various cooling rates. The cooling rates of 10, 13 and 28 °C/s were selected based on the dynamic continuous cooling transformation (CCT) diagram of a Nb–V–Ti microalloyed steel [4,34]. In addition, the thickness reductions of 10%, 23% and 28% correspond to industrial measurements and previous numerical analysis for one-pass hot rolling processes [7,9].

2.2. Sample preparation and analytical methodology

After the gold deposition of the oxidised samples, the edges for cross sectional analysis were ground by SiC papers with 2000 mesh, and then ion-milled at 6 kV for 5 h using a Leica electron microscope (EM) triple ion beam cutter (TIC020) system. As such, the sample surface of oxide growth is defined as the normal direction (ND). The gauge length and width of the rolling sample were parallel to the rolling direction (RD) and traverse direction (TD) of the hot-rolled strip, respectively.

Microtexture analysis was performed on a JEOL JSM 7001F Schottky field emission gun (FEG) scanning electron microscope (SEM) with a Nordlys-II (S) EBSD detector, an 80 mm^2 X-Max energy dispersive spectroscopy (EDS) detector, and the Oxford Instruments Aztec acquisition software suite. The sample was tilted 70°, and the corresponding EBSD map was acquired at an acceleration voltage of 15 kV, a probe current of around 2 nA, and a working distance of 15 mm. According to the average grain size and the different thickness of the deformed oxide scale after hot rolling, the fine step sizes were assigned 0.125, 0.095 and

Table 1
Chemical compositions of the microalloyed low carbon steel (wt.%).

Fe	C	Si	Mn	Cr	P	Al	V	Nb	Ti	S	N
Balance	0.1	0.15	1.61	0.21	0.014	0.034	0.041	0.041	0.016	0.002	0.003