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Tribological analysis of oxide scales during cooling process of rolled microalloyed steel

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Keywords
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Abstract. The composition and phase transformation of oxide scale in cooling process (after hot rolling) of rolled microalloyed steels affect tribological features of rolled strip and downstream process, and the produced steel surface quality. In this study, physical simulation of surface roughness transfer during cooling process with consideration of ultra fast cooling (UFC) was carried out in Hille 100 experimental rolling mill, the obtained oxide scale was examined with SEM to show its surface and phase features. The results indicate that the surface roughness of the oxide scale increases as the final cooling (coiling) temperature increases, and the flow rate of the introduced air decreases. The cracking of the surface oxide scale can be improved when the cooling rate is 20 °C/s, the strip reduction is less than 12 %, and the thickness of oxide scale is less than 15 µm, independent of the surface roughness. A cooling rate of more than 70 °C/s can increase the formation of retained wustite and primary magnetite precipitates other than the precipitation of α-iron. This study is helpful in optimising the cooling process after hot rolling of microalloyed steels to obtain quality surface products.

Introduction

Microalloyed steels are widely used in various structures and automotive components. Promising potential for the Ni-V-Ti microalloyed steels lies in the tight oxide scale formed on the surface of hot-rolled strip due to thermal oxidation at elevated temperature [1-4]. The tight oxide scale is expected to deform with steel substrate without cracking, and may act as self-lubricant at tribological contact surfaces between the workpiece and tool during downstream processing [5, 6]. Thus lower shear strength, good adhesion and dense microstructure could be preferred to self-lubricating oxide scale, which can provide a graded interface where a gradual transition of substrate properties to surface properties occurs [7]. This is the reason why the tight oxide scale offers a great potential to further develop the pickle-free microalloyed steels. Previous studies [1, 6] indicated that the tight oxide scale consists of more than 75 % magnetite and retained wustite, with the oxide-layer thickness of less than 15 µm. The deformation and tribological features of the oxide scale in cooling process after hot rolling is therefore of fundamental and great practical interest.

In our previous studies, the frictional properties of oxide scale were modelled using finite element method [5]. This present work mainly focus on physical simulation of surface roughness transferring during cooling process, particularly on effects of the thickness reductions during hot rolling and cooling rates in ultra fast cooling (UFC) system on surface morphologies, adhesion properties and surface roughness of the oxide scale formed on the microalloyed steel.
 Experimental

 Material and sample preparation. The material used was a commercial Ni-V-Ti microalloyed steel. The chemical compositions are listed in Table 1. The steel samples were cut into 400×100×3 mm³ sections with a tapered edge [8]. The specimens were ground using SiC papers of 2400 mesh to a surface finish of 0.5 µm, and cleaned in a solution of acetone.

<table>
<thead>
<tr>
<th>Table 1 Chemical compositions of the microalloyed steel.</th>
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<tbody>
<tr>
<td>Elements</td>
</tr>
<tr>
<td>wt.%</td>
</tr>
<tr>
<td></td>
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</tbody>
</table>

 Apparatus and experimental procedure. The experiment was performed on an ultra fast cooling (UFC) system attached to a 2-high Hille 100 experimental mill, as shown in Fig. 1. The UFC system consists of top and bottom double banks of water spray nozzles, which mean the flow rate is much greater than conventional cooling methods such as spray or water column cooling. Two Raytek GPs non-contact infrared pyrometers were located above the roll gap entry and exit of the cooling equipment to simultaneously monitor and control the temperature of the sample. The following procedure was carried out during the UFC tests for every cooling rate experiment. Each specimen was reheated to 900 °C in the furnace in a nitrogen atmosphere and soaked for 15 min to ensure a uniform temperature. Upon removal from the furnace, each specimen was placed on the entry roll platform of the rolling mill and allowed to air cool to a predetermined temperature, after which each individual strip was given a single rolling reduction at a rolling speed of 0.3 m/s. The rolled specimen was cooled by the UFC system down to the coiling temperature (600-350 °C) at different cooling rates. The experimental conditions of the samples used are tabulated in Table 2.

![Temperature detector at exit of cooling](image1)

![Ultra-fast cooling](image2)

![Hille 100 rolling mill](image3)

![Temperature detector at entry of cooling](image4)

![Conjunction table roller](image5)

Fig. 1 The ultra fast cooling (UFC) system attached to the 2-high Hille 100 experimental rolling mill, including temperature detectors located at the entry and exit of the UFC system, and the conjunction table roller between them.
Table 2 Experimental conditions for hot rolling combined with ultra fast cooling (UFC) tests.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>531</th>
<th>535</th>
<th>221</th>
<th>232</th>
<th>233</th>
<th>431</th>
<th>432</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cooling rate °C/s</td>
<td>10</td>
<td>20</td>
<td>10</td>
<td>50</td>
<td>100</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Reduction %</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>30</td>
<td>40</td>
</tr>
</tbody>
</table>

**Analytical methodology.** The surface morphology after UFC tests was examined in a JEOL JSM 6490 scanning electron microscope (SEM) operating at 15 kV with a working distance of 10 mm. For cross-section analysis the samples were mounted in epoxy resin and polished down to 0.5 µm using standard metallographic techniques [9]. A Keyence VHX-1000E Digital microscope (OM) was used to characterise the cracks and thickness of the deformed oxide scale. Also, the surface roughness of oxide scale after deformation was measured by a TR220 profilometer.

**Results and Discussion**

**Surface morphology.** The variation of surface morphologies was driven by thickness reductions during hot rolling and cooling rates in UFC system. Fig. 2 shows the surface morphologies of the hot-rolled oxide scale at the different thickness reductions. The oxidised microalloyed steels exhibit a much smoother surface as the thickness reduction increased from 5 % to 40 %. There are larger areas with blistering at the thickness reduction of 5 %, whereas, the narrow cracking in brighter colour appeared that of 40 %. This could be attributed to plastic flow in the oxide scale occurred at the entry of the roll gap during hot rolling.

![Fig. 2](image)

Fig. 2 Surface morphology of the hot-rolled oxide scale at a thickness reduction (Red), a) 10 %, b) 20 %, c) 30 %, and d) 40 %.

For various cooling rates, the oxide scale has been characterised through the surface cracking, as indicated in Fig. 3. These failure features are more apparent at the edge of the strip from the cooling rates of 10 to 100 °C/s. With a cooling rate of 50 °C/s, a few long and narrow cracks were produced across the width of the strip, there were more large cracks in the oxide scale, but when the cooling rate increased to 100 °C/s, the secondary cracks near the edge were wider than in the central region and become curved in the rolling direction. The regularly spaced cracking curvature could correspond to inhomogeneous plastic deformation which resulted from the strip widening at the roll gap [10].
Fig. 3 Cracks in the oxide scale after a thickness reduction of 20% and a cooling rate (CR) of 10, 50, and 100 °C/s.

The cracking depth and width above were measured under different cooling rates and strip reductions (Fig. 4). In most cases, small cracks can be obtained at a cooling rate of 20 °C/s and a reduction of 12%. There are relatively small differences between the cracking depth and width of the oxide scale at the cooling rates below 20 °C/s. It inferred that the temperature variation in surface and underneath of the steel induces the uniformity of thermal stress, and thus lead to the surface cracking. At a higher reduction, the crack spacing was found to be 5-10 % narrower than the others. This is possibly because the plastic elongation of the surface of the strip occurs near the roll gap, whereas, the sub-surface material shears to generate plastic compression in the thickness direction.

Fig. 4 Edge cracking of the oxide scale occurred at different a) cooling rates, b) thickness reductions.

**Adhesion properties.** The relationship between the cooling rates and adhesion of the oxide scale after UFC cooling system used, was established according to the cross sectional cracking shown in Fig. 5. The images at the right side were obtained using the binarisation processing based on the original OM images corresponding to the cooling rates. The failures occurred in the oxide scale generally have some micro-holes and cohesive oxide failure within the oxide scale, adhesive oxide failure at the interface of the oxide scale and steel substrate [11]. The oxidised sample cooled at 10 °C/s appears to be intact and the oxide layer adhered, although with some scattered pores. With the cooling rate of 20 °C/s, the cross sectional morphology of oxide scale is either adherent or not along the thickness with some brittle cracks and attached debris. There is a great deal of small debris in the oxide scale cooled at 40 °C/s, and the out-layer of oxide scale was in smaller pieces. When the cooling rate increased to 100 °C/s, the oxide scale was badly damaged and showed poor adherence,
although some small pieces of oxide remained attached next to regions that were almost free of scale. The results indicate that the adhesion properties of the oxide scale are quite well when the cooling rates were less than 20 °C/s. This could be explained by the compression stresses that developed as the oxide layers grew and then cooled.

![Fig. 5 Adhesion properties of the oxide scale at a cooling rate (CR) a) 10 °C/s, b) 20 °C/s, c) 50 °C/s, and d) 100 °C/s. Group 2 are binary images corresponding to the cooling rates.]

With the cross sectional morphology, various cracking failure such as blistering and extrusion were examined on the basis of different thickness of oxide scale and reductions, as indicated in Fig. 6. The oxide scale with the thickness less than 15 µm can be expected a plastic elongation at the thickness reduction of 15 %, whereas, that of much thicker than 100 µm has plastic flow at a lower thickness reduction. The extrusion of the fresh steel will be happened at much higher thickness reductions. In any case, the fracture at the roll gap became more significant under a reduction of more than 15 % to analyse the ductility of the oxide scale.

![Fig. 6 The relationship between cracking behaviour, the thickness of oxide scale, and the thickness reduction of microalloyed steel]

**Tribological feature.** The deformation behaviour of the oxide scale formed the microalloyed steel depends on the plastic flow of materials during fast cooling after hot rolling. Fig. 7 presents the variation in the surface roughness of the oxide scale with the thickness reductions and cooling rates. The value of average surface roughness, Ra, reduced by about 57.9 % as the thickness reduction increased from 10 % (Ra=2.416 µm) to 20 % (Ra=0.985µm). This decrease in surface roughness as the thickness reduction increases confirms our previous calculated results [5], and this could be due to
reduction of surface asperity. Nevertheless, there is only 14.5% increase in surface roughness at the cooling rates from 50 °C/s ($R_a=0.957 \, \mu m$) to 100 °C/s ($R_a=1.105 \, \mu m$). One possible reason is that the nucleation of the oxide scale at the higher cooling rates is greatly inhibited as insufficient time for atomic inter-diffusion.

Fig. 7 Surface profiles of the oxidised samples at a) a thickness reduction (Red.) 10 %, b) 20 %, and c) a cooling rate (CR) 50 °C/s, d) 100 °C/s.

Conclusions

Tribological properties of the oxide scale is varied in the thickness reduction and cooling rate in UFC system after hot rolling. The surface roughness is lower while the integrity of oxide scale deteriorates as the thickness reduction increases from 5 to 40 %. The increase in the cooling rates from 10 to 100 °C/s leads to significant cracks of the oxide scale. The uniform surface morphologies and good adhesion properties of oxide scale can be achieved at a cooling rate of 20 °C/s and a reduction below 12 %.

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References