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The role of oxide-scale microtexture on tribological behaviour in the nanoparticle lubrication of hot rolling

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Electron backscatter diffraction (EBSD) of the microtexture in the tertiary oxide scale after hot rolling were investigated. The surface asperity flattening and grain refinement of Fe_3O_4 were produced at a thickness reduction of 28% and a cooling rate of 28 °C/s. Microtexture development of Fe_3O_4 manifests as a strong θ fibre parallel to oxide growth, including the $\{100\}<001>$ and $\{001\}<110>$ textured components, whereas the $\{0001\}<10\bar{1}0>$ component dominates in α-Fe_2O_3 as the favoured basal slip. The tribological effect of α-Fe_2O_3 at the contact surface of steel and rolls was considered at low and high thickness reductions. The propagation of cracks along the Fe_3O_4 grain boundaries could create a dish to collect nanoparticles during lubrication and thereby changed the wear rates.

1. Introduction

Green tribology, similar to green chemistry, seeks to reduce and prevent pollution at its source and further improve efficiency [1]. Nevertheless, tribology involves not only surface chemistry but also the mechanics and physics of interacting surfaces [2,3]. The nature and consequence of interactions that take place at the interface control its friction, wear and lubrication. Environmentally friendly materials, lubricants and processes have become significant aspects of green tribology. Biodegradable lubrication, particularly water-based lubricants, has attracted the attention of researchers in recent years [4–6]. Innovative water-based lubricants containing nano-additives are viewed as viable substitutes for conventional oil-based lubrication [7–9]. These lubricants provide effective and uniform lubrication between rolls and workpiece surfaces under high-temperature processing [10]. Normally, they are environmentally friendly and ideally recyclable [11,12]. Moreover, surface texturing provides a means to control multiple surface properties relevant to making tribo-systems more ecologically friendly. These surface properties that are associated with tribological performance include surface energy, crystallographic orientation, grain boundaries, surface texturing and crystal structure [1,13].

The tribological properties of the lubricating system will lead to a better understanding of the overall situation when the concept of green tribology is applied to high-temperature metal processing, such as hot rolling. This is because oxide scale (metallic oxides) inevitably forms on the surfaces of steel strips at high temperatures, which poses a serious obstacle as this causes the surface quality of final products to further deteriorate [14,15]. Conventionally, lubrication in hot rolling aims to reduce rolling force, friction and wear, to improve the surface quality of end products, and finally, to reduce energy consumption [16,17]. As such, currently employed liquid lubricants are generally natural organics consisting of animal fat, vegetable oils, mineral (or petroleum) fractions, synthetic organics, and mixtures of two or more of these materials [1,18]. Various solid additives are used to improve specific properties of the lubricant, particularly lessening the temperature dependency on viscosity [19,20]. The most promising solid additives are graphite and molybdenum disulphide (MoS_2) [1,8,9]. Sputtered MoS_2 coatings are widely paired with bearings and other sliding friction components employed in non-oxidising environments, such as satellites, space shuttles, and other aerospace applications [1]. However, most metals and alloys

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inevitably grow surface oxide layers due to processing at elevated temperatures [15,21,22]. As a result, graphite nanoparticles are a candidate for use as additive in hot rolling lubricants [10]. In regard to solid additives in water-based lubricants, little is known about nano-additives in lubricants and their effects on hot rolling. Techniques to enhance water-based lubricant performance are of particular scientific interest and will lead to a substantial reduction in energy required for metal processing. As a compromise, it is widely expected that tailoring the preferred orientation of oxide scale can be used to enhance the tribological properties of nanoparticle lubrications, and further improve the surface qualities of steel [23,24]. However, the characterisation of microtexture evolution in deformed oxide layers remains incomplete.

Generally, multi-layered oxide scales form on steels at high temperature. The oxides consist of a thin outer layer of haematite (α-Fe_2O_3), an intermediate layer of magnetite (Fe_3O_4), and an inner layer of wustite ($Fe_{1\text{-}x}O$, with 1–x ranging from 0.83 to 0.95, to be abbreviated as FeO) situated immediately above the steel substrate [21,25]. The proportional thickness of the three layers depends largely on the heat treatment, the atmospheric conditions during hot rolling, and the alloying elements in the steel composition [26,27]. Specifically, FeO dominates at high temperatures (typically >650 °C for steel alloys), whereas most Fe_3O_4 dominates at low temperatures below 570 °C [28,29]. This variation is due to the equilibrium transformation of FeO, i.e., the pro-eutectoid precipitation of Fe_3O_4 followed by FeO decomposition into Fe_3O_4 and ferrite (α-Fe) below the eutectoid temperature of the FeO at 570 °C [30,31].

Recently, texture-based studies on the heat-treatment oxides of steels have emerged. Microtexture evolutions of spinel Fe_3O_4 and cubic FeO share a strong {100} $<001>$ texture component in undeformed oxide scale that is grown on IF steel at 760 °C [32]. The same $<001>$ fibre, which is parallel to the growth direction, has been investigated in low carbon steel and pure iron during oxidations above 600 °C and below 1200 °C [33]. Similarly, the microtexture evolutions of deformed Fe_3O_4 and FeO in the oxide scale grown on an ultra-low carbon steel from 650 to 950 °C, have also occurred with a strong cubic component that rotates towards $<100>$ directions when deformed. The final texture lies along the θ-fibre including {100} $<001>$ cube and {100} $<120>$ rotated cube components [34]. By contrast, the α-Fe_2O_3 texture is of practical interest because it is usually related to friction and tool wear during deformation. Specifically, the high hardness of α-Fe_2O_3 is liable to damage the rolls by abrasive wear during hot rolling. It is well known that α-Fe_2O_3 possesses a hardness value of up to 1000 HV, whereas the counterpart material of a high speed steel roll has a typical hardness only 660–710 HV (64–66 HRC) [35]. However, it is not yet known what type of texture evolution α-Fe_2O_3 will develop during hot rolling, particularly when a new α-Fe_2O_3 can grow at the steel/roll contact surface during hot rolling, as a temperature gradient is established at the interface.

In our previous studies, many efforts have been made towards obtaining a better understanding of the oxidation mechanism and the texture development of oxide scale. The initial motivation was to elucidate the formation mechanism of the tight oxide scale grown on a grain-refined micro-alloyed low-carbon steel during hot rolling [36]. Subsequent endeavours included experimental investigations on the effects of cooling temperature [37] and cooling rate [38] on oxide scale growth [39], numerical modelling of temperature-dependant growth kinetics [40] and of an enthalpy-based method for phase transformations in oxide scale [41], and tribological analyses of oxide scale during the cooling process [42] and the corresponding precipitation behaviour of magnetite [43,44]. We have found that microstructures of a steel substrate under SEM-SEI mode and a multi-layer oxide scale under SEM-BSE mode can be clearly distinguished from one another; however, both surfaces cannot be distinguished simultaneously under the same mode of SEM-SEI or SEM-BSE [45]. By using EBSD to characterise both the steel substrate and the oxide scale concurrently, we undertook an additional effort to further the texture-based analysis of oxide scale [46]. The general type of crystallographic texture developed in deformed Fe_3O_4 and α-Fe_2O_3 scale was identified [47], and its evolution was investigated [46]. The analysis on texture evolution has encouraged us to investigate how grain boundaries [48], grain size [49] and local strain [50] influence the properties of oxide scale during hot rolling. In the present study, the texture evolution has been fully quantified by means of EBSD-EDS, which was accurately represented with orientation distribution function (ODF) sections. Then, the contribution of these microtexture components to the tribological behaviour in nanoparticle lubrication of hot rolling has been evaluated. An attempt has also been made to establish the oxidation mechanism of Fe_3O_4 to α-Fe_2O_3 that occurs along the Fe_3O_4 cracks near the surface of the roll gap and to understand the corresponding tribological effects of nanoparticles lubrication.

2. Experimental and analytical procedure

2.1. Material and hot rolling tests

The material used was a micro-alloyed low-carbon steel, and its chemical composition is listed in Table 1. The steel sample was cut into $400 \times 100 \times 3$ mm^3 sheets with tapered edges to feed into the roll gap. These sheets were then ground to a surface finish of 0.5 μm using SiC papers with 2400 mesh, and cleaned in a solution of acetone prior to the hot rolling tests.

Hot rolling tests were performed on a 2-high Hille 100 mill combined with an accelerated cooling system. Full details of the experimental instruments have been given in our previous studies [38,42]. For clarity, some operational parameters are tabulated in Table 2. The procedures are concisely described here. Steel sheets were heated to 900 °C in nitrogen then, air-cooled to approximately 860 °C and subsequently one-pass hot rolled; finally, sheets were water-cooled at a cooling rate of 10–90 °C/s to the desired temperature. Thereafter, the sheets were air-cooled to obtain the tertiary oxide scale at room temperature. The thicknesses of oxide scales were in the range of 60–80 μm. It is noted that the deformability of the scale layers during hot rolling would be markedly affected because the strengths of the oxides were much greater than that of the steel substrate, and there was a chilling effect when the oxide scale came into contact with the roll surfaces. Because backlash could occur in the experimental mill system, the measured values of thickness reductions and cooling rates are highlighted in Table 3. In addition, the surface roughness of oxide scales was measured by a TR220 profilometer.

2.2. Sample preparation and analytical methodology

For ion milling, the samples were cut into blocks of $20 \times 20 \times 7.8$ mm^3 from the centre of the hot-rolled sheets along the planes of the rolling direction (RD) and the normal direction (ND). After gold deposition, the edges of the samples used for cross-sectional analysis were ground using 2400 mesh SiC papers, and ion milled at 6 kV for 5 h using a Leica triple ion beam cutter (TIC020).

Simultaneous EBSD – energy dispersive X-ray spectroscopy (EDS) analyses were 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 EDS detector and the Oxford Instruments Aztec acquisition software suite. The sample was tilted 70°, and the corresponding EBSD map was