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Keywords

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Analysis of TiO₂ nano-additive water-based lubricants in hot rolling of microalloyed steel

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Abstract: Hot rolling tests of microalloyed steel were performed at rolling temperatures of 1050, 950, and 850 °C under different lubrication conditions, including dry, water lubrication, and 1.0 vol.% oil-in-water (O/W) emulsion. The rolling force, surface roughness, and thickness of the oxide scale of the rolled steel were investigated in order to evaluate the lubrication effects of TiO₂ nano-additive water-based lubricants. The results show that the rolling force under a rolling temperature of 1050 °C does not vary significantly under all the lubrication conditions. The 4.0 wt.% TiO₂ water-based lubricant, however, leads to the lowest rolling force at rolling temperatures of 950 and 850 °C. The application of 4.0 wt.% TiO₂ water-based lubricant in hot rolling not only produces the best surface finish, but also results in the thinnest oxide scale of the rolled steels. The lubrication mechanisms of TiO₂ nano-additive water-based lubricants in hot rolling were also discussed.

Keywords: Microalloyed steel; Hot rolling; TiO₂ nano-additive; Water-based lubricant

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1. Introduction

Hot rolling is one of the key manufacturing processes in the steel rolling industry which is used to obtain not only the required dimensions and mechanical properties, but also the surface finish of the rolled steel in demand [1, 2]. The lubrication applied in the hot rolling of steels has been a long-standing research topic because it has a potential to improve both the roll life and the surface quality of the steel as well as to decrease energy consumption. This means that the rolling force can be minimised and the rolls can be replaced less frequently [3, 4]. It has been found that the application of lubricants during hot rolling process can decrease power consumption and rolling loads by 15-25%, and increase roll life by 20-40% [4]. Lubrication during hot rolling is also beneficial for the optimisation of the texture towards the rolled steels [5, 6].

Traditional oil-in-water emulsions have been used extensively in hot rolling [7-11]. The lubrication behaviour at the interface between the roll and the workpiece has been investigated and different lubrication mechanisms and models have been proposed [7, 8]. Previous research has been focused primarily on achieving the best lubrication effect of the oil-in-water emulsion based on the decrease of rolling force and roll wear, as well as the improvements in surface finish. For this purpose, a number of studies have been conducted on the effect of oil type and concentration, supplying method and spray angle, size and width etc. [8, 10, 12]. These studies have tested different interfacial lubrication conditions such as the dry condition, the use of distilled water or oil-in-water emulsions as well as commercial hot forging oil, and the results have shown that a ratio of 1:1000 oil-in-water emulsions attains the greatest reduction in mill load and also the best insulating ability of the rolled steel surface [9-11]. The oil film formed on the roll surface is adhesive and allows the achievement of low consumption of lubricants. It also enables the rolls and the workpieces to be separated from each other so as to reduce friction and wear, and thus enhance the surface quality of the strip [10].

One problem, however, is that oil-based lubricants are often applied directly onto the rolls through small hydraulic or air/steam atomisation nozzles, which are prone to be blocked and regular maintenance is required. Another problem is that oil-based lubricants can be flushed away by the cooling water with high pressure, which is used to suppress the surface temperature of the rolls. This means that the lubrication effect can be substantially reduced. Moreover, oil-based lubricants produce smoke during hot rolling process and the discharge of residual oil will be of environmental concern [13]. Therefore, a new type of lubricant which is environment-friendly and effective in lubrication needs to be developed in order to substitute the traditional oil-based lubricant.

Nanotechnology applied in lubrication has been becoming a hot research topic in the fields of metal machining and manufacturing [14-16]. With the increasing concern for environmental protection and energy conservation, the development of nano-additive water-based lubricants shows a great potential during hot rolling process [13, 17]. They play roles not only for lubricant but also for coolant, thus reducing the temperature of the roll surface and roll wear, prolonging the roll life, lowering down the friction between the roll and the workpiece, improving surface finish of the workpiece, and flushing away impurities from the roll surface [18]. It has been reported that the addition of a small amount of nanoparticles into water would make water-based lubricants with outstanding lubrication performance in the fields of tribology and rolling process [17, 19-26]. In particular, research efforts have been directed towards using TiO₂ nanoparticles as additives which can be dispersed uniformly in water, and the results have showed that it is capable of decreasing the rolling force, improving the surface quality, and reducing the coefficient of friction (COF) as well as the roll wear [17, 22, 25, 26]. Nevertheless, there has been little research into the water-based lubricants used in hot rolling, and more studies about the lubrication mechanisms of water-based lubricants with nano-additives are needed.

In the present work, different water-based lubricants with varying TiO₂ nano-additives were prepared to study their lubrication effectiveness on the rolling force, the surface roughness, and the oxide scale formed on microalloyed steels during hot rolling processes. The objective of this study is to reveal the lubrication mechanisms of TiO₂ nano-additive water-based lubricants and to provide an innovative method to substitute the traditional oil-based lubricants in order to solve the above-mentioned problems in the practical hot rolling production line.

2. Experimental

2.1 Material

A low-carbon microalloyed steel was used in this study. Its chemical compositions are listed in Table 1.

The steel samples with dimensions of 300×50×8 mm³ were machined with tapered edges to help feeding into the roll gap. These samples were ground and polished to ensure a surface roughness (R_a) of approx.0.5 μm, which eliminated the influence of the original surface conditions on the experimental results. Prior to the tests, the samples were cleaned in a solution of acetone and then stored in a desiccator.

2.2 Preparation of lubricants

The TiO₂ nano-additive water-based lubricants were prepared according to the flow chart shown in Fig. 1. The whole preparation procedure has been reported in previous study [26]. First, TiO₂ nanoparticles (P25 sourced from Sigma-AldrichTM with approx.20 nm in diameter) were mixed into deionised water by

mechanical stirring. Next, Polyethyleneimine (PEI) was gradually added into the solution followed by a high speed centrifuge at 20,000 rpm for 30 min to prepare a dispersive solution. PEI is a cationic polymer, which acts as a surfactant of TiO_2 in order to improve the dispersing property of the nanoparticles. Afterwards, glycerol was slowly added. Glycerol is a colourless, odourless, and viscous liquid, which is used to improve the viscosity of solutions. The solution was then processed by ultrasonication with stirring for 10 min to break down any remaining agglomeration. The prepared TiO_2 nano-additive water-based lubricant showed good colloid stability, and no sedimentation could be observed during the 7 days of aging.

The chemical compositions of as-prepared lubricants are listed in Table 2. The different lubrication conditions (shown in the Table 2 as numbered 1 – 8) were applied for the hot rolling tests. The dry condition, water lubrication, and 1.0 vol.% O/W emulsion were chosen as benchmarks compared to the lubrication effects of as-prepared water-based nano-additive lubricants. The reason for using 1.0 vol.% O/W emulsion as an oil-based representative in this study was that the COF during hot rolling decreased as the emulsion concentration increased in the range below 1.0 vol.%, but became constant at more than 1.0 vol.% [7, 8]. The water-based lubricants consisted of varying mass fractions of TiO_2 nanoparticles (from 0.4 to 8.0 wt.%), and corresponding mass fractions of PEI. The mass concentration of glycerol was fixed at 10.0 wt.% for each type of nano- TiO_2 lubricant.

2.3 Hot rolling tests

Hot rolling tests were performed on a 2-high Hille 100 experimental rolling mill with rolls of 225 mm in diameter and 254 mm in length. A flow chart of the hot rolling process is shown in Fig. 2. The steel samples were put into a high temperature electric resistance furnace and then heated up to 1100, 1000, and 900 °C, respectively, for 30 min with nitrogen inside flowing at a rate of 15 L/min in order to control the thickness of the oxide scale. The actual starting rolling temperatures were 1050, 950, and 850 °C, respectively, with a reduction of 30% and a rolling speed of 0.35 m/s. After hot rolling, the samples were immediately cooled down in the air. Various lubricants were uniformly sprayed onto the rolls prior to hot rolling, and the rolls were cleaned with acetone each time before a following test. Two pieces of samples were applied under the same rolling conditions in order to obtain average value of testing results. It should be noted that the volume of various lubricants adhered onto the roll surfaces is inconsistent because of their different wettability. In order to maintain a reasonable comparison of various lubrication effects, the maximum capacity of absorption for each type of lubricant on the roll surfaces was considered as a criterion. In this case, the lubricants were continuously sprayed manually onto the roll surfaces until a

saturated layer of lubrication film was formed. The saturation can be defined hereby when the droplet on the roll surface began to drop down after formation of a uniform and compact lubrication film.

2.4 Characterisation of nanoparticles

Powder X-ray diffraction (XRD) was implemented using a Philips PW1730 conventional diffraction meter with Cu-K α radiation. The XRD pattern of the nanoparticles is shown in Fig. 3, from which the phase of particles can be determined as typical P25 TiO $_2$ containing 75% of anatase and 25% of rutile by referring to the XRD standard atlas.

Micrographs of TiO $_2$ nanoparticles inside the water-based lubricants were obtained using a JEOL JEM-ARM200F Transmission Electron Microscope (TEM) coupled with an energy-dispersive spectrometer (EDS). Fig. 4 exhibits the TEM images of the TiO $_2$ nanoparticles dried from the as-prepared water-based lubricants with mass concentrations from 0.4 to 8.0 wt.%. It can be clearly seen from Figs. 4(a)-(d) that majority of the nanoparticles are spherical with a diameter of around 20 nm, and the nanoparticles are uniform and well-dispersed without apparent agglomeration when the concentration is below 8.0 wt.%, which produces bigger particle size than other types.

2.5 Analysis methodology

The rolling force during the hot rolling process was detected by two force sensors assembled in the rolling mill. The data were acquired simultaneously by a designed programme in the MATLAB software.

The surface roughness of the rolled steel was measured by a KEYENCE VK-X100 K 3D Laser Scanning Microscope. Three areas, including head, centre, and end of each rolled specimen surface were considered for the measurement of surface roughness. At least five measurement points along the longitudinal centre line of the surface were made to obtain average values of the measured results.

The surface morphology and cross-section of the rolled steel were observed and analysed under a KEYENCE VK-X100K 3D Laser Scanning Microscope and a JEOL model JSM-6490 Scanning Electron Microscope (SEM) equipped with an energy-dispersive spectrometer (EDS). Epoxy resin was mounted onto the rolled surface before cross-section observation to prevent oxide scale from being further oxidised. The cross-section was coated with Au deposition prior to SEM analysis, which was employed to characterise the lubrication mechanisms during the hot rolling process.

The wettability of lubricants was evaluated by the measurement of contact angle using a Rame-hart 290 Goniometer. The lubricant droplets with the same volume spread on the surface of roll material (High Speed Steel, abbreviated as HSS) were observed with amplified profile projection, followed by an

angulation in the affiliated software. All the surfaces of HSS specimens were polished to an identical R_a of 0.4 μm , which can eliminate the influence of the original surface condition on the measuring results.

3. Results

3.1 Rolling force

Fig. 5 shows the rolling force obtained under various lubrication conditions at rolling temperatures of 1050, 950, and 850 °C. It can be seen that the rolling force varies slightly at 1050 °C under all lubrication conditions and this indicates that all the lubricants cannot function successfully at such high temperature. Differently, when the rolling temperature decreases to 950 and 850 °C, the rolling force with TiO_2 nano-additive water-based lubricants is lower than those under the dry and water lubrication conditions. It is noted hereby that there is no significant impact on decrease of rolling force when the concentration of water-based lubricant is below 1.0 wt.%, whereas, the rolling force begins to be reduced successively as the TiO_2 mass fraction increases up to 4.0 wt.%, which leads to the lowest rolling force among all the lubricants. When the mass fraction of TiO_2 further increases to 8.0 wt.%, however, the lubrication effect on the reduction of the rolling force deteriorates. More importantly, the lubrication effect of the 4.0 wt.% TiO_2 nano-additive water-based lubricant is comparable to that of 1.0 vol.% O/W emulsion in terms of these reductions of rolling force. In order for revealing the specific values of rolling force, the actual experimental data and standard deviation have been given in Table 3. It is shown that the rolling force can be decreased by 6.0% and 8.0%, respectively, at rolling temperatures of 850 and 950 °C, compared to that of dry condition.

3.2 Surface roughness

The surface roughness of the steels rolled at 1050, 950 and 850 °C is shown in Fig. 6. It is clearly seen that the variation trends under these three temperatures are quite similar. The R_a under the dry condition have the highest values at 1.24, 1.1 and 0.95 μm at 1050, 950 and 850 °C, respectively, and the roughness of the rolled steels begin to decrease continuously along with the increase in the content of TiO_2 nano-additive. The R_a reaches its lowest point when 4.0 wt.% TiO_2 is used. Further increase in TiO_2 content to 8.0 wt.% results in a rise in R_a value. In comparison to 1.0 vol.% O/W emulsion, the 4.0 wt.% TiO_2 lubricant produces a slightly better surface finish.

3.3 Thickness of the oxide scale

Fig. 7 shows the thickness of the oxide scale formed on the steel surface after hot rolling at 1050, 950 and 850 °C under various lubrication conditions. It can be seen that TiO_2 nano-additive water-based lubricants induce thinner oxide scale than that of dry and water conditions. With the increase of TiO_2 mass fraction,

the thickness of the oxide scale decreases gradually until the thinnest point is reached when 4.0 wt.% TiO_2 is applied, after which an increase in the oxide scale thickness is observed when the mass fraction of TiO_2 further increases to 8.0 wt.%. By contrast, the 1.0 vol.% O/W emulsion yields slightly thicker oxide scale than that caused by the 4.0 wt.% TiO_2 suspension. It is obvious that the thickness of the oxide scale under dry condition at 1050, 950 and 850 °C can be decreased by 38.4%, 36.5% and 30.5%, respectively, with the application of 4.0 wt.% TiO_2 lubricant. The morphologies of the cross section with oxide scale formed at 950 °C are shown in Fig. 8.

4 Discussion

4.1 Wettability

Wettability, one of the most important lubricant characteristics, indicates how well a lubricant can wet a solid surface [27]. Wetting of surfaces in tribological interaction is extremely important to lessen friction and wear, which relates to the rolling force and roll wear in the rolling process [28]. Generally, superb wettability intends to facilitate the formation of a lubrication film which is able to separate the friction pair contacting each other [29]. Wettability can be characterised by contact angle measurement: the smaller the angle, the higher the wettability [30]. It has been reported that the addition of nanoparticles into liquid induces an improved wettability on substrate due to a reduction of contact angle [16, 31, 32].

Fig. 9 illustrates the wettability of various lubricants on the surface of HSS. It is shown that the water-based lubricants exhibit lower contact angle (θ) than water, and they also result in lower contact angle compared to 1.0 vol.% O/W emulsion when the concentration is above 4.0 wt.%. The contact angle of water-based lubricants on the HSS surface decreases gradually with the increase of mass concentration of TiO_2 and PEI until it reaches the lowest value ($\theta=52^\circ$) with 8.0 wt.% TiO_2 . The digital images of the adhered lubricants onto the roll surface are shown in Fig. 10, and the marked area in Fig. 10(a) shows the effective contact area in the hot rolling corresponding to the width of the workpiece. It should be noted that 4.0 wt.% TiO_2 presents comparable wettability to 8.0 wt.% TiO_2 , which indicates that the volumes of lubricants adhered to the roll surface in the contact area are nearly consistent in both cases. More importantly, the two lubricants enable the roll surface to accommodate more effective amounts of TiO_2 nano-additives, compared with other water-based lubricants with lower concentrations. Therefore, at low rolling temperatures of 950 and 850 °C, the lubrication effect for reducing rolling force could be ascribed to the effective amounts of TiO_2 nano-additives adhering to the roll surface: the higher the concentration of TiO_2 nano-additives, the lower the rolling force. Taking into consideration the instant loss of the lubricants at these high temperatures, there should be surplus nanoparticles left to form a lubrication film on the roll surface to separate the roll and workpiece from direct contact with each other at the low rolling

temperatures [33-35]. The nanoparticles can also have a rolling effect as balls between the roll and workpiece [35-37]. Both the lubrication mechanisms of water-based lubricants can reduce the friction during hot rolling process and then lead to better lubrication effect than the benchmark lubricants. The 4.0 wt.% TiO₂ water-based lubricant also shows the lowest rolling force among all the lubricants, including the 1.0 vol.% O/W emulsion. When the fraction of TiO₂ nano-additives is lower than 4.0 wt.%, only a small number of nanoparticles behave lubrication effect, however, when the fraction is higher (8.0 wt.%), the nanoparticles will agglomerate with a larger particle size, and thereby increase the friction and this produces a higher rolling force during hot rolling process [22].

At the rolling temperature of 1050 °C, instead, the TiO₂ nano-additive water-based lubricants are inclined to running off instantly due to the apparent Leidenfrost effect [38, 39]. This effect implies that when the lubricants adhered onto roll surface contact the hot steel surface at a temperature greater than the Leidenfrost point, they bunch up into small balls of liquid and skitter around, resulting in a huge loss of nanoparticles. Therefore, few nanoparticles are able to act between the roll and the workpiece, so that the lubrication effect of water-based lubricants is reduced markedly. In a similar way, the oil in the 1.0 vol.% O/W emulsion is prone to be burnt at such high temperature, leading to little lubrication effect [40, 41].

4.2 Formation of oxide scale

Generally, a multi-layered oxide scale formed on a steel surface at high temperature is composed of a thin outer layer of hematite (Fe₂O₃), an intermediate layer of magnetite (Fe₃O₄), and an inner layer of wustite (FeO) [42, 43]. The formation of oxide scale comprises two different types of chemical reactions, which are outlined as follows:

It can be seen that the main factors affecting the formation of oxide scale include temperature, oxygen and water.

On one hand, the water in the TiO₂ water-based lubricants has a capability to cool down the temperature of the steel surface, and thus reduce the oxidation rate. On the other hand, the TiO₂ nanoparticles in the lubricants are able to form a protective film which can isolate air, and then reduce the chemical reaction, which should decrease the thickness of the oxide scale. Water lubrication is subjected to being evaporated promptly, and hence limits the cooling effect on decreasing scale thickness. Nevertheless, the chemical reaction between water and steel at high temperatures often produces additional oxide scale, so the

water's effect on decreasing the scale thickness becomes insignificant. With the addition of TiO_2 nanoparticles into water, the dominant factors including temperature and oxygen are both restricted by the cooling effect of the water and the protective film of nanoparticles, respectively. When it comes to the highest concentration of lubricant (8.0 wt.%), in which the water content is relative low so that the temperature of the steel surface arrives at a higher level than that of 4.0 wt.%. Because of this, 8.0 wt.% TiO_2 leads to a slightly thicker oxide scale than that of the 4.0 wt.% TiO_2 lubricant, even though a thicker protective film is formed. 1.0 vol.% O/W emulsion behaves in a similar way to water-based lubricants with the oil being burned at high temperature [40, 41]. A thinner oxide scale will be beneficial for pickling in the practical steel manufacturing and it will also increase the productivity.

4.3 Surface morphology

Fig. 11 shows the surface profile and 3D morphology of rolled steel under dry condition and 4.0 wt.% TiO_2 lubricant after hot rolling at 950 °C. The colour levels in the 3D image represent the height information of the surface, indicated by the colour bar displayed in the image. It can be seen clearly in Fig. 11(a)-(d) that considerable cracks exist on the oxide scale of the rolled strip surface. The surface under dry condition shown in Fig. 11(c) instead possesses deeper cracks than that with 4.0 wt.% TiO_2 shown in Fig. 11(d). To measure the depth of the cracks, line scanning across the flat-area point and the crack point is performed, as shown in Figs. 11(a) and (b). The height difference between the two points on the rolled surfaces are 21.06 and 14.35 μm for dry condition and 4.0 wt.% TiO_2 , as shown in Figs. 11(e) and (f), respectively. In this case, it is obvious that the deeper cracks on the rolled surface are inclined to result in higher surface roughness, and the TiO_2 nano-additive water-based lubricants should create shallower cracks due to the lower rolling force during the deformation process in hot rolling, compared with that of dry condition. The other possible reason is that the TiO_2 nanoparticles may polish the strip surface and the debris can also fill in the surface cracks, acting as polishing and mending effects [35, 44, 45]. A higher fraction of 8.0 wt.%, however, may cause agglomeration of TiO_2 nanoparticles, which would aggravate the friction and wear [46], and thereby increase the surface roughness of the rolled strip.

4.4 Cross section characterisation

The resin/oxide scale interface of the steel rolled at 950 °C with 4.0 wt.% TiO_2 lubricant is shown in Fig. 12. The SEM image shown in Fig. 12(a) and EDS mapping with distribution of element Ti shown in Fig. 12(d) indicate that the scale surface deposits a large amount of TiO_2 nanoparticles, which are finally retained after the instant loss of the lubricant at high temperature. The surplus nanoparticles on the scale surface are deemed to behave as a rolling effect [35, 45, 47], which decreases the COF, and hence reduces the rolling force during the hot rolling process. Moreover, the layer of TiO_2 nanoparticles shown in Fig.

12(d) contributes to forming a protective film, which enables to isolate steel matrix from the air as discussed in Section 4.2.

The resin/oxide scale interface of the rolled steel lubricated by the 4.0 wt.% TiO_2 is further observed as shown in Fig. 13. The nanoparticles intend to penetrate throughout the oxide scale, especially in the crack areas, as can be seen in Figs. 13(a) and (d). This lubrication effect can be defined as a mending effect [35, 44], which is favorable to mending the surface defects and as such reducing the surface roughness of the rolled strip.

4.5 Lubrication mechanisms

As mentioned above, the lubrication mechanisms of TiO_2 nano-additive water-based lubricants acting during hot rolling process can be illustrated in Fig. 14. On one hand, the TiO_2 nanoparticles retained on the roll surface behave as rolling bearing on the flat areas under the applied rolling force to separate the work rolls and steel substrate, thus preventing them from contacting each other directly. The rolling effect of the nanoparticles contributes to the reduction of the abrasive friction, and as such not only polish the rough surface but also decrease the rolling force during the hot rolling process. On the other hand, the TiO_2 nanoparticles are able to spread over the steel substrate to form a protective film, which isolates air resulting in a decrease of oxide scale. Last but not least, the nanoparticles are small enough to fill in the surface defects, producing a mending effect to improve surface finish. Mending hereby is beneficial to alleviate the wear of work rolls in the subsequent hot rolling processes.

5 Conclusions

The lubrication effects of TiO₂ nano-additive lubricants were evaluated by hot rolling tests at 1050, 950, and 850 °C, in comparison to the performances under dry, water lubrication and 1.0 vol.% O/W emulsion conditions. The following conclusions can be drawn from this study.

- (1) The rolling force varies slightly at the rolling temperature of 1050 °C under all lubrication conditions. When the rolling temperature decreases to 950 and 850 °C, the rolling force can be significantly reduced by 6.0% and 8.0%, respectively, when the 4.0 wt.% water-based lubricant is applied, compared to that of dry condition.
- (2) The 4.0 wt.% TiO₂ water-based lubricant produces the best surface finish of the rolled steels under various lubrication conditions, and yields the thinnest oxide scale formed on the rolled steels after hot rolling at 950 and 850 °C.
- (3) Both rolling and mending effects together with formation of protective film dominantly contribute to the lubrication mechanisms of the TiO₂ water-based lubricants.
- (4) The 4.0 wt.% TiO₂ water-based lubricant demonstrates its potential to substitute the traditional 1.0 vol% O/W emulsion as it produces similar rolling force, surface roughness, and oxide scale thickness to those produced by the O/W emulsion.

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Table Captions:

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

Table 2 Chemical compositions of lubricants

Table 3 Experimental data of rolling force (KN)

Figure Captions:

Fig. 1 The flow chart of preparation of TiO₂ nano-additive water-based lubricants.

Fig. 2 The flow chart of hot rolling tests.

Fig. 3 XRD pattern of TiO₂ nanoparticles.

Fig. 4 TEM images of TiO₂ nanoparticles dried from different mass concentrations of water-based lubricants: (a) 0.4 wt.%, (b) 1.0% wt.%, (c) 4.0 wt.%, and (d) 8.0 wt.%.

Fig. 5 The rolling force obtained under various lubrication conditions at 1050, 950 and 850 °C.

Fig. 6 Surface roughness values of the rolled steels after hot rolling at 1050, 950 and 850 °C under various lubrication conditions.

Fig. 7 The thickness of the oxide scale after hot rolling at 1050, 950 and 850 °C under various lubrication conditions.

Fig. 8 The morphologies of the cross sections of the oxide scale formed at 950 °C under lubrication conditions of (a) dry, (b) 0.4 wt.% TiO₂, (c) 4.0 wt.% TiO₂, and (d) 1.0 vol.% O/W.

Fig. 9 The contact angle values of various lubrication conditions.

Fig. 10 Images showing the adhesion of lubricants to the roll surface: (a) water, (b) 1.0 wt.% TiO₂, (c) 2.0 wt.% TiO₂, (d) 4.0 wt.% TiO₂, (e) 8.0 wt.% TiO₂, (f) 1.0 vol.% O/W.

Fig. 11 The rolled surface profile and 3D morphologies at 950 °C: (a, c, e) dry condition; and (b, d, f) 4.0 wt.% TiO₂ lubrication.

Fig. 12 The SEM image (a) and EDS mappings for the resin/oxide scale interface of the steel rolled at 950 °C under 4.0 wt.% TiO₂ lubrication with elements of (b) Fe, (c) O and (d) Ti.

Fig. 13 The SEM image (a) and EDS mappings for the resin/oxide scale interface of the steel rolled at 850 °C under 4.0 wt.% TiO₂ lubrication with elements of (b) Fe, (c) O and (d) Ti.

Fig. 14 The schematic of lubrication mechanism of TiO₂ nano-additive water-based lubricants in hot rolling process.

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

C	Si	Mn	P	Cr	S	N	Nb+V+Ti
0.05	0.02	0.25	0.014	0.01	0.002	0.003	<0.01

Table 2 Chemical compositions of lubricants

Lubricant type	Description
1	Dry condition
2	Water
3	0.4 wt.% TiO ₂ +0.004 wt. % PEI + 10.0 wt.% glycerol + balance water
4	1.0 wt.% TiO ₂ +0.01 wt. % PEI + 10.0 wt.% glycerol + balance water
5	2.0 wt.% TiO ₂ +0.02 wt. % PEI + 10.0 wt.% glycerol + balance water
6	4.0 wt.% TiO ₂ +0.04 wt. % PEI + 10.0 wt.% glycerol + balance water
7	8.0 wt.% TiO ₂ +0.08 wt. % PEI + 10.0 wt.% glycerol + balance water
8	1.0 vol.% O/W emulsion

Table 3 Experimental data of rolling force and standard deviation (KN)

Lubrication conditions	Rolling force at 1050 °C	Standard deviation	Rolling force at 950 °C	Standard deviation	Rolling force at 850 °C	Standard deviation
dry	127.1	6.9	185.8	6.2	245.7	5.5
water	125.2	2.1	183.2	3.1	241.6	3.2
0.4 wt.% TiO ₂	124.4	2.5	182.5	4.1	240.5	0.2
1.0 wt.% TiO ₂	123.1	2.3	182.4	3.8	238.2	0.8
2.0 wt.% TiO ₂	121.9	2.9	178.5	3.7	227.0	4.3
4.0 wt.% TiO ₂	122.0	1.2	174.6	4.2	226.1	4.5
8.0 wt.% TiO ₂	123.1	2.2	175.5	4.3	228.2	4.6
1.0 vol.% O/W	121.3	5.8	176.0	1.8	230.4	2.1

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