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Effect of extreme pressure additives on the deformation behavior of oxide scale during the hot rolling of ferritic stainless steel strips

Liang Hao
University of Wollongong, lh421@uowmail.edu.au

Zhengyi Jiang
University of Wollongong, jiang@uow.edu.au

Xiawei Cheng
University of Wollongong, xiawei@uow.edu.au

Jingwei Zhao
University of Wollongong, jzhao@uow.edu.au

Dongbin Wei
University of Wollongong, dwei@uow.edu.au

See next page for additional authors

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Abstract

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Keywords

steel, extreme, pressure, additives, deformation, behavior, effect, oxide, strips, scale, during, hot, rolling, ferritic, stainless

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Authors

Liang Hao, Zhengyi Jiang, Xiawei Cheng, Jingwei Zhao, Dongbin Wei, Laizhu Jiang, Suzhen Luo, Ming Luo, and Li Ma

Effect of Extreme Pressure Additives on the Deformation Behavior of Oxide Scale during the Hot Rolling of Ferritic Stainless Steel Strips

Liang Hao[1], Zhengyi Jiang[1], Xiawei Cheng[1], Jingwei Zhao[1], Dongbin. Wei[1,2] Laizhu. Jiang[3], Suzhen Luo[3], Ming Luo[3], Li Ma[3]

[1] School of Mechanical, Materials and Mechatronics Engineering, University of Wollongong, Wollongong, NSW, Australia;

[2] School of Electrical, Mechanical and Mechatronic Systems, University of Technology Sydney, Sydney, NSW Australia;

[3] Stainless steel Research Centre, Research Institute (R&D Centre), Baoshan Iron & Steel Co., Ltd., Shanghai, China

Abstract: High-speed steel (HSS) materials are universally used as work rolls for the hot rolling of stainless steels. Their use has increased the output of the rolling mill and decreased roll material consumption and grinding. Sticking defects often occurs, however, during the hot rolling process. In this paper, extreme pressure (EP) additives were dropped on HSS samples at high temperature. Zinc dialkyl dithio phosphate (ZDDP) was chosen as the most effective EP additive by scratch tests on the HSS samples. In order to determine the optimum proportion of ZDDP in the lubricant, two reduction rates were tested on a Hille 100 experimental rolling mill by hot rolling ferritic stainless steel 445J1M at five different concentrations of ZDDP. The mechanism of EP additive action during the hot rolling process was also investigated. By analyzing the deformation behavior of the oxide scale of samples after hot rolling using different proportions of ZDDP, it was found that 20% ZDDP in the lubricant is the preferred concentration for industrial application.

KEY WORDS: Extreme Pressure Additives; Steel; Rolling; Adhesion; Friction Mechanisms

INTRODUCTION

Ferritic stainless steels, containing little or no nickel, have properties such as excellent resistance to oxidation and corrosion, and lower cost. There are number of problems which frequently arise in the manufacture of stainless steels. One of these is surface defects such as scoring, scratching and sticking. Scoring means that the metal is removed either through cutting or plastic deformation and the surface appears grooved or ridged. Scratching is the metal damage due to the contact of the metal with small abrasive particles (1). Sticking refers to the phenomenon where the fragments of the rolled materials are detached and get stuck to the work roll surface (2, 3). This is a defect which frequently occurs during the hot rolling process, deteriorating the surfaces of both the rolls and the rolled materials (4). It has been found that the sticking does not occur in the surface region containing oxides, but most likely in the surface region without oxides (Ha et al. (5)). This indicates that the resistance to sticking is increased by the increase in the surface hardness brought about by a large amount of oxides being formed in the surface region. Sticking occurs most frequently for those steels containing higher levels of chromium. There was a critical value which was found to be 3 μm of the scale thickness in the suppression of the sticking phenomenon (Jin et al. (6)). It has also been found that the addition of Zr, Cu, or Si has a beneficial effect on the sticking resistance, while the addition of Ni does not have a large effect on the sticking (Ha et al. (7)). In Si-rich steel, Si oxides form first in the initial stage of the high temperature oxidation, and act as initiation sites for Fe-Cr oxides. This accelerates the formation of Fe-Cr oxides, and thus decreases the sticking. Once sticking defects occur, the operators must grind the sticking areas on the strip surface, which prolongs the processing time and increases the cost.

Although extensive research has been conducted on sticking, measures which are both effective and practical for industrial manufacturers have not been put forward. The application of lubrication onto the work rolls in hot strip mills leads to cost reductions (roll material, energy) and improves product quality (surface defects) (8, 9). The first essential in successfully using a lubricant for hot rolling is the formulation of the lubricant. Of all components in the lubricant, extreme pressure (EP) additives play a key role in preventing sticking and scratch defects. The additives form extremely durable protective films by thermo-chemically reacting with the metal surfaces. The films can withstand extreme temperatures and mechanical pressures and minimize direct contact between surfaces, thereby protecting them from scoring and seizing(10).

The current paper reports on two types of experiments, one for the selection of the effective EP additive, the other in order to determine the optimum proportion of the selected EP additive to be added to the lubricant. The first experiment consisted of dropping a series of EP solutions on heated HSS specimens and conducting scratch tests on the films which formed on the samples. The second experiment comprised hot rolling processing of ferritic stainless steel 445J1M on a Hille 100 experimental rolling mill. These experiments were conducted with different proportions of the selected EP additive in the commercial lubricant. The deformation behavior of the oxide scales on 445J1M was observed in order to determine the optimum proportion for industrial practice. The microstructure and surface morphology after tests were analyzed using scanning electron microscope (SEM). The scratch resistance of the reaction films on the HSS samples was tested using a Revetest Xpress Scratch Tester to select the effective EP additive. Hot rolling tests of ferritic stainless steel 445J1M were carried out on a 2-high Hille 100 experimental rolling mill following by observation of the deformation behavior of the rolled strips using SEM.

EXPERIMENTAL

Tests on the Selection of EP Additives

Because the lubricant is sprayed on the work rolls in the actual rolling process, the EP additive selection tests were conducted by dropping different types of EP additives on the heated HSS (work roll material) specimens. The HSS specimens, the chemical compositions of which are shown in Table 1, were cut into cubes with the dimensions of 10×10×5 mm^3. Only one of the broad faces of each sample was ground and polished up to 1 μm diamond suspension, and the remaining faces were ground using 1200 grit sand paper. The samples were ultrasonically cleaned in acetone and alcohol prior to testing (10).

Because of heat transfer from rolled products, and deformation and friction heat, the rolls are heated to 600 ℃ (11). In order to do this, the experiments were conducted in a muffle furnace where the samples were heated to 600 ℃ for 2 h. A series of EP solutions was dropped through a copper tube inserted into the furnace, which simulated the industrial conditions where lubricants are directly sprayed on heated work rolls. The EP additives used were dibenzyl disulphide, dibenzyl sulphide, di-tert-butyl disulphide (12, 13), chlorinated paraffin (chlorine content 43 wt. per cent) and zinc dialkyl dithio phosphate (ZDDP) (14-16). 0.5% EP solutions were used in this study. This represents the typical weight percentage of sulfur or chlorine in a formulation. Water was also applied as a comparison.

Tests to determine the optimum proportion of the EP additive

After the selection of the effective EP additives, hot rolling tests were carried out. The chemical compositions of 445J1M are listed in Table 2, and the specimens were cut into the dimensions of 400×100×10 mm^3, heated to 1100 ℃ for 30 min in a high temperature electric resistance furnace. Two rolling reduction ratios (15 and 30%) of the specimens were employed. The industrial lubricant (1C321) solution was kept at 0.5% (volume ratio of the lubricant to distilled water), and proportions of ZDDP from 0% to 50% (volume ratio of the ZDDP to the lubricant) were added. For each test, a 200 ml lubricant solution was sprayed on the roll surface before hot rolling, and acetone was used to clean the rolls during the interval. The rolled specimens cooled down to room temperature in air.

Analysis Methods

The microstructures of the tested HSS samples and the deformation behavior of the oxide scales on rolled samples were examined using a JEOL LV scanning electron microscope (SEM) equipped with energy- dispersive spectrometry (EDS) analysis. After dropping the EP solutions to the surfaces of the samples, scratch tests were performed by scratching the reactant films with an indenter to determine the critical load at which failure occurs using a Revetest Xpress Scratch Tester. In order to verify the reproducibility of the results, three scratch experiments were conducted on each sample tested.

RESULTS AND DISCUSSION

Microstructure of High-Speed Steel

Fig. 1 presents the backscattered electron (BSE) image of the polished HSS prior to the experiments. Three different carbides can be distinguished according to different contrasts and morphologies in the BSE microstructure (17), namely the dark slender petal-like zones are V-rich MC carbides, the white long regions are Mo-rich M_6C carbides and the grey zones are Cr- Fe rich M_7C_3 carbides. The EDS analyses of these zones reveal that a certain amount of molybdenum is found in V-rich MC carbides and Cr-Fe rich M_7C_3 carbides, while Mo-rich M_6C carbides contain traces of vanadium.

Surface Microstructure Characteristics after Dropping EP Solutions

Fig. 2 shows the surface microstructure characteristics of HSS samples after dropping different EP solutions. It can be clearly seen that carbides were visible after the tests in all the samples except in the sample testing ZDDP, since the decomposition products of ZDDP covered the surface making the carbides totally invisible. In sulfur-type EP additives (Fig. 2b, c and d), the sulfur element was detected both in the matrix and in the carbides, but its content differed. Analysis using EDS indicated that the sulfur content on the specimens' surface is the highest in di-tert-butyl disulphide (1.96%), less in dibenzyl disulphide (0.71%) and the lowest in dibenzyl sulphide (0.67%). This means that a reaction between the HSS sample and the sulfur-type EP solution has occurred. Unlike the findings from Najman et al. (18) that inorganic sulfur films were produced from the reaction between sulfur-type EP additives and the metal, we found that it is due to the fact that the concentration of the rolling lubricant oil sprayed on the roll surface is very low (less 1%) in industrial applications, and this fails to produce inorganic solid iron-sulfur films. The chlorine element was not detected, however, on the HSS sample after dropping chlorinated paraffin solution onto it. The thermal decomposition products of ZDDP were rich in oxygen, phosphorus and zinc, but had no sulfur (Harrison et al. (19, 20).

Scratch Resistance of the Reactant Films

The scratch test is generally accepted as a reliable and efficient method for the quality assessment of coated surfaces (21). Reactive films were obtained on the HSS samples after dropping EP solutions onto the heated samples. These are very important in protecting the surface quality of rolled strips against sticking defects. Scratch tests were conducted to assess the scratch resistance of the oxidation layers or reactant films. A scratch tester equipped with a Rockwell C diamond stylus (cone apex angle 120°, tip radius 200 μm) was used. A progressive load ranging from 1000 mN to 50000 mN for a length of 6 mm was used in order to obtain the critical load at which failure occurred, and both the acoustic emission (AE) and the friction force were recorded during the tests. A more detailed explanation can be found in (22). A short summary of the result is just present in Fig. 3. The critical loads of the tests dropping water and chlorinated paraffin were comparatively low, only 1816 mN and 2742 mN respectively. This may be because only oxidation occurred. For sulfur-type EP solutions, even though oxidation predominates, a sulfuration reaction between the

HSS and EP additives is expected to occur, because a sulfur element was detected in the samples. The critical load is higher for di-ter-butyl disulphide (15285 mN) than it is for dibenzyl disulphide (3927 mN) or dibenzyl sulphide (2893 mN). This may be because of the higher content of sulfur in the sample. The critical load for the reactant films using ZDDP solution reaches 26818 mN, however, showing the strongest scratch resistance of all.

Deformation Behavior of the Oxide Scales

Compared with other EP additives tested, ZDDP (14, 23) manifested the highest scratch resistance of the EP additives tested. Hence ZDDP was selected as the additive for further hot rolling tests to determine its optimum proportion in the lubricant. Hot rolling experiments of 445J1M were carried out at two reduction rates, 15% and 30% individually with different proportions of ZDDP varying from 0 to 50%. Fig. 4 shows the SEM images of the deformation behavior of the oxide scale on the rolled strips after 10% reduction. It is evident that the oxide scales reveal different deformation behaviors with the addition of different proportions of ZDDP in the lubricant. Without adding ZDDP (Fig. 4a), the oxide scale was crushed into relatively large particles during the rolling process and tended to detach from the matrix, and large gaps between the oxide scales were the regions where the matrix was easily exposed, therefore, the most sticking could occur. When 10% ZDDP was added for hot rolling, large particles were still found, as shown in Fig. 4b. However, the deformation behaviors of the oxide scale were improved with the increase in the amount of ZDDP in the lubricant. As shown in Fig. 4c to f, the oxide scale was rolled into smaller particles and stuck to the matrix. The smaller the particles in the oxide layer after rolling, the more extensive the areas which will be covered. Thus, no large gaps among the oxide layer were observed. Taking factors such as the coverage condition of the oxide scale after hot rolling and cost/performance into consideration, 20% ZDDP is the proportion found by this research to be the most suitable for industrial application.

In order to verify the reproducibility of the results, 30% reduction was also employed for hot rolling tests with different ZDDP concentrations, as shown in Fig. 5. At such a high reduction rate, the oxide layer peeled off and the matrix was exposed when ZDDP was not added (Fig. 5a). The matrix still exposed through the large gaps in the oxide layer when 10% ZDDP was added (Fig. 5b), whereas the oxide scales were rolled into small particles and still covered the matrix with ZDDP concentrations above 20% (Fig. 5c to f). Therefore, it is expected that the sticking problem will be reduced with the addition of ZDDP in the lubricant. 20% ZDDP is still suggested for industrial tests because of the cost/performance benefit.

While sticking problem has been found in ferritic stainless steels and in steels containing higher amount of chromium (6), it is rarely reported in carbon steels, and the sticking areas show no coverage of oxides (5). Therefore, the coverage of oxides on the strip matrix is very important in order to prevent sticking defects. Fig. 6 shows the comparison of the deformation behavior of the oxide scale in the roll bites between non-sticking strips and sticking strips. In hot strip mills, an oxide scale layer is inevitably formed on the steel surface. When strips such as carbon steel which have a relatively thick oxide scale on the surface are rolled, the extension of the oxide scale is capable of covering the matrix (Fig. 6a), producing sticking-free strips. However, when the strips with a thin oxide scale on the surface are rolled, the extension of thin oxide scale fails to cover the matrix (Fig. 6b), resulting in the emergence of sticking defects.

The surface of the oxide scale is made up of complicated asperities. Therefore when the surfaces between rolls and strips are placed in contact in the roll bite, only the tips of the asperities touch (24-26). Rolling forces were applied by rolls to strips through such tips in contact with each other. This compressed and sheared the oxide scale on the strip and its matrix as well. Fig. 7a schematically demonstrates the mechanism of the deformation behavior of the oxide scale without ZDDP film and the microscopic view of the contact interface in the roll bite. It can be seen that the contacts only occurred at some asperities and areas. This means that the oxide scale on the strip was subjected to a non-uniform force, producing the large particles of the oxide scale after hot rolling, as shown in Figs. 4a and 5a. Tse et al. (27) proposed that the anti-wear property of ZDDP is due to the formation of chemically connected networks as a result of pressure-induced cross-linkage of phosphate groups of thermally decomposed ZDDP. This was demonstrated via *in situ* high-pressure and high-temperature infrared (IR) spectroscopy using synchrotron radiation. The experiments showed that ZDDP undergoes substantial decomposition at high pressures (18.4 GPa) and high temperatures (225 ℃) but no hint of the devastation of cross-linkage of phosphate groups. Mosey et al. (28) also found that the anti-wear theory of ZDDP is based on the idea that pressure-induced cross-linking leads to chemically connected networks, which enhances the properties of wear inhibition. Furthermore, the networks remain intact upon release of the pressure, which resists flow of the film out of the contact area. Therefore, tribofilms from thermal decomposition of ZDDP can develop their substantial strength at high-temperature and high-pressure. Fig. 7b schematically demonstrates the mechanism of the deformation behavior of the oxide scale with the ZDDP film and its microscopic view of the contact interface in the roll bite. With ZDDP added to the lubricant for hot rolling, the ZDDP film filled the surface valleys and provided separation between the roll and the strip. Rolling forces were applied by rolls through the ZDDP film to the oxide scale on the strip. Consequently, the oxide scale was subjected to the uniform force, producing small particles of oxide scale after hot rolling, as shown in Figs. 4c-f and 5c-f (the extensive areas of the strip matrix are covered by the small oxide particles). As shown in Figs. 4b and 5b, the large particles of the oxide scale were observed when 10% (volume ratio of ZDDP to the lubricant) ZDDP was added into the lubricant. This may be because the ZDDP film was too thin and failed to separate the surfaces between the roll and the strip. In addition, the rougher surface of the rolls, the thicker ZDDP films may be required to separate the strip from the roll.

CONCLUSION

In order to prevent the sticking defects during the hot rolling of ferritic stainless steel 445J1M, experiments for selecting the most effective EP additive and determining its optimum proportion in the lubricant were conducted. ZDDP exhibits the greatest scratch resistance of all EP additives tested and was, therefore, chosen as the preferred additive in the lubricant. The hot rolling tests of 445J1M were carried out at two reduction rates and different ZDDP concentrations. The results indicate that 20% ZDDP in the lubricant is suggested for industrial trials.

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