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The Influence of a Novel Inorganic-Polymer Lubricant on the Microstructure of Interstitial-Free Steel during Ferrite Rolling

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Abstract: A novel polyphosphate lubricant was used and evaluated during hot (ferrite) rolling of an interstitial-free (IF) steel. The texture evolution of these rolled IF steels have been examined by means of X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) measurements. The polyphosphate lubricant shows an improved lubrication performance in terms of the texture optimization compared with lubricating oil and with unlubricated conditions. The γ-fiber texture is enhanced, and less shear texture is produced. This microstructure is responsible for enhanced drawability of ferrite rolled IF steels. The very high thermal stability of the polyphosphate enabled its use at very high temperatures (from 700 to 800 °C). Rolling temperature exerted limited influence on the resulting rolling texture evolution. The polyphosphate lubricant stabilizes the surface texture and reduces the gradient of shear texture through the thickness. The in-grain shear bands are reduced significantly (48.5%) compared with the unlubricated condition. Measured grain orientations indicate that the favorable texture of {111}<112> along the γ-fiber is developed while the undesired α-fiber texture of {001}<110> is effectively suppressed.

Keywords: polyphosphate; lubricant; ferrite rolling; microstructure

1. Introduction

Ferrite rolling has been extensively studied with different kinds of steels, such as interstitial-free (IF) steel [1–4], ultra-low carbon steel [5], low carbon steel [6–8], and stainless steel [9]. Many difficulties in these studies arise during hot rolling due to the phase transformation from austenite to ferrite, such as a detrimental shear texture appearing throughout the rolled strip thickness during the rolling process [10]. The formation of rolling texture is dominated by factors such as chemical compositions and processing parameters [11]. Normally, a ferrite rolled specimen shows a shear gradient from the rolled surface to the interior of the strip. The sheared surface layer is subject to up to 2.5 times the strain compared to the specimen as a whole [10]. Oil-based lubricants applied during hot and ferrite rolling reduce the strain gradient, improve the surface quality, and reduce the cost of production [12,13]. Such lubrication can also reduce the rolling force and torque, save energy, and extend the roll service life. The shear action is responsible for developing the <100> texture in the sheared layers of the ferrite rolled strips while preventing the formation of compression textures with a {111} component [14].

Since the application of lubrication during ferrite rolling of IF steel can improve significantly the material drawability, many investigations have been focused on this research area [10,15,16]. Barrett [10] reported application of mineral and ester based oil during the ferrite rolling of IF steel

and pointed out that the ester-based oil showed a better lubrication performance by suppressing the detrimental shear texture throughout the rolled strip thickness. He also addressed the economic benefits derived from a proper use of lubricants in the industry. Zhao et al. [15] concluded that a large reduction and higher rolling temperature combined with lubrication during ferrite rolling can yield a better drawability. The lubrication is responsible for improving the microstructure in terms of reducing the number of in-grain shear bands and producing more favorable texture during ferrite rolling of IF steel [16]. Moreover, new data processing techniques are required in order to reduce the time taken to perform microstructural analysis, reduce human error, and give repeatable results. Digital image processing theoretically offers a solution to explore microstructural aspects in metallographic terms. Some works [17,18] have reported methods to extract microstructural information and understand process evolution from the polished and etched surfaces of metal materials obtained through quantitative microscopy.

Proper use of lubricant affects both the ferrite rolling process and the quality of the final product. The lubricants conducted in these related works mainly involve liquid lubricating oils (ester and mineral oils). However, at a higher temperature, the oil is burnt, and the oil film between the roll and strip is destroyed, so that lubrication performance is reduced. It is uncertain if an effective lubrication of liquid mediums can be achieved on the sliding interfaces since a number of oil-based lubricants may degrade seriously due to their high sensitivity to temperature [19]. At the same time, replacing oil with more environmental-friendly water-based lubricant results in a reduction of hazardous waste by-products [20]. The polyphosphate based lubricant described here, which can form a tribochemical film with steel, is a potential candidate for ferrite rolling of IF steels at high temperature and load [21]. It is a water-based inorganic-polymer lubricant. It melts at high temperature and is able to form a continuous lubrication film with a glassy state, which can stabilize the contact conditions. The chemical production from the reaction between lubricant and steel improves the tribological property as well. On the consideration of other lubricants such as glass powder or nano-powder, it improves the contact conditions from the lubrication mechanism of the rolling/ball-bearing effect, protective film/tribofilm, mending effect, polishing effect, synergistic effect, third-body effect, etc. However, it is mainly on a physical level instead of chemical/physical actions. The microstructural evolution effects of polyphosphate lubricants on IF steel rolling have received little attention in the literature to date. The microstructural evolution mechanisms during ferrite rolling with polyphosphate based lubricants need to be investigated further.

In this paper, the influence of a polyphosphate lubricant on the microstructural evolution of IF steel after ferrite rolling was studied as a function of different rolling and lubrication conditions. The evolution of macrotexture and shear bands, as well as information on individual grain orientation were measured and analyzed by means of X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) measurements.

2. Materials and Methods

Three lubrication conditions were evaluated during ferrite rolling. The performance of an inorganic-polymer lubricant was studied by comparing it with a conventional lubricating oil (Quaker HB28) and with unlubricated conditions. The water-based inorganic-polymer lubricant (polyphosphate) was composed of sodium polyphosphate and potassium dihydrogen phosphate, whereas the lubricating oil was a specialized commercial ester based oil with additives. The chemical composition of the IF steel strip can be found in Table 1. Ferrite rolling of the IF steel strip under different lubrication conditions was conducted on a 2-high Hille 1000 experimental rolling mill at 700 and 800 °C. The rolling reduction was subjected to 25% and 40% with a single rolling pass. The original strips (5 mm) were rolled to a final thickness of 3.75 mm or 3 mm, which depended on the reduction. The rolled IF steel strips were quenched by water immediately after the ferrite rolling process in order to eliminate possible recrystallization. Prior to XRD measurement, specimens were cut from the ferrite rolled strips with dimensions of 25 mm × 25 mm × 3 mm, and then one broad surface of the specimens was ground

by SiC abrasive and polished and finished with 1.0 μm diamond paste in order to obtain a flat and smooth surface free from metallographic damage.

Pole figure (PF) measurements of the ferrite rolled specimens were carried out on a PANalytical X'pert-PRO X-ray goniometer (PANalytical, Eindhoven, the Netherlands) in order to calculate the corresponding orientation distribution function (ODF). RD was the rolling direction, and the TD and ND presented the transverse direction and normal direction, respectively. The Cu Kα radiation was produced at 45 kV and 40 mA for the texture measurement. The defocusing of a standard iron specimen was measured for the correction of the specimen background. Incomplete pole figures of {110}, {200}, and {211} were measured for all the specimens, which allowed the analysis of textural information of specimens averaged over a large area/volume (macrotexture). The ODFs were then calculated by the X'pert Data Collector (v2.0, PANalytical, Eindhoven, Netherlands) and MTEX from the MATLAB toolbox (R2006b, MathWorks, Natick, MA, USA) with a background correction.

The microtextures of ferrite rolled IF steel samples were studied by EBSD measurement (Oxford Instruments Plc., Abingdon, UK). The ferrite rolled IF steel strips were cut into small rectangular specimens that were parallel to the normal direction–rolling direction (ND–RD) plane with a size about $10 \times 5 \times 3$ mm^3. The specimens were then metallographically ground and polished. Due to the lower hardness and good drawability of IF steel compared with other carbon steel, an additional electrolytic polish was conducted on the cross-section of specimens from the mid-thickness to the sub-surface at ambient temperature in order to eliminate the deformation layer associated with metallographic preparation. The details of the electrolytic polish are shown in Tables 2 and 3.

Table 1. Chemical composition of disc (interstitial-free (IF) steel) (wt. %).

Strip Material	C	P	Mn	Si	Ni	Cr	Ti
IF Steel (%)	0.0027	0.011	0.14	0.02	0.052	0.11	0.067

Table 2. Chemical composition of the electrolyte for electrolytic polish of electron backscatter diffraction (EBSD) specimens.

Chemical Composition	Perchloric Acid	Distilled Water	Ethanol	Butoxyethanol
Volume (mL)	55	65	510	70

Table 3. Parameters for the electrolytic polish of ferrite rolled IF steels.

Area (cm^2)	Temperature (°C)	Voltage (V)	Flow Rate	Time (s)
1	22	50	16	60

The fine scanning electron microscope (JEOL, Tokyo, Japan) used for EBSD was focused accurately at high magnification (55,000×). The magnification was then reduced to 100× for EBSD measurement. An area of 2000×1500 pixels (1.2×0.9 mm^2) was used for all the EBSD measurements. The scanning step size was 0.6 μm with a tilt angle of 70°. The total scanning time for one measurement was around 20 h.

3. Results

3.1. Evolution of Through-Thickness Macrotexture

The ODFs were calculated in order to study the evolution of through-thickness orientations under various lubrication conditions. The γ-fiber mainly included {111}<110> and {111}<112>, and the α-fiber texture comprised {001}<110>, {112}<110>, and {111}<110>. The Goss texture of {111}<001> was also detected. All of the orientations discussed above can be represented in the φ2 = 45° section.

The texture intensities of the strip surface and centerline along the α-fiber and along the γ-fiber, and Goss texture, as a function of the lubricating conditions are shown in Figure 1. The friction between contact pairs (roll and steel surfaces) introduced a clear shear strain on the steel surface [22]. A strain gradient was therefore developed throughout the strip thickness, which gave rise to texture variations.

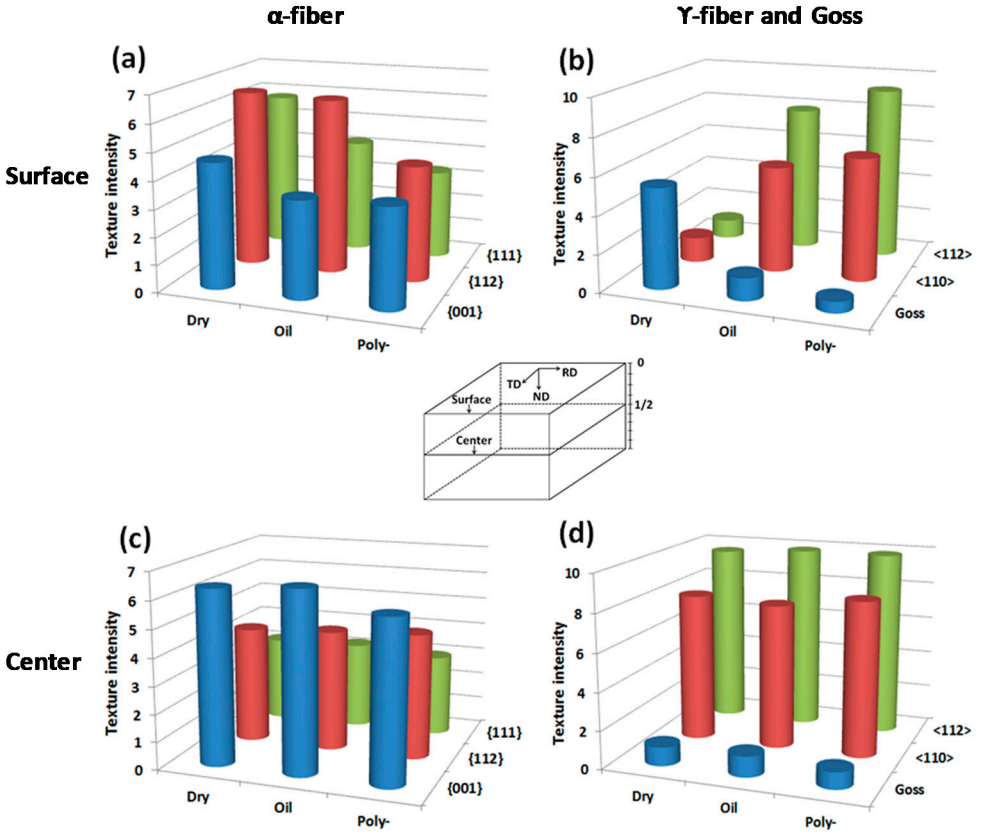

Figure 1. The influence of lubrication conditions on the surface and centreline textures of the ferrite rolled strip: (**a**) α-fiber and (**b**) γ-fiber and Goss texture of surface layer; (**c**) α-fiber and (**d**) γ-fiber and Goss texture of center layer under different lubrication conditions with rolling reduction of 40% at 700 °C.

3.2. *Effect of Rolling Temperature on Macrotexture with Polyphosphate Lubricant*

Figure 2 shows the influence of rolling temperature on the texture of IF steel samples subjected to 40% reduction under polyphosphate lubrication condition. The influence of temperature on the texture was limited between 700 and 800 °C. The intensive orientations at 800 °C were similar as those at 700 °C with α-fiber, γ-fiber, and Goss texture. Normally, the viscosity of lubricating oil drastically decreases from 40 to 100 °C. It is likely that the oil-based lubricant will be burnt out completely at high temperatures [23]. Compared to lubricating oil, polyphosphate lubricant possessed a superior performance to compress the shear texture at a higher temperature.

3.3. *Effect of Rolling Reduction on Macrotexture with Polyphosphate Lubricant*

Figure 3 shows the effect of rolling reduction on the texture through the thickness. From the literature [10], an increase of reduction during rolling without lubrication causes an increase of the shear texture. Increasing rolling reduction is also responsible for the sharpness of the texture formation [24]. However, the results were quite different in our case with the polyphosphate lubricant. The orientation distribution of shear texture changed little when the reduction increased from 25% to 40% (Figure 3a,b). Moreover, the increase of the reduction resulted in a further suppression of the orientation of the shear texture ({111}<001>) and improved the orientation of {111}<112> on the γ-fiber. Consequently,

the drawability could be improved at the central layer, as shown in Figure 3c,d. The results confirmed that polyphosphate lubricant was extremely effective in preventing the formation of shear texture with increasing rolling reduction. It also gave some improvements with respect to through thickness texture formation during ferrite rolling.

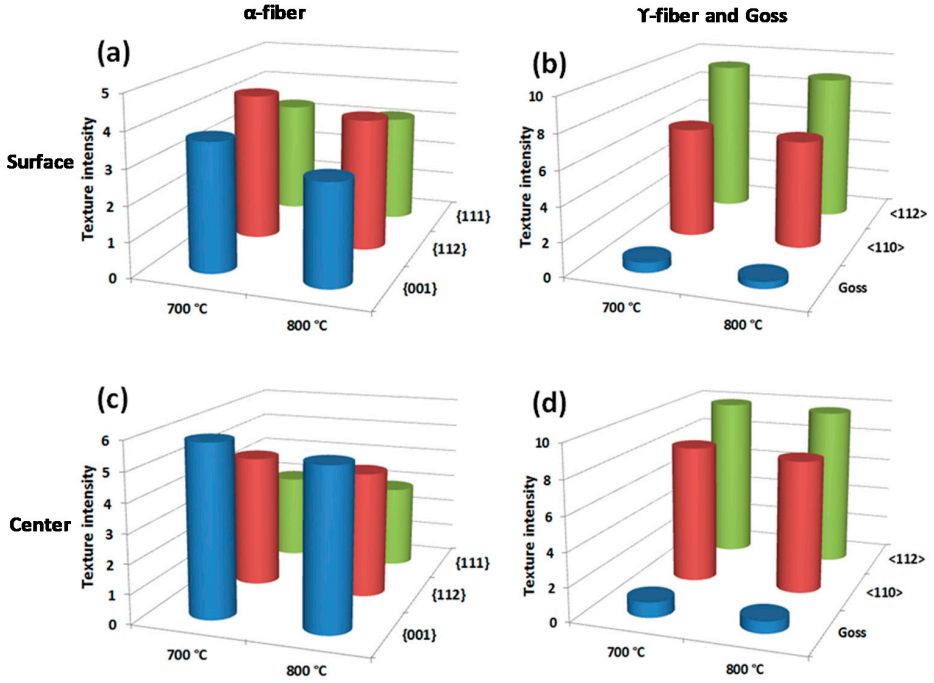

Figure 2. The influence of rolling temperature on the surface and centreline textures of the ferrite rolled strip: (**a**) α-fiber and (**b**) γ-fiber and Goss texture of surface layer; (**c**) α-fiber and (**d**) γ-fiber and Goss texture of center layer with rolling reduction of 40% at 700 and 800 °C with polyphosphate lubricant.

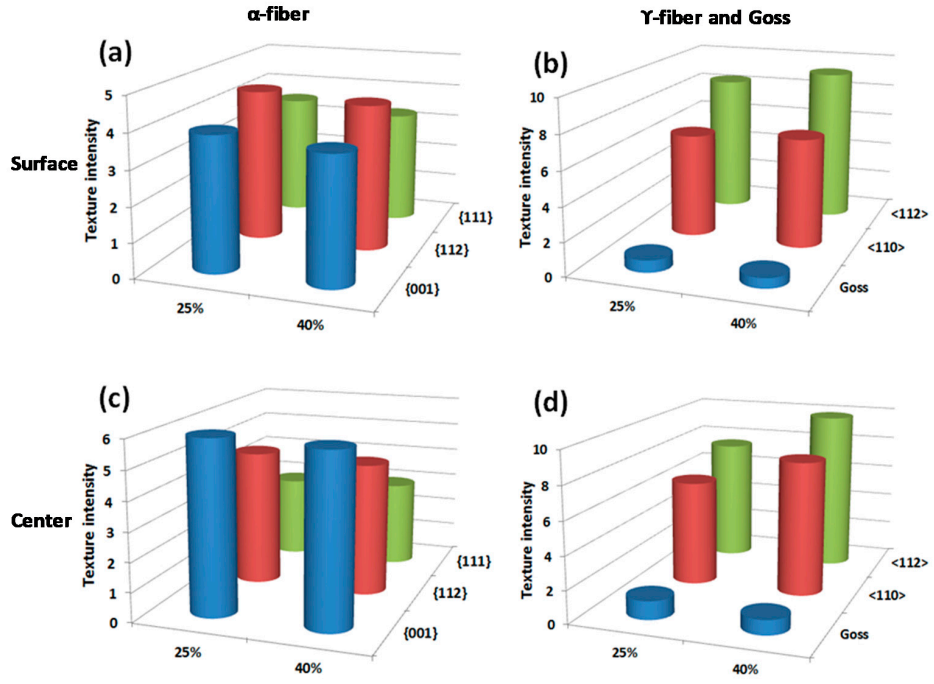

Figure 3. The influence of rolling reduction on the surface and centreline textures of the ferrite rolled strip: (**a**) α-fiber and (**b**) γ-fiber and Goss texture of surface layer; (**c**) α-fiber and (**d**) γ-fiber and Goss texture of center layer with rolling reduction of 25% and 40% at 700 °C with polyphosphate lubricant.

3.4. Shear Band Evolution

The deformation microstructure of the cross-section clearly showed the in-grain shear bands after rolling (Figure 4). As reported by previous work [25], these shear bands presented fish bone structures at angles around 30° to 35° to the rolling direction (horizontal). The similar in-grain shear bands can be observed in detail in Figure 4b. The in-grain shear bands could be found under all the lubrication conditions, as shown in Figure 4a,c,d. Polyphosphate lubricant showed a pronounced effect on the reduction of in-grain shear bands by a fraction of 48.5% compared with the unlubricated condition.

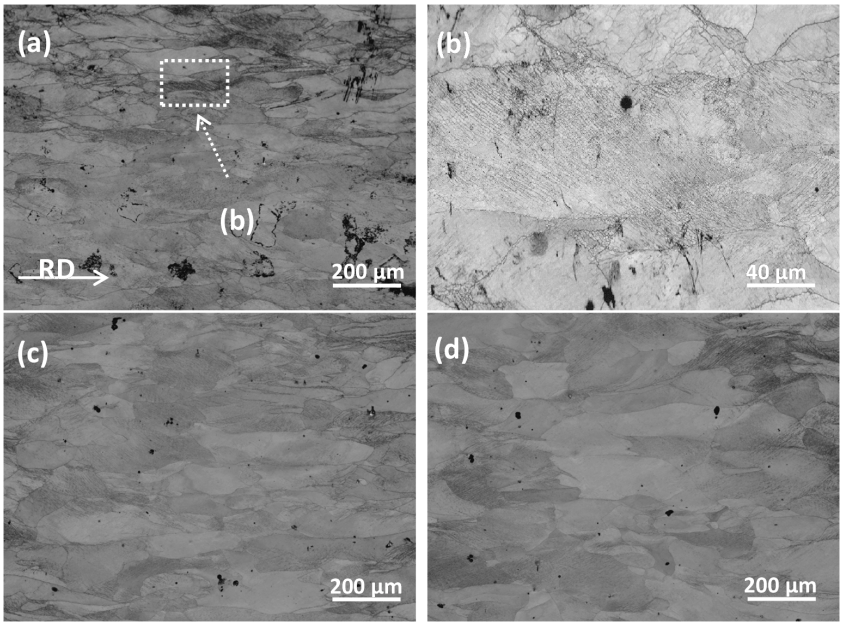

Figure 4. Band contrast images of ferrite rolled IF steels with (**a**) dry, (**b**) the magnified shear band in (**a**), (**c**) lubricating oil, and (**d**) polyphosphate lubricant conditions at 700 °C with 40% rolling reduction.

3.5. Microtexture Analysis

3.5.1. Influence of Polyphosphate Lubricant on the Misorientation Angle Distribution

Figure 5 compares misorientation angle distribution of the grain boundaries of ferrite rolled IF steel samples under different lubrication conditions by EBSD measurement. Misorientation is defined as the crystallographic orientation relationship between two neighboring grains in the same phase [26]. It is critical to the material's properties, particularly to the deformation behaviors. In the case of ferrite rolling, the misorientation of rolled IF steel showed a significant number of low angle boundaries in the microstructure. Most of the misorientations were located at angles areas below 10°, as shown in Figure 5a–c.

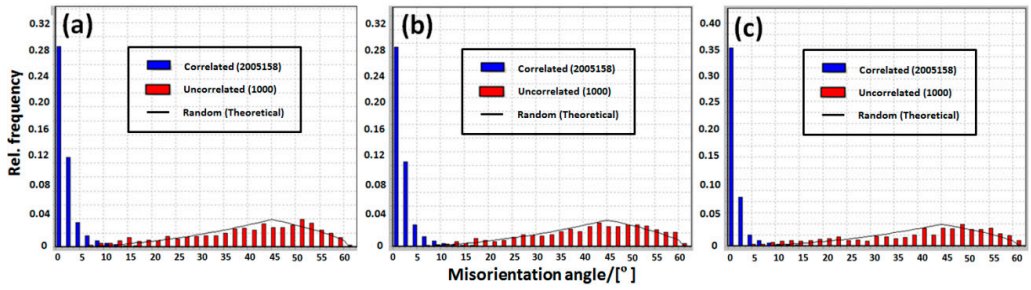

Figure 5. Misorientation angle distribution of the grain boundaries under (**a**) dry, (**b**) lubricating oil, and (**c**) polyphosphate lubricant conditions at 700 °C.

3.5.2. Microtexture Formation

As shown above, the polyphosphate lubricant successfully suppressed the shear texture and improved the drawability of IF steels after ferrite rolling. The ODFs were calculated via individual grain orientation from EBSD results in an area of 1.2 × 0.9 mm^2 from the strip cross-section under different lubrication conditions, as shown in Figure 6. To better understand the location of important deformation texture fibers in Euler space, Figure 7a,b shows the ideal orientation of <110> and <111> fiber tubes after ferrite rolling with polyphosphate lubricant. The projection of the ideal orientations of <110> and <111> fiber tubes on the Φ-φ1 plane matched well the ODF (φ2 = 45°) distribution of the test specimen. Therefore, the calculation was trustable for the investigation of the microtexture at the specific locations. Figure 7c shows the orientations and texture intensity of the ODFs along the α-fiber and γ-fiber (Φ = 54.7°). Nearly all the texture intensity under unlubricated condition was great along the α-fiber, especially on the orientations of {114}<110> and {111}<110>. A large number of grains with an undesired texture could be found along the α-fiber with {001}<110> component under unlubricated condition. In contrast, the polyphosphate lubricant developed a favorable texture of {111}<112> with an intensity of 6.54 along the γ-fiber, while the favorable orientation was weak under the unlubricated condition. The increase of γ-fiber intensity (the orientation of {111}<110> and {111}<112>) was likely favorable to the drawability of the ferrite rolled IF steel samples. Polyphosphate lubricant significantly improved the texture after ferrite rolling. As for the lubricating oil case, its contribution to the required texture formation was intermediate to that of the dry and polyphosphate lubricated conditions.

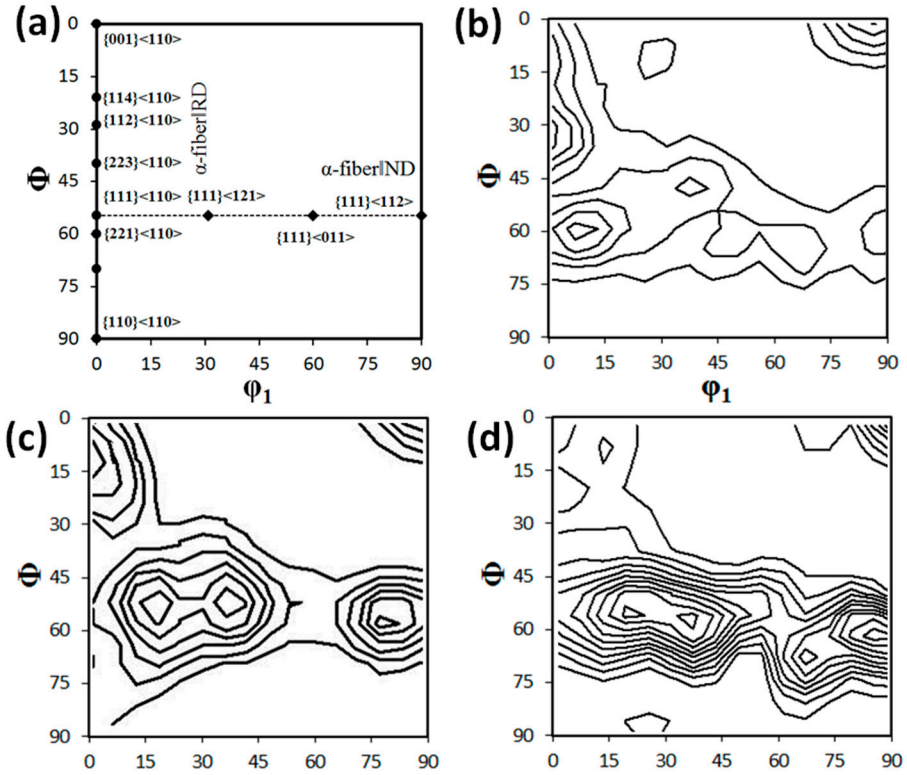

Figure 6. φ2 = 45° orientation distribution function (ODF) sections of (**a**) the ideal body-centered cubic fibers and under conditions of (**b**) dry, (**c**) lubricating oil, and (**d**) polyphosphate lubricant.

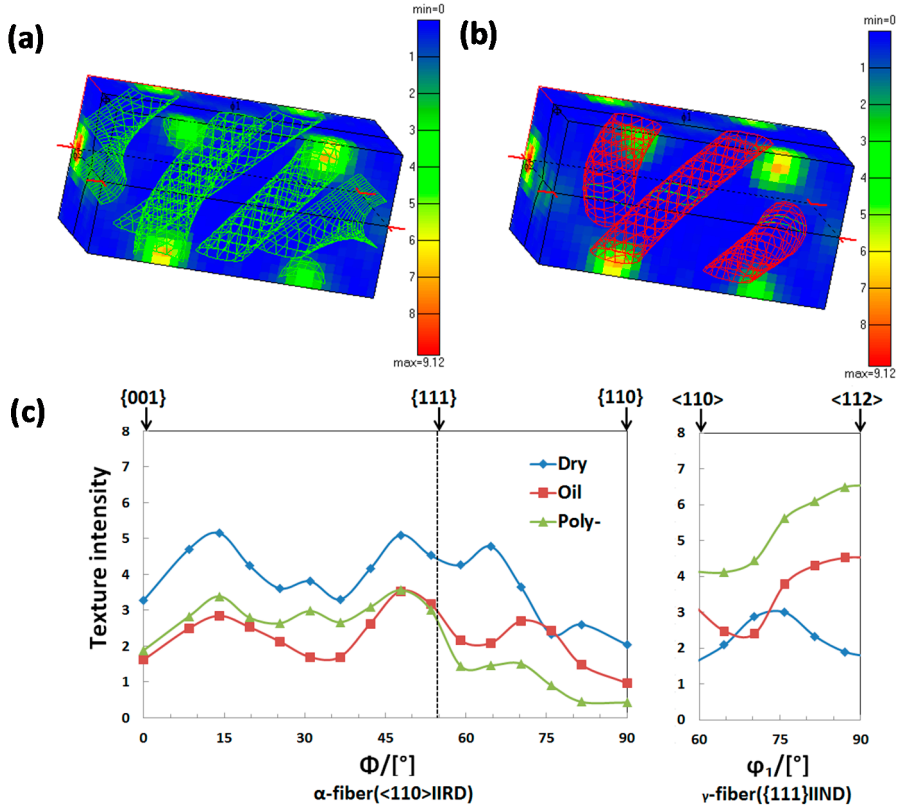

Figure 7. Ideal orientation of (**a**) <110> and (**b**) <111> in Euler space and (**c**) texture intensity along the α-fiber and γ-fiber at 700 °C.

4. Discussion

Compared with other lubricant such as synthetic oil, the polyphosphate lubricant is proposed for high temperature applications, such as hot/warm rolling, forging, and extrusion. The lubrication mechanism is that the polyphosphate lubricant melts at high temperature and acts on the interfaces between the tool and workpiece to improve the contact conditions. It also shows a potential to prolong the tool life and improve the surface quality due to its excellent lubrication performance. Ray and colleagues [5,14,27,28] investigated the texture obtained from hot and ferrite rolling. Their work summarized the important orientation for rolling texture and provided a good reference for further study of the rolling texture. The Goss texture is responsible for the deterioration of drawability [15,29,30]. As reported by Lee [31], the Goss texture remained even after annealing. However, it can be effectively eliminated when lubrication is applied. The control of Goss texture during ferrite rolling is critical for the deep drawability of the final rolled product. The texture intensity on the surface and central layers was totally different under various lubrication conditions in the present work. On the surface layer, a strong shear texture of {111}, {112}, and {001} on the α-fiber could be observed under dry conditions, while both oil and polyphosphate lubricants showed some effect on the reduction of α-fiber texture. The lubrication influence on this texture was minor. The corresponding texture on the γ-fiber did change significantly under different lubrication conditions. The texture intensity of Goss orientation was significantly reduced by lubricating oil, as shown in Figure 1b. The polyphosphate lubricant enhanced the texture on the γ-fiber texture and reduced the Goss orientation. As reported in reference [10], shear texture can be avoided if the friction coefficient is less than 0.15. This indicates that a uniform texture throughout the thickness would not be achieved until the friction decreased to a certain level, and that lubrication would have a great impact on the texture formation. As for the texture of the center layer, the polyphosphate lubricant and lubricating oil did not exert notable

influence. The typical ferrite rolling texture could be observed under all lubricating conditions. It was clear that shear deformation was the dominant factor in the texture formation during ferrite rolling.

As discussed in the previous publication from our research group [32,33], molten polyphosphate lubricant reacts with the iron surface to form a hierarchical tribofilm. The polar molecules bond to the steel surface with different chain lengths. The boundary film with a thickness of around 100 nm not only reduces the friction and wear on the surface during the ferrite rolling, but also eliminates the shear strain of the ferrite rolled IF steels. Barrett et al. [10,34] developed a schematic showing the influence of different liquid lubricants on the shear strain through-thickness. Without lubrication (dry condition), very severe shearing occurs, with only a small central region experiencing plane strain conditions. In contrast, the specimens rolled with lubrication shows only slight shearing, with the ester oil giving better results than the mineral oil. After the study of polyphosphate lubricant effect on the through thickness shear texture, a new curve can be added into the schematic to show the polyphosphate lubricant performance (Figure 8). The polyphosphate lubricant showed better shearing resistance than the liquid lubricants.

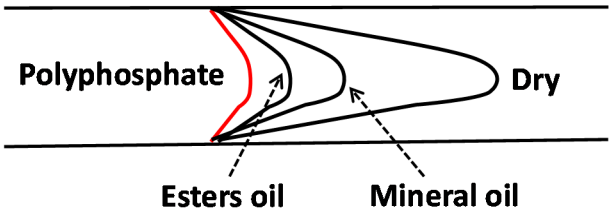

Figure 8. Schematic of shear strain profile under different lubrication conditions including polyphosphate lubricant effect on the ferrite deformation of IF steel [10,30].

Engler et al. [30] reported that a lower finishing temperature developed more deformation, strengthened the hot-band texture, and enhanced the texture gradient [35]. The results indicate that polyphosphate lubricant can maintain good lubrication performance suppressing the shear texture at high temperatures. Barrett [10] reported that the lubricating effect of mineral oil was greatly reduced at 800 °C, resulting in a serious shear texture below the surface of the strip. Four times amount of lubricating oil was required on the rolls to equivalent lubrication to that achieved at 700 °C. In contract, the polyphosphate lubricant is quite stable at such high temperatures, since it produces a molten glassy state with a high viscosity, which provides a good lubrication performance in the ferrite temperature range [21,32].

Shear bands often associate with the formation of texture [36]. The in-grain shear bands are responsible for the formation of Goss texture [37]. Barnett [38] pointed out that the shear bands affect the internal structure of most deformed ND fiber grains during the ferrite rolling of IF steels. In the ferrite temperature range, the in-grain shear bands in the IF steel seems unaffected by the rolling temperature [25]. However, the local flow tendency of IF steel during ferrite rolling depends on the strain rate sensitivity [39]. When lubrication is applied, the strain rate sensitivity can be eased as a result of the boundary film formation between contact surfaces. A reduction of strain rate sensitivity will decrease the fraction of in-grain shear bands within the ferrite rolled IF steel.

The lubrication effect on the reduction of shear bands is shown in Figure 9. The severe shear stress in the roll bite is produced by the intense friction between the roll and strip. A pronounced direction transition can be found for the deformed micro-structure through the strip thickness. Moreover, the microstructure of the strip surface is quite similar to that at the strip center for the rolled strips with polyphosphate lubricant. Compared to the unlubricated condition, both oil and polyphosphate lubricants showed pronounced influence on the reduction of the in-grain shear bands during ferrite rolling. The polyphosphate reduced the fraction of in-grain shear bands by 48.5%, which was 16.2% more than the ester oil. These results also confirmed that the polyphosphate lubricant enhanced the deep drawability of IF steel after ferrite rolling. The characteristics of misorientation angle

distribution showed a slight difference for different lubrication conditions. Interestingly, compared to the unlubricated condition, the misorientation angle between 3 and 10° was slightly reduced after applying polyphosphate lubricant. However, the lubricant had no significant effect on the misorientation angle during ferrite rolling. It can be concluded that the application of polyphosphate lubricant had little effect on the small misorientation angle below 10° during ferrite rolling.

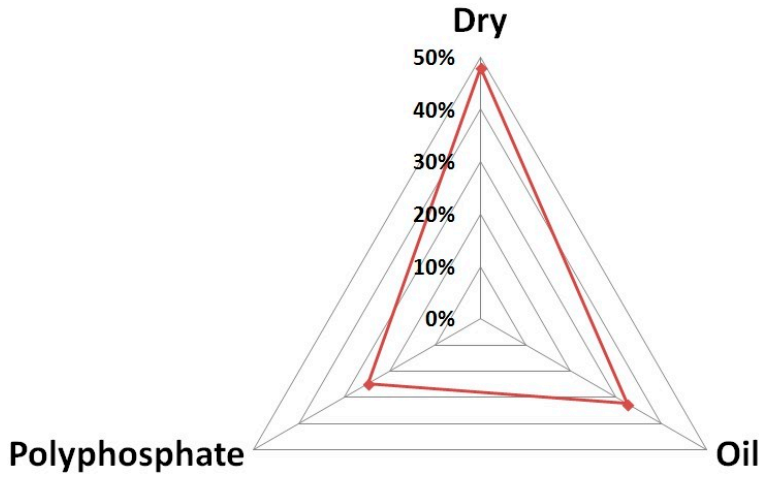

Figure 9. Fraction of grains in the detected area (1.2 × 0.9 mm^2) containing in-grain shear bands for different lubricants at 700 °C.

Jia et al. [16] pointed out that friction played a great role in affecting through-thickness texture gradients due to the variation of shear strain along the thickness direction. The polyphosphate lubricant, which had good thermal stability, reduced the shear strain and the texture gradient from the surface to the center layer at elevated temperatures. The polyphosphate lubricant can effectively prevent the formation of shear texture and therefore likely improve the deep drawability of IF steel. In summary, the polyphosphate lubricant not only improves the surface quality and antiwear properties [21], but also suppresses the shear strain between contact surfaces, shown by the enhanced γ-fibers with less shear texture. Finally, it achieves an overall improvement on the ferrite rolled IF steel with a good surface quality as well as the rolling texture with potentially improved drawability.

5. Conclusions

The polyphosphate lubricant shows significant macrotexture improvement on the surface layer with less shear texture and enhanced γ-fiber, while the texture in the center layer is insensitive to lubrication conditions. The polyphosphate lubricant presents a better lubrication performance on the texture than lubricating oil. The rolling temperature shows limited effect on the texture evolution due to the stability of polyphosphate at elevated temperature. Taking the rolling reduction into account, the orientation distribution of shear texture changed little when the reduction increased from 25% to 40%. The polyphosphate stabilizes the surface texture and reduces the gradient of shear texture through the thickness. Polyphosphate lubricant also reduces the fraction of in-grain shear bands by 48.5% compared with the unlubricated condition. This has the potential to result in enhanced drawability of IF steel after ferrite rolling. The EBSD result shows slight differences of misorientation angle distribution of rolled IF steel under various lubrication conditions. The application of polyphosphate lubricant has little effect on the small misorientation angle below 10° during ferrite rolling. The microtexture in a selected area of 1.2 × 0.9 mm^2 indicates the polyphosphate lubricant develops the favorable texture of {111}<112> along the γ-fiber and suppresses the undesired texture along the α-fiber with a {001}<110> component.

Author Contributions: All authors have read and agree to the published version of the manuscript. Conceptualization, N.K. and J.Z.; methodology, N.K. and J.Z.; formal analysis, N.K.; investigation, B.W. and

D.R.G.M.; data curation, N.K.; writing—original draft preparation, N.K.; writing—review and editing, N.K., J.Z., and H.L.; project administration, J.Z. and H.L. All authors have read and agreed to the published version of the manuscript.

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