THE EFFECTS OF LUBRICANTS AND SURFACE TREATMENT ON THE COLD ROLLING PROPERTIES OF COMMERCIALLY PURE TITANIUM STRIP

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Introduction

Under a plane-strain condition such as thin strip rolling, HCP titanium exhibits a high yield strength of approximately two times the uniaxial yield stress. In addition, the adhesion of titanium to the roll surface easily occurs during cold rolling. These deformation and frictional properties cause difficulty in the cold rolling of commercially pure titanium strip. The adhesion of titanium to the roll surface takes place when the coefficient of friction reaches more than approximately 0.05 (1). Therefore, selecting a suitable lubricant and surface treatment is very important to prevent galling and thereby to increase the efficiency of cold rolling. It has been reported that lubricant containing halogen elements is effective on the fabrication of titanium and its alloys (2,3). Halogens and their compounds, however, have disadvantages with regard to industrial applications because of their corrosive interaction with metalworking equipment and toxicity and pollution problems.

In this study, the effects of the physical and chemical properties of the lubricant and the oxidizing treatment of a titanium surface on the cold rolling properties of a commercially pure titanium strip were examined to find a practical lubricant and a method to prevent galling. In addition, the effects of a base oil and extreme pressure (EP) agent were also studied.

Experimental Procedure

Test lubricants - Series 1: Fourteen lubricants (A-M,Z) were prepared as shown in Fig. 1. Synthetic ester (hindert ester) and mineral oil were used as base stocks. The saponification value (Z, A-C) and the viscosity (A, K-M) were conditioned only by changing their mixing ratio. The acid value was conditioned by oleic acid (D-G) and polymerized fatty acid (D,H-J).

![Fig. 1: Physical and chemical properties of test lubricants - Series 1.](image-url)
Test lubricants - Series 2: Eight lubricants (No.1 - 8) were prepared to examine the effects of base oil and EP agent as shown in Table 1. One percent of the EP agent containing P and/or S was added to each of four kinds of base oils.

Surface treatment: Surface oxidizing treatment was done by heating the titanium strip for ten minutes at air temperatures of 200 - 800°C. A fine and thin oxide film with a thickness of less than 1500 Å (4) was formed by heating at air temperatures below 700°C. A thicker and more brittle oxide scale was formed by heating at an air temperature of 800°C.

The commercially pure titanium (JIS Class 2) strip 2mm thick x 30mm wide x 300mm long was cold rolled to 0.7mm thick by a reduction of 15% per pass. The rolling mill is two-high with work rolls 154mm in dia x 200mm long. The work roll surface was circumferentially ground with No.500 emery paper before rolling and the surface roughness along the barrel was approximately 0.2µm of R_max.

The rolling was carried out at a constant speed of 6.8m/min. Every lubricant was used as a neat condition and applied to both the rolls and strip for each pass.

The frictional properties and generation of galling were evaluated by the mean roll pressure and the coefficient of friction which were calculated from the measured load and forward slip. Other characteristics, including surface finish, were also examined.

Results and Discussion

Effect of lubricant

Table 1: Compositions and properties of test lubricants - Series 2.

<table>
<thead>
<tr>
<th>Lubricant No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<tr>
<td>Base</td>
<td>Palm Oil</td>
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<td></td>
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<td></td>
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<tr>
<td></td>
<td>Synthetic Ester</td>
<td>98</td>
<td>98</td>
<td>61</td>
<td>61</td>
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<td></td>
<td></td>
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<tr>
<td></td>
<td>Oleic Acid</td>
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<td>20</td>
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<td></td>
<td>Mineral Oil</td>
<td></td>
<td></td>
<td>18</td>
<td>18</td>
<td></td>
<td></td>
<td></td>
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<td>EP Additive, %</td>
<td>A: Phosphorus</td>
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<td>1</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>B: Sulfur-Phosphorus</td>
<td></td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>C: Phosphorus</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>Proper-</td>
<td>Sap. Value</td>
<td>235</td>
<td>186</td>
<td>182</td>
<td>234</td>
<td>187</td>
<td>181</td>
<td>153</td>
</tr>
<tr>
<td>ties</td>
<td>Acid Value</td>
<td>2.6</td>
<td>5.2</td>
<td>8.2</td>
<td>2.5</td>
<td>4.9</td>
<td>7.9</td>
<td>28.3</td>
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<td></td>
<td>Viscosity SUS</td>
<td>152</td>
<td>223</td>
<td>223</td>
<td>150</td>
<td>224</td>
<td>306</td>
<td>198</td>
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<td>5.0</td>
<td>5.0</td>
<td>4.5</td>
<td>&gt;15</td>
</tr>
</tbody>
</table>

Four Ball Wear Tester, 500 rpm at 50°C

The effect of saponification value on the mean roll pressure and the coefficient of friction during cold rolling is shown in Fig. 2. The strain in Fig. 2 indicates the logarithmic thickness strain for each pass during the rolling. The lubricant C with the highest saponification value provided the smallest rolling load and coefficient of friction and the slightest galling. In cases of lubricants Z, A and B, the galling was accumulated as rolling proceeded and the coefficient of friction was rapidly increased after four or five rolling passes.

The higher acid value conditioned by oleic acid provided a smaller coefficient of friction (Fig. 3 - (a)). While no significant trend was found in cases of lubricants D, H, I and J with various acid values conditioned by polymerized fatty acid (Fig. 3 - (b)).

No significant effect of viscosity on the rolling properties was found, though the mean roll pressure was markedly increased in the case of lubricant A.
with the lowest viscosity (Fig. 4). There was little difference in the rolling properties and in the degree of galling among the lubricants K, L and M.

Figure 5 shows the relationship between the lubricant properties and the coefficient of friction at the initial four passes, at which galling was scarcely caused and therefore the coefficient of friction seemed to be affected only by the lubricant property. Of all chemical and physical properties of lubricants tested here, the saponification value and the acid value conditioned by oleic acid are most effective and the acid value conditioned by polymerized fatty acid and the viscosity are less effective.

The cold rolling properties with various lubricants listed in Table 1 are shown in Fig. 6. There is no difference between the lubricants with an EP agent containing phosphorus (NO.1 - NO.3) and those with an EP agent containing both sulfur and phosphorus (NO.4 - NO.6), and little galling was caused in these cases. On the other hand, in cases of lubricant NO.7 and NO.8, the galling was noticed at the third or fourth pass, and the mean roll pressure and the coefficient of friction were higher than those for lubricants NO.1 - NO.6. The reason for poor lubricity of NO.7 and NO.8 seems not to be the EP agent,
but rather their properties caused by the base oil. Lubricants NO.7 and NO.8 have a lower saponification value and a higher acid value than those for NO.1 - NO.6 as shown in Table 1. The viscosity and the oil film strength are nearly the same among them. As is evident in Fig.5, the saponification value and the acid value improve lubricity. Therefore the lower saponification value seems to be the reason for the poor lubricity of NO. 7 and NO.8.

To clarify the effects of the saponification value and EP agent two base lubricants were prepared with the saponification values of 100 and 190 conditioned by the mixing ratio of tallow and mineral oil. One percent of the EP agent A, B or C listed in Table 1 was added to each lubricant, and eight lubricants with or without the EP agent were prepared in all. The cold rolling properties using these lubricants are shown in Fig. 7. Only those lubricants with the saponification value of 190 provided excellent lubricity, while there was no difference among those lubricants with or without the EP agent.

The multiple regression analysis was carried out to relate the lubricity with the characteristics of all lubricants described above. The coefficient of friction at the first pass was selected as the independent variable. The saponification value, acid value, viscosity and oil film strength were selected as dependent variables. The result is shown in Fig. 8. The coefficient of friction showed a strong interrelation to the saponification value and the acid value, and the relationship was expressed by the regression

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Fig. 4: Effect of viscosity on mean roll pressure and coefficient of friction.

Fig. 5: Effect of lubricant properties on coefficient of friction at initial four passes.
Effect of surface oxidizing treatment
The cold rolling properties of the surface oxidized titanium strip are shown in Fig. 9. A lubricant with relatively poor lubricity, composed of tallow (45%) and mineral oil (48%), was used to clarify the effect of the surface oxidizing treatment. The saponification and acid values were 80 and 9 respectively. A remarkable galling was caused at the third pass and the coefficient of friction reached 0.18 at the final pass for the as-received (as-annealed in vacuum) strip. For those strips surface-oxidized at air temperatures of 200°C - 600°C, no galling took place, resulting in the lowest values of the mean roll pressure and the coefficient of friction. On the other hand, the coefficient of friction markedly increased at the initial two passes for those strips oxidized at air temperatures of 700°C and 800°C, though no galling occurred.

The surface appearance of the rolled strips after the final pass are shown in Photo. 1. The surface appearance after the first pass for the strip
oxidized at an air temperature of 800°C is also shown in Photo. 1. Many scratches existed on the surface of the as-received strip. A few scratches were observed on the strip oxidized at 200°C. No scratch was observed on those strips oxidized at temperatures above 200°C and wavy surface irregularities were formed on the strip oxidized at 600°C. White areas not covered with oxide film were formed on the strip oxidized at 700°C, which were considered to be new surfaces. On the strip oxidized at 800°C, many fine surface cracks were observed after the first pass and many white areas not covered with oxide film appeared after the final pass.

As a result, the increase in the coefficient of friction in the latter half of the rolling process of the as-received strip is concluded to be caused by the galling. On the other hand, the increase in the coefficient of friction in the first half of the rolling process for those strips oxidized at 700°C and 800°C is concluded to be caused by the fracture and the exfoliation of a thicker oxide scale. Moreover the oxide film formed by heating at 200°C - 600°C is considered to be fairly ductile, because there is no appearance of fracture.

![As received](image1)
![Oxidized at 200°C](image2)
![Oxidized at 600°C](image3)

![Oxidized at 700°C](image4)
![Oxidized at 800°C (after first pass)](image5)
![Oxidized at 800°C (after final pass)](image6)

Photo. 1: Surface appearance of oxidized strip after cold rolling.
Figure 10 shows cold rolling properties of the surface oxidized strip with and without a lubricant. The surface treatment was done by heating for ten minutes at an air temperature of 500°C. For rolling without a lubricant, galling was observed at the initial third pass and the frictional condition became similar to that of the as-received strip with a lubricant. On the other hand, no galling was caused during the entire rolling with a lubricant. As a result, oxide film is effective in preventing galling, but not fully effective under the condition without a lubricant.

Conclusion

The saponification value of the lubricant and the surface oxidizing treatment for the titanium strip are effective in improving the lubricity and preventing galling. The saponification value is determined by the amount of carboxyl contained in fatty acid. Therefore, the excellent lubricity of a lubricant with a high saponification value is considered to be caused by any reaction between the carboxyl and the strip surface. The surface oxide film on the strip, even a very thin film, is fairly effective in preventing galling. Consequently, an increase in efficiency of the cold rolling commercially pure titanium strip can be achieved by the adoption of both the lubricant and surface treatment described above.

References