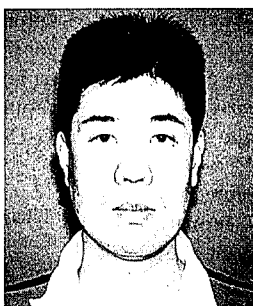


Micro Crack Prevention Technology in Cold Tandem Rolling of Pure Titanium



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Abstract

Cold-rolled Strip Coils of pure titanium are manufactured using a Sendzimir mill. Although a Sendzimir mill is suitable for manufacturing the cold-rolled strip coils with excellent surfaces, productivity is low, so, there is a desire to manufacture the cold-rolled strip coils that do not require excellent surface by cold tandem rolling mill. However, a surface defect called micro-cracks, occurs if the strip without the problem by Sendzimir mill is rolled out by cold tandem mill. In this report, the generation mechanism and the prevention method of micro-cracks in cold tandem rolling are examined to establish the higher productivity cold tandem rolling technology of pure titanium.

1. Introduction

Nippon Steel cold rolls the commercial pure titanium strip product on a Sendzimir mill equipped with small-diameter work rolls which is used for stainless steel. The Sendzimir mill is suited for producing a strip product with excellent surface luster, but is poor in productivity. There is demand for efficient manufacture of commercial pure titanium strip product without the needs of high surface luster, using a tandem cold mill equipped with large-diameter work rolls.

When commercial pure titanium is cold rolled on the Sendzimir mill, it has no surface quality problems. When it is rolled on the tandem cold mill, it develops continuous cracks (hereinafter referred to as microcracks) that are about 0.1 to 0.2 mm long and about 10 to 20 μm deep in the width direction. These microcracks randomly occur on the strip surface and induce surface defects during secondary

fabrication. There was a pressing need for development of technology for preventing the occurrence of microcracks in cold tandem rolling.

To establish the technology of tandem cold rolling commercial pure titanium with high productivity, the authors studied the occurrence mechanism and prevention method of microcracks in tandem cold rolling and developed an appropriate lubricant. The results are reported here. The lubricant was developed jointly with Kao Corporation so that it would not interfere with the tandem cold rolling of carbon steel.

2. Experimental Method and Conditions

To clarify the conditions of microcrack occurrence, cut sheets were experimentally rolled in multiple passes at a high-speed two-high laboratory mill with large-diameter work rolls by changing the friction coefficient and pass schedule. The friction coefficient was

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changed by exchanging the type of lubricant and by inducing roll coating or not.

The types and properties of lubricants used in the experiment are shown in **Table 1**. Oil A is the lubricant used at the batch-type tandem cold mill of Nippon Steel's Hirohata Works. The other lubricants were designed for use in cold rolling of both commercial pure titanium and carbon steel. They were confirmed beforehand to have no problems with cold rolling stability, detergency, rust prevention, and emulsification stability investigated for carbon steel rolling. Emulsion rolling lubrication adopted a direct application system. A tank with a capacity of 30 liters was filled with a mixture of industrial water and lubricant to a concentration of 5% at a temperature of 55°C. The lubricant was supplied through upper and lower sprays at a combined rate of about 2 l/min into the roll bite inlet at the entry side of the mill.

The cut sheet rolling conditions are listed in **Table 2**. The work rolls were polished with #600 emery paper before each rolling experiment. The roll coating was treated by repeatedly rolling the cut sheet with a reduction of 20 to 30% under water lubrication until the change in the roll surface roughness was saturated. Two types of pass schedules, or heavy reduction pattern and light reduction pattern, were adopted.

Whether or not microcracks occurred was judged by tension test and surface observation of rolled sheets. The forward slip was determined by scribing two lines at a distance (l_1) on the work roll surface, measuring the distance (l_2) between the scribed lines transferred to the rolled sheet, and calculating the ratio ($l_1/l_2 - 1$).

Table 1 Lubricants used in experiment

	Oil A	Oil B	Oil C	Oil D
Tallow/ester (%)	93/1	34/60	0/93	51/37
Oiliness (%)	4	4	2	4
Extreme-pressure additive (%)	0	0	2	6
Viscosity (cSt, 50°C)	25.2	38.2	57.0	68.6
Acid value (KOHmg·g ⁻¹)	9.0	10.0	10.5	9.2
Saponification value (KOHmg·g ⁻¹)	195.0	181.0	219.0	191.0
Average particle size (μm)	9.7	13.7	8.3	36.0

Table 2 Cut sheet rolling conditions

Item	Description	
Specimen	Commercially pure titanium: Acid pickled and shot blasted sheet 3 mm ^l × 50 mm ^w × 500 mm ^l	
Roll diameter	400mm	
Roll surface roughness	0.09-0.10 μmRa (after grinding) 0.23-0.27 μmRa (after roll coating)	
Rolling speed	250m · min ⁻¹	
Reduction schedule	Number of passes	
	Pass 1	25%(25%) 15%(15%)
	Pass 2	23%(42%) 15%(28%)
	Pass 3	24%(56%) 15%(39%)
	Pass 4	20%(65%) 15%(48%)
	Pass 5	6%(67%) 15%(56%)
	Pass 6	- 15%(62%)
	Pass 7	- 15%(68%)

(): Cold reduction ratio

3. Experimental Results and Discussion

The rolling conditions and the occurrence of microcracks are shown in **Table 3**. The occurrence of microcracks was confirmed to increase with decreasing lubricant viscosity, increasing reduction per pass, and increasing roll coating.

Representative microcrack photos are shown in **Photo 1**. With oil A, microcracks were confirmed to have occurred on the sheet surface after pass 2 and grown after pass 3. With oil D, many oil pits were confirmed on the sheet surface after rolling, but no microcracks occurred after pass 3. When the sheet without microcracks after pass 1 with oil A was tension tested, similar cracks were confirmed to occur. This suggested that factors responsible for the occurrence of microcracks formed during rolling in pass 1.

Fig. 1 shows the effects of the reduction taken in pass 1 and the friction coefficient on the occurrence of microcracks. The friction

Table 3 Rolling conditions and occurrence of microcracks

Lubricant	Reduction pattern	Roll coating	Microcracks
Oil A	Heavy reduction	No	Occurred in pass 2
Oil A	Light reduction	No	Did not occur
Oil A	Heavy reduction	Yes	Occurred in pass 3
Oil A	Light reduction	Yes	Occurred in pass 4
Oil B	Heavy reduction	No	Occurred in pass 3
Oil C	Heavy reduction	No	Occurred in pass 3
Oil C	Light reduction	No	Did not occur
Oil D	Heavy reduction	No	Did not occur
Oil D	Heavy reduction	Yes	Occurred in pass 3
Oil D	Light reduction	Yes	Did not occur

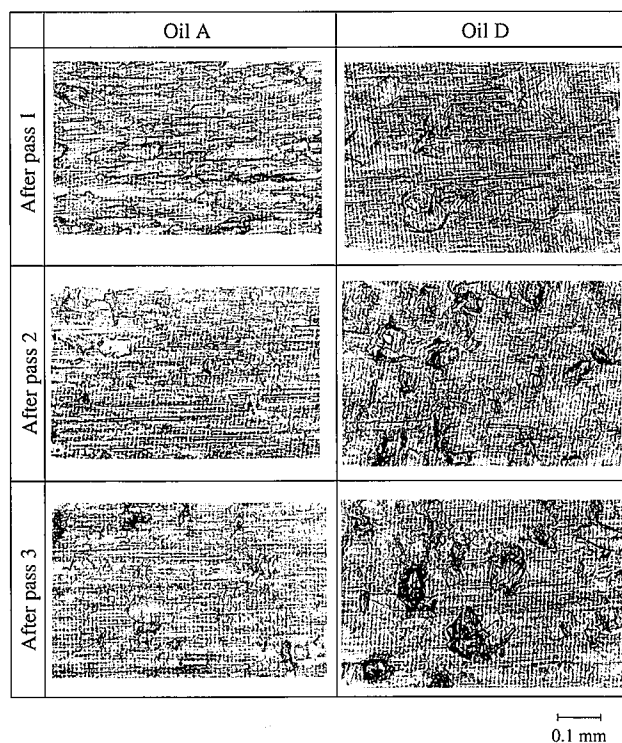


photo 1 Occurrence of microcracks (heavy reduction pattern, without roll coating)

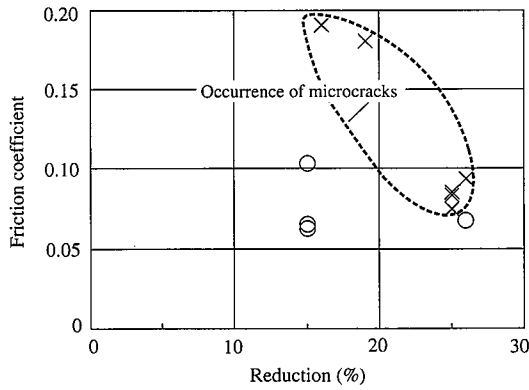


Fig. 1 Effects of reduction and friction coefficient on occurrence of microcracks

coefficient was obtained so that the experimental and calculated values¹⁾ of the rolling load and forward slip would agree with each other.

In the experiment, when the reduction taken in pass 1 was practically the same, microcracks occurred in later passes when the friction coefficient exceeded a certain level. As can be seen from Fig. 1, however, the occurrence of microcracks cannot be predicted from the absolute value of the friction coefficient alone. Assuming that the occurrence of microcracks is largely affected by surface work hardening and that the effect of surface work hardening and the effect of heat generation at the interface during rolling are largely correlated with each other, interface temperature calculation²⁾ was attempted.

The interface temperature rise with rolling is obtained as the sum of the interface temperature rise with heat generation by friction and the interface temperature rise with heat generation by plastic deformation, and is given by

$$\begin{aligned}
 T &= T_{f \max} + T_{d \max} \\
 T_{f \max} &= q_{fm} \frac{\sqrt{\alpha_p} \beta \sqrt{L} / V_r}{\lambda_p (1 + \beta \tanh(\eta))} \\
 T_{d \max} &= \frac{2 \cdot K_m}{\rho_p \cdot C_p} \ln \left(\frac{1}{1-r} \right) \frac{\beta}{\beta + \coth(\eta)} \quad \dots(1) \\
 q_{fm} &= \mu p_m \Delta V \\
 \beta &= (\lambda_p / \lambda_r) / \sqrt{\alpha_r / \alpha_p} \\
 \eta &= h_m \sqrt{V_r} / 2 \sqrt{\alpha_p L}
 \end{aligned}$$

where $T_{f \max}$ = interface temperature rise with heat generation by friction; $T_{d \max}$ = interface temperature rise with heat generation by plastic deformation; K_m = deformation resistance; ρ_p = density of sheet; C_p = specific heat of sheet; r = reduction; λ_p = thermal conductivity of sheet; λ_r = thermal conductivity of roll; V_r = rolling speed; α_p = thermal diffusivity of sheet; α_r = thermal diffusivity of roll; ΔV = average relative slip speed; L = length of contact; h_m = average sheet thickness in roll bite; q_{fm} = average heat generation by friction; μ = friction coefficient; and p_m = average rolling pressure.

Fig. 2 shows the effects of the reduction taken in pass 1 and the interface temperature rise on the occurrence of microcracks. This calculation shows that microcracks occur in later passes when the interface temperature rise in pass 1 is about 230°C or more and does not occur in later passes when the interface temperature rise in pass

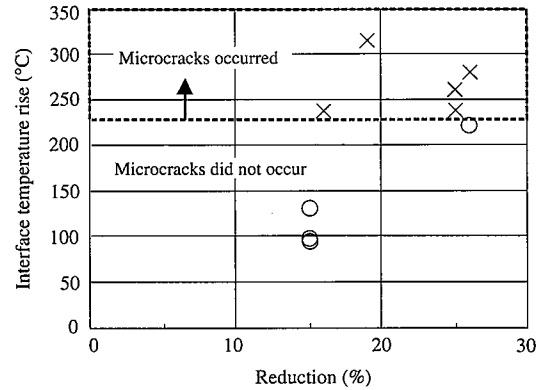


Fig. 2 Effects of reduction and interface temperature rise on occurrence of microcracks

1 is less than about 230°C. It was thus found that the critical interface temperature rise above which microcracks occur is 230°C.

To verify the versatility of the above-mentioned critical interface temperature rise, the interface temperature rise in each stand was calculated under the experimental tandem cold rolling conditions in which the commercial pure titanium sheet developed microcracks and under the experimental tandem cold rolling conditions in which the commercial pure titanium sheet developed no microcracks. The calculation results are given in Tables 4 and 5. Like the results of the laboratory experiment, the calculation results show that microcracks occur when the interface temperature rise in each stand is about 230°C or more and do not occur when the interface temperature rise in each stand is less than about 230°C. It was thus found that the critical interface temperature rise above which microcracks occur is 230°C, irrespective of the rolling conditions.

The above discussion suggests that microcracks can be prevented by setting such a pass schedule that the interface temperature rise in each pass remains below the above-mentioned critical level of 230°C.

Table 4 Experimental results without occurrence of microcracks (oil A)

Stand	Sheet thickness (mm)	Speed (m/min)	Roll (mm)	Load (tf/mm)	Friction coefficient	Interface temperature(°C)
	2.4					
#1std	2.2	69	440.2	0.96	0.052	34.6
#2std	2.0	78	508.0	1.28	0.298	86.4
#3std	1.8	88	541.3	1.29	0.208	106.6
#4std	1.6	96	578.1	0.90	0.176	68.0
#5std	1.5	103	439.4	0.767	0.182	56.2

Table 5 Experimental results with occurrence of microcracks (oil A)

Stand	Sheet thickness (mm)	Speed (m/min)	Roll (mm)	Load (tf/mm)	Friction coefficient	Interface temperature(°C)
	3.0					
#1std	2.5	47	405.7	1.70	0.134	120.1
#2std	2.0	61	495.7	1.46	0.300	239.2
#3std	1.7	76	522.7	1.46	0.164	225.7
#4std	1.5	85	579.9	1.10	0.162	111.0
#5std	1.4	96	428.4	1.53	0.179	56.6

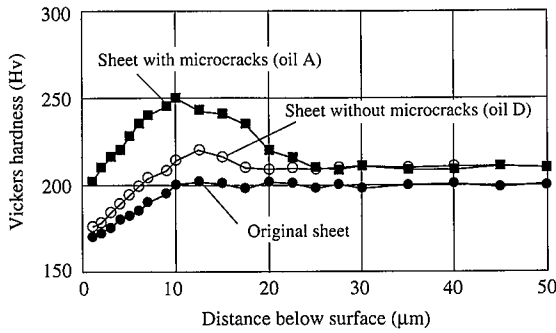


Fig. 3 Sheet hardness distribution (after pass 1, heavy reduction pattern, without roll coating)

4. Occurrence Mechanism of Microcracks

The sheets after pass 1 and shown in Photo 1 were obliquely ground, and their cross-sectional hardness distribution below the surface, was measured with a Vickers microhardness tester at a load of 10 g. The results are shown in Fig. 3. The cross-sectional hardness distribution of the original sheet is also shown. The cross-sectional hardness of the original sheet increases over about 10 μm below the surface and then remains practically constant. The cross-sectional hardness of the sheet with microcracks is clearly increased over about 25 μm below the surface, as compared with the sheet without microcracks. The cross-section hardness of these two sheets is constant and equal at 30 μm and deeper below the surface. As already discussed, when the sheet with microcracks occurring in later passes is tensioned by a few percent, it develops similar cracks. The hardened layer is thus confirmed to be embrittled.

Cross-sectional line analysis was conducted by electron probe microanalysis (EPMA) for elements with atomic weight greater than that of carbon. The EPMA failed to recognize chemically significant difference between the sheets that developed microcracks. Although the cause of this embrittlement was not identifiable, the occurrence mechanism of the microcracks is presumed as described below.

In the first stage, as the interface temperature rise in a given stand exceeds the critical level, an embrittled hardened layer is nonuniformly formed in the roll bite. In the second stage, the hardened surface layer does not elongate fast enough to follow the interstand tension or rolling operation in later stands, resulting in the occurrence of microcracks. This phenomenon is very similar to the surface embrittlement that arises from the heat generated when cold-rolled strip is ground.

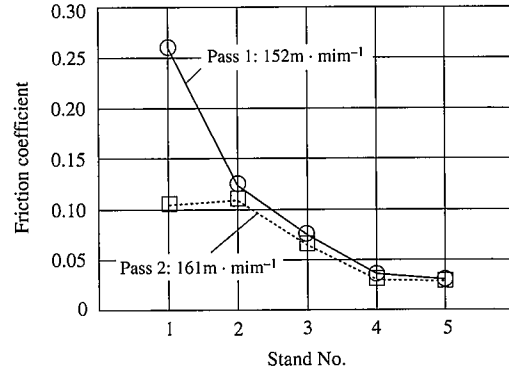
5. Experimental Verification with Large-Diameter Work Rolls

The findings obtained so far indicated that the microcracks could be prevented by setting a pass schedule that takes into account the interface temperature rise in each stand. Reducing the friction coefficient is effective in restraining the interface temperature rise. Since the conventional lubricant A is poor in rolling lubricity for commercially pure titanium, tandem cold rolling experiment verification was performed by using the lubricant D containing newly developed heat-resistant ester.

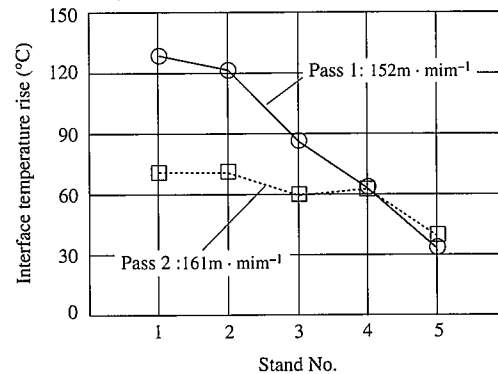
In the experiment, a 2-mm thick sheet of commercially pure titanium was cold rolled in two times to a thickness of 0.5 mm on a five-stand tandem cold mill (batch mill) at the Hirohata Works. The experimental conditions are listed in Table 6.

Fig. 4 shows the friction coefficient and interface temperature

Rolling mill	5-stand tandem cold mill (batch mill)
Cold reduction	Pass 1: 50% (2.0mm→1.0mm) Pass 2: 50% (1.0mm→0.5mm)
Speed	29-161m · min ⁻¹
Material	Commercially pure titanium (sheet width: 1,000 mm)
Lubricant	Oil D (concentration: 4.6%, temperature: 60°C)



a) Friction coefficient



b) Interface temperature rise (°C)

Fig. 4 Results of tandem cold rolling experiment

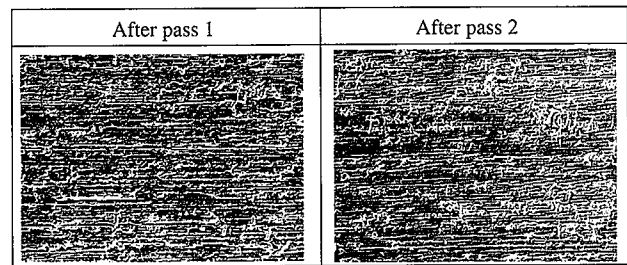


Photo 2 Sheet surface appearance after rolling

rise in each stand, and Photo 2 shows the surface of the sheet after pass 1 and pass 2. Use of the high-viscosity lubricant D succeeded in lowering the friction coefficient and preventing the occurrence of microcracks.

The plate-out amount in the experiment is about 350 mg · m⁻² and is about three times as large as that of the conventional oil A. No increase was recognized in the surface roughness of the work rolls

after rolling due to roll coating. The new lubricant D was also confirmed to be capable of preventing roll coating. It was also confirmed that the rolling power consumption with the new oil D is about 10 to 20% smaller than that with the conventional oil A.

The interface temperature rise in the experiment was a maximum of about 130°C and was lower than the critical temperature above which microcracks would occur. As evident from Photo 2, irregularities 3 to 4 μm deep were observed on the sheet surface after rolling. Sharp bend test and U-bend test were conducted to clarify the effect of the surface irregularities on secondary fabricability. No bend portion abnormality and no initiation and extension of new cracks from the surface irregularities were recognized, and satisfactory surface quality was indicated.

6. Conclusions

To establish the technology of tandem cold rolling commercially pure titanium with high productivity, the authors studied the occurrence mechanism and prevention method of microcracks during tandem cold rolling. The following findings were obtained:

1) The occurrence of microcracks is strongly correlated with the in-

terface temperature rise. When commercially pure titanium is rolled with an interface temperature rise of about 230°C or more in a given pass, it develops microcracks in later passes.

- 2) The possible occurrence mechanism of microcracks is considered as follows: In the first stage, as the interface temperature rises in a given stand exceeds a critical level, an embrittled hardened layer is nonuniformly formed in the roll bite. In the second stage, the hardened surface layer does not elongate fast enough to follow the interstand tension or rolling operation in later stands, resulting in the occurrence of microcracks.
- 3) Lowering the friction coefficient is effective in preventing the microcracks. When tandem cold rolling experiment verification was performed using a lubricant containing newly developed heat-resistant ester, it was found that a 2-mm thick sheet of commercially pure titanium could be cold rolled down to 0.5 mm in two passes without microcracks.

References

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