Influence of Heating Temperature on Edge Crack in Hot Rolling of 36%Ni-Fe Alloy

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For the purpose of edge crack control in hot rolling of 36%Ni-Fe alloys, a high temperature tensile test and laboratory-scale hot rolling experiment were carried out. Intergranular oxidation has been considered to be one of the major factors in edge cracking. Edge cracks which initiate from intergranular oxidation grow to the inner side along coarse grain boundaries as the thickness reduction ratio increases. The depth of intergranular oxidation increases at higher reheating temperatures. However, recrystallization occurs at the crack tip, and this has the effect of suppressing crack growth. This suggests that promoting recrystallization during hot rolling by increasing the reheating temperature, rather than inhibition of intergranular oxidation as such, is effective for suppressing edge cracking.

KEY WORDS: rolling; oxidation; edge crack; recrystallization; heating temperature; 36%Ni-Fe alloy.

1. Introduction

36%Ni-Fe alloy steel shows the feature of a low thermal expansion coefficient at room temperature due to the spontaneous volume magnetostriction effect. This steel has been widely used in electronic components, taking advantage of its small change in dimensions and shape.1–3) Because the high Ni–Fe alloy is solidified as single-phase austenite, formation of coarse columnar crystals and segregation of impurities occur easily.4,5) In addition, the grain boundaries oxidize preferentially when the material is heated to high temperature.6) As a result, 36%Ni-Fe alloy has been regarded as a hard material which is prone to surface or edge cracks due to vulnerability at the grain boundaries during hot working.7) In industrial manufacturing of 36%Ni-Fe alloy steel sheets, two or more processes were required as slab reheating preceding hot rolling after the metallographic structure was subtilized by forging or slabbing mill rolling, so a more efficient manufacturing process is needed.

Several studies from various viewpoints have been reported in connection with improvement of the workability of high Ni–Fe alloy steel. Research focusing on chemical composition has a long history, and research on the influence on hot ductility of additive elements such as Mn, S, Al and B has been reported.5,6,9) A heating method using a non-oxidizing atmosphere furnace7) and the influence of additive elements on high temperature oxidation10) were also studied as techniques for suppressing grain boundary oxidation.

The authors carried out research that considered the thermal stress transforming behavior of the slab in the heating furnace as part of an examination of the hot workability of 36%Ni-Fe alloy steel.11) It was shown that tensile stress can act on the slab surface as a result of the cross-sectional temperature difference during slab heating. In addition, hot ductility was remarkably decreased by intergranular fracture under a low strain rate. The possibility of an influence on surface and edge cracks was clarified, and an appropriate temperature pattern to prevent surface and edge cracks in the reheating furnace was presented.

In this report, hot workability is examined focusing on the reheating temperature with the aim of preventing edge crack in the hot rolling of 36%Ni-Fe alloy steel. In hot rolling of steel sheets, tensile stress acts in the rolling direction at the edge portion due to material expansion in the width direction, and as a result, edge crack occurs in hot rough rolling, in which rolling tension is not applied. Edge crack not only decreases product yield but can also cause strip breakage in the following thinner strip rolling process. The reheating temperature of the material is a factor that influences both internal oxidation and hot ductility, which are considered to be factors in edge crack. Therefore, the influence of the reheating temperature on the hot workability of 36%Ni-Fe alloy steel, especially its influence on edge crack, was investigated by a high temperature tensile test and a hot rolling experiment.

2. Experimental Conditions

2.1. Metallographic Structure and High Temperature Oxidation Behavior of 36%Ni-Fe Alloy Steel

Table 1 shows the chemical composition of 36%Ni-Fe alloy. Figure 1 shows that the microstructure of the as-cast 36%Ni-Fe alloy steel consists of coarse columnar crystals. Figure 2 shows an example of a cross-sectional photograph of the oxidized test piece after a 36%Ni-Fe alloy steel...
test piece was reheated in the air at 1 423 K for 7 200 s.

36%Ni-Fe alloy is a typical alloy that forms subscale with grain boundary oxidation and an intragranular oxidation layer (metal/oxide mixture) under the surface scale by high temperature oxidation. In Fig. 2, it can be understood that straight grain boundary oxidation, in which the grain boundary was oxidized with priority toward the ground ferrite side, has expanded from the grain boundary oxidation layer.

The authors considered the growth behavior of the grain boundary oxidation of high-Ni-Fe alloy steel, and showed that the grain boundary oxidation depth can be expressed by applying a parabolic law to the reheating temperature and time. A formula for scale generation was also presented.12)

In the hot rolling of steel sheets involving such grain boundary oxidation, it has been considered that opening of the grain boundary oxidation part like a wedge is a factor in expanding edge cracks and surface defects.6–8)

2.2. High Temperature Tensile Test Conditions

To examine the influence of internal oxidation of 36%Ni-Fe alloy steel on hot ductility, a high temperature tensile test in the air atmosphere was performed using round bar specimens, as shown in Fig. 3. Here, the gage zone size of the specimens was φ6 mm×16 mm, and the specimens were extracted from portions of the slab across the above-mentioned columnar crystals.

In the high temperature tensile test, the prescribed heating was given to the test piece by induction heating, and the cooling rate was controlled by Ar gas spraying. The basic heat cycle is shown in Fig. 4. After soaking at 1 423 K for 300 s, the test piece was cooled to the test temperature of 1 073–1 373 K with the holding time of 30 s, after which the tensile test was performed. The strain rate in the tensile test was set at 10 s⁻¹ assuming hot rough rolling. To compare the influence of grain boundary oxidation, a tensile test at the test temperature of 1 273 K was also carried out after soaking for 3 600 s at 1 423 K. After fracturing, the test piece was cooled rapidly by Ar gas spraying to prevent scale formation. The area of the fracture cross section after the tensile test was measured, and hot ductility was evaluated by the reduction of area (aperture value of the fracture cross section).

2.3. Experimental Conditions of Hot Rolling

To examine the influence of the reheating temperature on workability, and in particular, the influence on edge crack in hot rolling, a hot rolling experiment was performed with a laboratory-scale mill. The experimental conditions are shown in Table 2. A two-high rolling mill with 200 mm diameter work rolls was used for the rolling experiment. The test pieces were cut out from portions of the 36%Ni-Fe alloy steel slab so that the rolling direction crossed the columnar crystals shown in Fig. 2. The test pieces were prepared with a shape in which the test piece thickness changed continuously from 14 mm to 30 mm in the rolling direction, as shown in Fig. 5. By using this wedge shape, it is possible to obtain the conditions of various thickness reduction rates in the same test piece by one passing rolling. The roll gap was set as 15 mm in this experiment, so the maximum reduction rate was 50%. An electrically heated furnace was used to heat the test pieces in the air atmosphere, and their temperatures were measured by embedded thermocouples. The heat condition was set to two conditions of 1 323 K and 1 493 K with soaking time of 7 200 s assuming actual hot rolling. The transportation time from extraction of the
3. Results of Experiments

3.1. Results of High Temperature Tensile Test

Figure 6 shows the relationship between the temperature and reduction in area in the high temperature tensile test. When the tensile test temperature was more than 1 300 K, reduction in area displayed high ductility of near 100%, and reduction in area decreases gradually at lower temperatures. The reduction in area with the holding time of 3 600 s at 1 423 K is also plotted in Fig. 6, and is almost equal to the case of 300 s. Cross-sectional photographs of the fractured part of the specimens after the tensile test under the conditions of holding times of 300 s and 3 600 s at 1 423 K are shown in Fig. 7. The test temperature was set to 1 273 K. Scale was formed and squeezed on the test specimen in both cases, but many cracks were observed at parts other than a fracture point. In the case of 300 s holding in Fig. 7(a), thin external scale and subscale with a thickness of the same degree were observed. There is a part where the scale has flaked off locally. It is comparatively slight, although the crack partially reached the ground ferrite side. On the other hand, formation of scale was remarkable at the holding time of 3 600 s in Fig. 7(b). The external scale was 300 μm or more in thickness and mostly flaked off, and only subscale with a thickness of 200–300 μm was observed in the majority area. Moreover, the depth of the crack in the ground ferrite side was also more than 1 mm. Figure 8 shows the metallographic structure etched by nitric hydrofluoric acid in the cross section of the 3 600 s holding specimen. It can be understood that these cracks occurred along the grain boundary. Moreover, fine recrystallized grains can be observed at the tip with the excellent squeezing ability. Although it has been thought that the internal oxidation caused edge crack, recrystallized grains were confirmed in the squeezed part. Thus, the results of the high temperature tensile test suggested that it is necessary to consider the

<table>
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<th>Table 2: Experimental conditions of a laboratory mill.</th>
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Fig. 5. Schematic view of laboratory rolling using wedge-shaped test piece.

Fig. 6. Effect of deformation temperature on hot ductility of 36%Ni-Fe alloy.

Fig. 7. Cross section of 36%Ni-Fe alloys after tensile test (Held for 300/3 600 s at 1 423 K in air, tensile test at 1 273 K).

Fig. 8. Metallographic structure of 36%Ni-Fe alloys after tensile test (Held for 3 600 s at 1 423 K in air, tensile test at 1 273 K).
influence of recrystallization as a factor which reduces the hot workability of 36% Ni-Fe alloy steel.

3.2. Hot Rolling Experiment Result

Next, the results of the laboratory rolling experiment are described as an evaluation of workability in hot rolling. In this experiment, a test piece of the wedge shape with thicknesses in the range of 14–30 mm shown in Fig. 5 was rolled in the roll gap set to 15 mm. After rolling, width expansion averaging approximately 10% was observed in the test piece. The distribution of the thickness reduction rate in the rolling direction was calculated from the relationship between thickness change and longitudinal direction growth. **Figure 9** shows the longitudinal direction thickness of the test piece and the distribution of the thickness reduction rate after rolling. In this experiment, it can be considered that the influence of abnormal rolling due to the wedge shape of the test piece was small, as no difference was observed in the rolling behavior in the parallel portion and the wedge-shaped portion.

The transportation time from extraction of the test piece from the heating furnace until charging to the rolling mill was about 10 s. The temperature decrease of the test piece was calculated by a heat transfer calculation using the finite-difference method. The temperature decrease in the test piece surface part is larger than that in the central portion, as shown in **Fig. 10**, and was about 40 K in 1,493 K reheating and about 60 K in 1,323 K reheating. Here, the coefficient of heat transfer by air cooling was set to 11.6 W/m² K, and the radiation rate was set to 0.8.

The side surfaces of rolled test pieces with thickness reduction rates of 0%, 20%, and 40% are shown in **Fig. 11**.

A slight vertical stripe has occurred on the side surface at the reduction rate of 0%, which means only heating without reduction. However, at the reduction rates of 20% and 40%, the vertical stripe becomes prominent and can be recognized as a crack on the side surface. At the reheating temperatures of 1,493 K and 1,323 K, it can be understood that many vertical stripe patterns, which can be recognized as cracks, exist in 1,493 K reheating. A large crack was observed at the upper center on the side surface at the reduction rate of 40% with 1,323 K reheating.

Next, a test piece was cut out horizontally from the center of thickness of the specimen after rolling, as shown in **Fig. 12**, and the crack which formed in the vicinity of the side was observed in detail. **Figure 13** shows a photograph of the horizontal cross section of the side area at reduction rates of 0%, 20%, and 40%. At the thickness reduction rate

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**Fig. 9.** Thickness reduction rate after rolling.

**Fig. 10.** Temperature of test piece.

**Fig. 11.** Side surface of rolled test piece.

**Fig. 12.** Observed area of edge crack.
of 0%, external oxidation, intragranular oxidation and intergranular oxidation occurred from the side surface where it was in contact with the air. The external scale has flaked off almost completely in 1493 K reheating. The depth of the intergranular scale in 1323 K reheating was shallow, being about 200–300 µm, whereas the depth in 1493 K reheating was about 400–500 µm. Both forms of the intergranular scale were straight. Here, the depth of the intergranular scale was defined as the depth from the boundary between the external scale and intragranular scale. At the reduction rate of 20%, the intergranular scale in 1493 K reheating grew to a wedge-shaped crack with a depth of more than 500 µm and a caudal crack extending to 200–300 µm. At the reduction rate of 20% with 1323 K reheating, the depth of the intergranular scale was 500 µm, and the crack form was also the same as in 1493 K reheating. At the reduction rate of 40% with 1423 K reheating, the depth of the crack was about 500 µm, and the crack did not propagate, although the caudal crack extended to more than 1 mm. Here, the thickness of the intragranular oxidation layer decreases as the thickness reduction rate increases because the rolled test piece expands in the longitudinal direction. On the other hand, the depth of the crack expanded up to 2 mm or more in the case of the reduction rate of 40% with 1323 K reheating. It is presumed that this crack is pertinent to the large edge crack observed on the side surface in Fig. 11. Based on the hot rolling experiment, when the reheating temperature is low, a large edge crack is generated with the progress of rolling, even though the intergranular scale at the beginning is small. On the other hand, when the heating temperature is high, the crack does not progress beyond the initial depth of the intergranular scale.

4. Discussion

4.1. Depth of Intergranular Scale and Strain Behavior at the Tip

First, the validity of the depth of the intergranular scale in this experiment is considered. The intergranular scale depth \( L_1 \) (µm) and the intragranular scale depth \( L_2 \) (µm) are calculated by the growth speed formula \(^{(12)}\) of the oxidation depth of the 36%Ni-Fe alloy steel for the experimental conditions shown in Table 2.

\[
L_1 = \sqrt{2.45 \times 10^9 \cdot \exp \frac{-14134}{T \cdot t}} \\
L_2 = \sqrt{6.33 \times 10^{12} \cdot \exp \frac{-26482}{T \cdot t}}
\]

Here, \( T \) is temperature (K), and \( t \) is scale generation time (h).

Figure 14 shows the relationship between the reheating temperature and the scale depth at the heat holding time of 7200 s (2 h). According to Fig. 14, the intergranular scale depth is about 430 µm in case of 1493 K reheating and about 240 µm in case of 1323 K reheating. This is generally consistent with the experimental results shown in Fig. 13 for both conditions. The results of calculations by the above formula also agree with the experimental results for the intragranular scale depth.

Therefore, under the 1493 K reheating condition in this experiment, it is reasonable that the intergranular scale depth before rolling was slightly less than 2 times as large as that in 1323 K reheating. From the viewpoint of crack extension due to stress concentration, it considered that the...
1493 K heating conditions are unfavorable in comparison with 1323 K heating. However, since the crack extension under the two thermal conditions changes in the subsequent rolling process, consideration from a different viewpoint is needed. Therefore, a numerical analysis of stress and strain in the vicinity of the intergranular scale was carried out.

For the horizontally cut surfaces at the center of thickness including the side surface of the rolled material, such as that in Fig. 12, a crack due to intergranular scale was given as a linear slit having a length of 0.5 mm. A two-dimensional FEM analysis was performed for the stress and strain behavior under tensile deformation in the vertical direction with respect to the slit. The commercial FEM code Abaqus was used for the numerical analysis. Stretch transformation was given from both end parts to a plane which was divided into a $0.05 \times 0.05$ mm mesh.

**Figure 15** shows the FEM analysis results for the reduction rate of 0.5%, which corresponds to a very early stage of rolling. Figure 15(a) shows the equivalent stress, and (b) shows the distribution of the equivalent plastic strain. The stress is concentrated at the vicinity of the crack tip, which causes the strain. While the average equivalent plastic strain in the material is about 0.006, the equivalent plastic strain at the crack tip is about 0.1. The stress and strain have occurred in a slanted direction from the tip of the notch toward the front/rear and the inner side.

Larger crack propagation occurred during rolling in 1329 K reheating condition where depth of grain boundary oxidization is smaller in comparison with 1493 K reheating condition. To explain this phenomenon, change of ductility property caused by recrystallization, considering stress and strain behavior near crack should be taken into account.

### 4.2. Effect of Recrystallization in Suppressing Crack Extension

Next, the edge cracking behavior in the hot rolling of 36% Ni-Fe alloy steels is discussed from the viewpoint of recrystallization. **Figure 16** shows a cross-sectional photograph of a metallographic structure etched by nitric hydrofluoric acid at the vicinity of the edge in the observation area of a rolled test piece. At 0% reduction, corresponding to the condition immediately after reheating, the coarse crystal grains of the metal structure were observed at both reheating temperatures, and intergranular oxidation had occurred along the grain boundaries at the surface side.

In the case of the reduction rate of 20% with the reheating temperature 1323 K, the edge crack expands in such a way as to push the grain boundary. When the reduction rate is 40%, the crack seems to be bent in the middle. This occurred as a result of bending it at the intersection of the grain boundary from the left side. It is understood that the crack is extended to the inside along the grain boundaries. There is no change in the size and shape of the crystal grains, including the vicinity of the grain boundary.
On the other hand, at the high reheating temperature of 1,493 K, it can be seen that small crystal grains have occurred in the crack tip when the rolling reduction rate is 20%. As shown in Fig. 15, strain has concentrated in the vicinity of the crack tip, which is reasonable to think that this is due to dynamic recrystallization. When the reduction rate is 40%, the region of small grains formed by recrystallization spreads to a larger area, whereby it is inferred that extension of the crack is suppressed.

The conditions for the occurrence of dynamic recrystallization are determined by the strain rate and deformation temperature. In addition, it is generally thought that there is temperature range where dynamic recrystallization occurs in a very high temperature region in comparison with static recrystallization. For example, the temperature of the static recrystallization of 18Cr-8Ni stainless steel is 973 K, but the dynamic recrystallization temperature exceeds 1,373 K. Thus, under the strain rate and chemical composition conditions of the present experiment, the temperature is in the range for dynamic recrystallization between 1,323 K and 1,493 K. From the above discussion, it is suggested that setting a high reheating temperature to promote dynamic recrystallization is effective for suppressing edge cracking in hot rolling of 36%Ni-Fe alloy steel. This is the opposite of the conventional concept of suppression of grain boundary oxidation.

Because the conditions for dynamic recrystallization depend on material conditions such as chemical composition and the initial grain and hot rolling conditions such as strain rate, those conditions should be considered when determining the optimal reheating temperature.

5. Conclusions

The influence of the reheating temperature on the hot workability of 36%Ni-Fe alloy steel, especially the influence on edge cracking, was investigated by a high temperature tensile test and a hot rolling experiment, and the following conclusions were obtained.

(1) In 36%Ni-Fe alloy steel with coarse columnar crystals, intergranular scale occurs along the grain boundaries. The depth of the intergranular scale increases at higher reheating temperatures or longer holding times. This intergranular scale is a major cause of edge cracking in hot rolling. The growth of an edge crack initiating from intergranular scale increases along the coarse grain boundary with the thickness reduction rate.

(2) When the reheating temperature is high, small crystal grains occur in the crack tip vicinity with the progress of rolling. These small grains have the effect of suppressing the extension of the crack due to the large strain in the vicinity of the crack tip and the occurrence of dynamic recrystallization at high temperature.

(3) In order to suppress edge cracking in hot rolling of 36%Ni-Fe alloy steel, the effectiveness of setting a high reheating temperature to promote dynamic recrystallization is suggested, which is the opposite of the conventional concept of suppression of grain boundary oxidation.

REFERENCES