Recrystallization Behavior and Texture Formation of Rapidly Annealed Cold-Rolled Extralow Carbon Steel Sheets

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The influence of the heating rate up to 1000°C/s on recrystallization behavior and texture formation of an extralow carbon steel sheet was investigated. To discuss the experimental results of this investigation, a mathematical model for predicting recrystallization behavior was developed. An analysis using the model shows that the heating rate influences the apparent thermal activation energy of recrystallization. At a higher heating rate, C dissolved from cementite and AlN precipitated during heating decreases and as a consequence the apparent thermal activation energy is lower. This can also be a valid explanation for the fact that recrystallization of a steel sheet cooled at low temperature is retarded.

The main orientations of the recrystallization texture are weakened if the heating rate is increased. This tendency has already been observed at the initial stage of recrystallization. If the specimen is heated slowly up to the initial stage of recrystallization, the decrease in the intensity of the main orientations is reduced even though the heating rate subsequently increases. This result indicates that, concerning formation of the recrystallization texture, the heating rate mainly influences the nucleation process. [doi:10.2320/matertrans.47.1769]

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1. Introduction

The influence of the heating rate on recrystallization behavior and texture formation was once intensively studied for the purpose of optimizing batch annealing processes. The study determined an optimum heating rate for deep drawing sheets at which the precipitation of AlN and recrystallization occurs simultaneously and the development of {100} is remarkably hindered.¹)

To improve productivity, the first continuous annealing and processing line (CAPL) was developed around 40 years ago. At that time, the effect of the heating rate on mechanical properties was also investigated in detail.²) Since a heating facility being considered for installation at that time consisted of radiating tubes, a heating rate of less than 50°C/s, was expected. Therefore, the influence of a heating rate higher than 100°C/s on recrystallization behavior and texture formation was hardly studied at the time.

In recent years, a limited number of investigation have been conducted on the influence of a heating rate higher than 100°C/s on recrystallization behavior and the resultant texture formation of steel sheets.³⁻⁵) General findings were that the recrystallization temperature increased and ND // (111), the main orientation of the recrystallization texture, became weaker upon the heating rate being increased. Concerning recrystallization behavior, Ushioda et al.⁵) proposed a model describing recrystallization behavior considering the influence of the heating rate. Their model was adjusted for only two experimental data at the heating rates of 0.04 and 8°C/s and they did not discuss the validity of the model for a heating rate higher than 10°C/s in detail. They also discussed the decrease in the intensity of ND // (111) of low carbon steels due to an increasing heating rate in relation to the dissolution of massive cementite.

Matsuo et al.⁵) concluded that the decrease of ND // (111) was caused by the change in the nucleation sites of recrystallization. If the heating rate was high, the nucleation sites were not only grain boundaries that were the main nucleation site in the case of a low heating rate but also the vicinity of massive cementite. The orientations of grains recrystallized at grain boundaries were mainly ND // (111)⁵) and various orientations were observed in grains recrystallized in the vicinity of massive cementite,²⁹) and as a consequence the intensity of ND // (111) was reduced.

In this paper, the influence of a heating rate up to 1000°C/s on recrystallization behavior and texture formation of an extralow carbon steel sheet in which massive cementite does not exist has been investigated. A model has been developed to discuss the influence of the heating rate on the driving force for recrystallization and the mobility of grain boundaries between a deformed and a recrystallized grain which control recrystallization behavior.

2. Experimental Procedures

2.1 Material

The chemical composition of the steel used in the experiment is presented in Table 1. This steel was produced in an industrial LD converter and continuously cast into 250mm-thick slabs. An extra low carbon steel was selected for this study because it is expected to be widely used as deep drawing steel sheets or mild tin plate sheet in the future and the phenomena to be investigated can be extracted in relatively simple form because of the absence of massive cementite.

Table 1 Chemical composition of the steel used in the experiment.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0029</td>
<td>0.017</td>
<td>0.10</td>
<td>0.005</td>
<td>0.003</td>
<td>0.066</td>
<td>0.0022</td>
</tr>
</tbody>
</table>

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2.2 Hot rolling and cold rolling conditions
The slab was reheated at 1250°C for 1 h and hot rolled into 30 mm-thick plate at a finishing temperature of around 1000°C, and smaller plates 30 mm thick, 150 mm wide and 200 mm long were machined from this plate. These plates were reheated and kept at 1000°C for 60 mins. and then hot rolled at a finishing temperature of around 930°C according to a pass schedule of 30 → 20 → 12 → 10 → 7 → 4 → 3 mm. The hot rolled specimens were subsequently cooled at an average rate of 50°C/s to about 50°C above the furnace temperatures of 650 and 750°C, respectively, and kept at these furnace temperatures for 1 hour and then cooled down to room temperature in the cutout furnace. Different coiling temperatures were chosen to investigate the influence of AlN precipitates on recrystallization behavior during a rapid heating.

After pickling, the hot rolled specimens were cold rolled 80% into 0.6 mm thick sheets. For the annealing tests, the sheets were machined to specimens 0.6 mm thick, 40 mm wide and 140 mm long.

2.3 Annealing condition
The annealing tests were carried out on a Gleebe current resistance heating machine. A typical heating pattern consists of a heating at a constant heating rate and subsequent water quenching. The heating rates mainly applied were 1, 10, 300 and 1000°C/s. A heating pattern consisting of a slow heating of 10°C/s up to 600°C and a rapid heating of 300°C/s from 600°C up to higher temperatures was also employed.

The temperature was measured by means of a thermocouple welded at the center of the specimen and the obtained electrical current change was stored in a computer and then the temperature history was printed out after the experiment. Errors in the temperature measurement caused by thermal inertia were reduced by using a thin thermocouple of 0.1 mm in diameter. It was confirmed that the accuracy of the temperature measurement was about 5°C even at a high heating rate of 1000°C/s.

2.4 Microstructure and texture analysis
Observations of the microstructure of the specimen were carried out in the midplane thereof. The fraction recrystallized was determined by making a mesh of 20 × 20 and counting the number of cross points where the microstructure was recrystallized. The grain size of the specimen fully recrystallized was determined by counting the number of grains in 5 different areas of 0.08 mm² in the specimen.

The texture of the specimen was measured using an automatic goniometer with an Mo tube. Detailed studies of the texture have been carried out by means of a three-dimensional analysis based on the vector method. The results of this texture analysis are described schematically in the form shown in Fig. 1, in which the main crystal orientations appearing in rolling and recrystallization textures are presented. Furthermore, the EBSP technique was used to determine the orientation of grains recrystallized at the initial stages of recrystallization.

3. Experimental Result
Figure 2 shows an example of a thermal history of rapid heating and quenching. A setting program of heating was modified to obtain a constant heating rate even in a higher temperature region as shown in Fig. 2. To realize a sharp bend at the maximum temperature, quenching was started before reaching the end of heating in the program in which the maximum temperature was set a little higher than required. The cooling rate in this example of a 0.6 mm thick specimen was about 4500°C/s.

Figures 3 and 4 show the influence of the heating rate on the recrystallization behavior of specimens cooled at 650°C.
and 750°C, respectively. Here, the ferrite grain size of the hot bands were 21.8 and 25.2 μm, respectively. C and N in solution were determined by an internal friction measurement and were C: 3.47 ppm and N: 2.34 ppm in the specimen coiled at 650°C and C: 3.22 ppm and N: 0.25 ppm in that coiled at 750°C. The Figures show that an increase in heating rate increases the recrystallization temperature. For example, a 50% recrystallization temperature is about 70°C higher at a heating rate of 1000°C/s than at 10°C/s.

To show the influence of the coiling temperatures on the recrystallization behavior more clearly, cases in which the heating rate was 10 and 1000°C/s respectively are shown in Fig. 5 and the cases in which the heating rate was 300°C/s from the beginning and after a slow heating of 10°C/s up to 600°C are presented in Fig. 6. As shown in these figures, the recrystallization temperature is higher at a lower coiling temperature in the case of a heating rate of 10°C/s. If the heating rate increases, the influence of the coiling temperature on recrystallization behavior is weakened.

Figure 6 shows that slow heating up to 600°C retards the recrystallization and retardation of recrystallization is more remarkable in the specimens coiled at 650°C than that in the specimen coiled at 750°C.

The grain size of the specimen just after full recrystallization is around 23 μm regardless of the heating rate, heating pattern and coiling temperature in the present experimental conditions. Comparing the grain sizes of the specimen at the same temperature, the grain size of the specimen heated at a higher heating rate is smaller than that heated at a lower heating rate. For example, the grain sizes of the specimen coiled at 750°C and heated at heating rates of 10 and 1000°C/s up to 775°C are 31 and 23 μm, respectively. In the former case, the specimen underwent grain growth after recrystallization.

Figure 7 shows the change in the intensities of (111) // ND, (100) // ND and r-values calculated by Nagashima’s method in the specimens coiled at 650°C during the progress of recrystallization. The increasing heating rate causes an increase in (100) // ND and a decrease in (111) // ND intensities. The r-values decrease if the heating rate increases. Specimens heated at a constant heating rate of 10°C/s and heated at 10 to 600°C and at 300°C/s afterwards
show a similar change in r-values during the progress of recrystallization, while specimens heated at a constant heating rate of 300°C/s show r-value behavior similar to that of specimens heated at 1000°C/s. These results indicate that the decrease of r-values caused by rapid heating may be reduced by introducing slow heating up to a certain temperature.

To study the difference between the recrystallization textures of specimens heated at various heating rates, a three-dimensional texture analysis was carried out. Figure 8 shows the recrystallization texture of a specimen heated at 10°C/s and the difference between the intensity of this specimen as compared with that of a specimen heated at 1000°C/s. The recrystallization texture is characterized by strong (111) // ND with a main orientation of [111](011) and (110) // RD with a sub-main orientation of [100](011). The increase in the heating rate decreases the intensities of these orientations and tends to make the texture uniform.

4. Discussion

4.1 Recrystallization behavior

To study the different recrystallization behaviors due to different coiling temperatures and heating patterns in detail, a mathematical model has been developed based on the following assumptions:

The recrystallization occurs based on the nucleation and growth mechanism described by the Johnson-Mehl-Avrami-Kolmogorov equation at the initial stage up to about 10% recrystallization and afterwards recrystallization occurs only on the growth mechanism (site saturation). Figure 9 shows the data which supports this assumption. The number of recrystallized grains increases until about 10% recrystallization and then tends to be saturated. Here, both the nucleation rate and the growth rate are assumed to be a function of
temperature and the J-M-A-K equation is described as a function of \( \exp(Q/RT) \), where \( Q \) is the apparent thermal activation energy and \( R \) the gas constant.

Since not only nucleation but also annihilation of grains simultaneously occur in real recrystallization and recrystallization behavior strongly depends on the cold rolling texture, the model developed here is simplified. The model, however, satisfies the purpose of this analysis on the influence of the heating rate on recrystallization behavior, as will be described later.

Another assumption is that a recrystallized grain is spherical in form.

The model is described by the following equations after differentiation of the J-M-A-K type equation with time which expresses the progress of recrystallization \( dx \) in the time \( dt \).

\[
\frac{dx}{dt} = K_A \exp(-Q_A/RT)\ln[1/(1-x)]^{1/2}(1-x)
\]

for N&G

\[
\frac{dx}{dt} = K_B \exp(-Q_B/RT)(1-x)
\]

for site saturation

The constants \( K_A, K_B, Q_A \) and \( Q_B \) are determined as \( K_A = 8 \times 10^{16} \), \( Q_A = 292 \text{ KJ/mole} \) and \( Q_B = 84 \text{ KJ/mole} \) using the data from specimens coiled at 750°C and heated at 1 and 10°C/s. \( K_B \) is determined under the assumption that \( dx/dt \) in eqs. (1) and (2) are equal when a fraction recrystallized is 8%. The apparent thermal activation energy \( Q_A \) is approximately equal to that of the self-diffusion in \( \alpha-\text{Fe} \). This indicates that the fundamental process at the nucleation stage of recrystallization is controlled by the bulk self-diffusion. In the site saturation stage, the apparent thermal activation energy \( Q_B \) becomes smaller. This might be explained by the fact that the mobility of a grain boundary is controlled by grain boundary diffusion whose thermal activation energy is about half that of bulk self-diffusion.

Figure 10 shows recrystallization curves calculated by the model in comparison with the experimental results for the heating rates of 10, 300 and 1000°C/s. This result shows that the calculated fraction recrystallized is less than the experimental results at higher heating rates. This means that if the model is fitted based on the experimental data at low heating rates, the recrystallization progress calculated at higher heating rates is slower than experimental findings show.

A possible explanation for this deviation might be expressed by the difference in the driving force of recrystallization, \( E \), which is described as \( E = Gp b^2 \). Here, \( G \) is the stiffness constant, \( p \) is the dislocation density and \( b \) is Burger’s vector. At a higher heating rate, there is little time for recovery and consequently a high density of dislocation exists at the stage of recrystallization. From the qualitative aspect, this explanation seems reasonable. However, a quantitative evaluation leads to a conclusion that the difference in the driving force does not affect recrystallization significantly. There is a relationship between hardness and dislocation density, namely, \( H_v \sim \rho^{1/2} \). The hardnesses of the specimens just before the onset of recrystallization were \( H_v = 170 \) and 175 in cases where the heating rates were 10 and 1000°C/s respectively. The result calculated by the model reveals that recrystallization is hardly influenced by the difference in driving force derived from hardness.

A change in the activation energy at higher heating rates may be another reason for the difference in recrystallization behavior. Fitting the calculated results to the experimental ones with various activation energies, it was found that good agreement between calculation and experimental results is obtained if the activation energy of recrystallization at a heating rate of 1000°C/s is assumed to be smaller by 4% than that at a heating rate of 10°C/s, as shown in Fig. 11. There are some possible explanations for the change in thermal activation energy. First of all, it is conceivable that the interaction between dislocation and impurity atoms during heating has an effect. If less time for the interaction is available due to a higher heating rate, dislocations move more easily. After forming the subgrain structure, the progress of recrystallization is retarded by an interaction between dislocations forming sub-boundaries and impurity atoms. During the progress of recrystallization, sub-boundaries are drawn to grain boundaries and disappear there through an annihilation process of dislocations. The last two phenomena are also retarded if the dislocations interact with impurity atoms. At higher heating rates, these inter-
actions are only partially intact and the apparent activation energy decreases.

The number of impurity atoms interacting with dislocations depends not only on the available time during heating but also on the number of existing impurity atoms. The impurity atoms mobile enough to interact with dislocations are C and N. C exists as carbon in solution and as cementites before annealing, while N exists in the form of precipitates of AlN and as N in solution. During heating, C dissolves from the cementite and the amount of C in solution increases. Matsuo et al. and Ushioda et al. reported that the dissolution of cementites during annealing was accelerated if they were refined. In this experiment, the size of cementites is hardly influenced by the different coiling temperatures since the amount of carbon is less than the equilibrium C in solution at the lower coiling temperature. During heating, C dissolves from the cementite and the amount of C as cementite is about 17 ppm in the hot bands regardless of the coiling temperature. During heating, C dissolves from the cementite and the amount of C in solution increases. Matsuo et al. and Ushioda et al. reported that the dissolution of cementites during annealing was accelerated if they were refined. In this experiment, the size of cementites is hardly influenced by the different coiling temperatures since the amount of carbon is less than the equilibrium C in solution at the lower coiling temperature of 650°C. C dissolving from the cementite during heating was calculated using a mathematical model under the assumption that the form of the cementite was cylindrical, the average interval between two cementites was 20μm and C as cementite was 17 ppm. The amount of carbon dissolving during heating up to 600°C was 14.73 and 0.34 ppm at 10 and 1000°C, respectively.

About 2 ppm of N in solution exists in the specimen coiled at 650°C while the specimen coiled at 750°C hardly contains N in solution. Although the amount of N in solution is small, it is likely that N will interact with Al atoms and form fine precipitates, clusters and/or dipoles during heating. The mechanism for the retardation of recrystallization caused by these Al-N products is fundamentally the same as that for impurity atoms, but the effect is expected to be much stronger because of the additional interaction of Al, whose diffusion is much lower than that of C or N. The amount of the Al-N products increases as the heating rate decreases.

Based on these considerations, it has been concluded that the reduction of the apparent activation energy at a higher heating rate is caused by the reduced presence of C and N in solution and fine precipitates, clusters and/or dipoles of AlN. The experimental result, shown in Fig. 4, that slow heating up to 600°C retards recrystallization and that retardation is more remarkable in the specimen coiled at a temperature of 650°C can also be explained in the same way. During slow heating, the amount of elements retarding recrystallization increased and the recrystallization was significantly retarded during the subsequent rapid heating.

Figure 12 shows an experimental result using a steel with 0.05% Ti added to the steel given in Table 1. The experimental condition was the same as the case shown in Fig. 4. The steel was coiled at 750°C and therefore C and N in solution do not exist in the hot band and any dissolution of C is not expected in the heating process up to 600°C. In this case, a slow heating up to 600°C does not affect recrystallization behavior during rapid heating. This result supports the conclusion mentioned above.

4.2 Recrystallization texture

The result shown in Fig. 7 that a specimen heated at a constant heating rate of 10°C/s and another heated at 10°C/s to 600°C and then at 300°C/s have nearly the same texture indicates that the process determining recrystallization texture is the nucleation process and the heating rate influences the nucleation process. The microstructure of the specimen heated at 10°C/s to 600°C and rapidly cooled is characterized by a subgrain structure and the fraction recrystallized is less than 5%. This kind of microstructure is established, the subsequent heating rate has no significant influence on the formation of recrystallization texture.

Nearly the same microstructure as this was observed in the specimen heated at 1000°C/s to 670°C and rapidly cooled. Figure 13 shows the (200) pole figures showing the crystal orientations of recrystallized grains of the two specimens at the initial stage of less than 5% recrystallization determined by EBSP. This result shows that the increase in the heating rate randomizes the nucleation texture consisting of recrystallized grains at the initial stage of recrystallization, which has a similar feature to the recrystallization texture of a completely recrystallized specimen. This finding supports the statement in the previous paragraph.

Why does recrystallization texture randomizes, as the heating rate increases? Nuclei of recrystallized grains are formed through the coalescence or growth of subgrains during the recovery process. The finer the subgrains are, the
quicker the nuclei of recrystallized grains form. The size of the subgrains depends on the orientation thereof. The higher the stored energy of a deformed grain is, the finer the subgrains are. The stored energy measured in deformed grains with various orientations is higher in the succession of subgrains are. The stored energy measured in deformed carbon steel sheets heated at 10°C/s was carried out using the EBSP technique from the viewpoint of micro-scale texture. The crystal orientation-dependent succession of recrystallization mentioned above was confirmed by the study and the recrystallization texture is dominated by {111}.

On the other hand, if a material is heated rapidly and brought up to a high temperature spontaneously, every grain is apt to nucleate and the effect of the driving force on recrystallization which differs from grain to grain depending on crystal orientation is reduced so that the increase in the heating causes the recrystallization texture to be randomized as seen in Fig. 8.

5. Conclusion

The influence of the heating rate on the recrystallization behavior and the resultant texture formation has been investigated in a broad range of heating rates from 1 to 1000°C/s. using an extra low carbon steel, and the following results were obtained:

1) The increase in the heating rate raises the recrystallization temperature.
2) The coiling temperature markedly affects the recrystallization temperature at low heating rates, but scarcely does so at high heating rates.
3) If the specimen is heated slowly up to the initial stage of recrystallization and then heated rapidly, the recrystallization temperature becomes much higher than that in cases where the specimen is heated rapidly from the beginning.
4) A model for predicting recrystallization behavior has been developed. An analysis of the experimental results using a model indicates that the apparent thermal activation energy for recrystallization is changed by the heating rate. Rapid heating decreases the apparent activation energy for recrystallization.
5) This change in activation energy seems to be caused by the formation of fine precipitates or clusters of AlN and the dissolution of carbon from cementite during annealing the amount of which depends on the heating rate.
6) The intensity of the main orientations in the recrystallization texture decreases as the heating rate increases. This tendency can already be observed at the initial stage of recrystallization.
7) If the specimen is heated slowly up to the initial stage of recrystallization, the decrease in the intensity of the main orientations is reduced even though the heating rate subsequently increases.

REFERENCES