Development of a Continuous Annealing Process for the Drawing Quality Steel Strip

By Haruo KUBOTERA,** Kazuhide NAKAOKA,** Kenji ARAKI,** Kaoru WATANABE,** Akihiko NISHIMOTO** and Koji IWASE**

Synopsis
A continuous annealing heat cycle is established for manufacture of drawing quality cold rolled low-carbon capped steel strip. The heat cycle is composed of the recrystallization treatment, rapid cooling incorporating water quenching and subsequent over-aging treatment. Sufficient over-aging can be realized in less than 1 min by virtue of the rapid cooling, in contrast to the past results in which an over-aging time of 30 min to 1 hr is invariably necessary.

A water quenching technique is developed to realize the proposed heat cycle. Sound as-quenched strip shape can be secured by means of sheet water spray that washes away the vapor film forming on the strip surface; the cause of the shape deterioration is non-uniform adherence and disappearance of the vapor film. Influence of the spray nozzle arrangement and operating conditions such as the nozzle header pressure, water temperature and strip travelling speed are clarified.

I. Introduction
The continuous annealing processes have long been applied to the production of the black plate and galvanized sheet.

Investigations into the possibilities of its application to the field of the sheet gauge soft material were conducted already over a decade ago,1,2) but no feasible process was established at that time. The rapid heating, short time soaking and rapid cooling result in the product with finer ferrite grain size, unfavorable recrystallization texture and the super-saturation of interstitial carbon, leading to the poor ductility, lower r-value and the higher aging susceptibility. Among the deficient features, the super-saturated carbon exerts influence on both the ductility and the aging susceptibility, and thus it was pointed out that "if we are to obtain the excellent ductility of batch annealed steel we must find some way to reduce the carbon in solution in the ferrite of continuously annealed steel to level equivalent to those in batch annealed steel."3)

To this objective, two possibilities have been investigated so far, the heat cycle and the steel chemistry. Promising influence of the over-aging treatment was reported earlier by several authors.1,4-6) The over-aging conditions in those papers, however, were commonly at temperatures around 300°C and with the duration of 30 min to 1 hr, and, in the industrial sense, the time is too long for the treatment of continuously traveling strip. The vacuum degassed titanium bearing steel proved to show superior properties,7,8) although it may not be economically feasible for the mass production of commercial quality sheets.

Several years ago, the present authors started to find a feasible method for the mass production of the commercial and drawing quality product from the conventional low carbon steels. After the preliminary investigation on the possibility of the shortening of the over-aging time, they found that the time could be reduced to less than 1 min if the driving force for the precipitation was increased by means of the preceding water quenching.9,11) Subsequently, the industrial feasibility has been recognized through the production test conducted at Fukuyama Works.12) In this paper, the annealing heat cycle and the water quenching method established for the low carbon capped steel are described.

II. Heat Cycle with the Water Quenching and Subsequent Over-aging
The fundamental idea on the heat cycle is to realize the short time annealing cycle by combination of a recrystallization treatment followed by water quenching and a subsequent accelerated over-aging. The recrystallization treatment should be sufficient to develop a proper grain size, a favorable recrystallization texture and at the same time to prevent undesirable carbide structures. A prerequisite to the microstructure and the texture is to use a material cooled high in the hot strip mill as already reported elsewhere.13) The water quenching to be applied prior to the over-aging is to increase the super-saturation of the solute carbon in order to reduce the over-aging time. The starting temperature of the quenching is an important factor to control the level of super-saturation: the over-aging temperature should be in a proper range in which the solubility of carbon is sufficiently low and its mobility is adequately high. In a preliminary brief survey, it was found that the tensile test results comparable to those for the batch annealing could be obtained when a cold-rolled sheet was held at 700°C for 1 min, water-quenched and subsequently treated at 400°C for 1 min.

In view of the above information and past investigations,14,10) effects of the quenching temperature, the overaging condition and the recrystallization condition were studied in that order. The pattern of the heat cycle, nomenclature and the ranges covered for each parameters are indicated in Fig. 1.

1. Material and Experimental Procedure
Hot-rolled samples of ordinary low carbon capped steel were subjected to the experiment. The chemistry and hot rolling condition are listed in Table 1. The 2.8 mm thick hot-rolled sheet was cold reduced by an experimental mill to 0.8 mm and cut into rectangular

pieces 35 mm wide and 200 mm long with the longitudinal direction parallel to the rolling direction. The rectangular sheet were recrystallized in a salt bath of a predetermined temperature, air cooled to the quenching temperature desired, then immersed into still water and subsequently over-aged in another salt bath of a predetermined temperature.

The heat treated samples were machined to the JIS No. 5 tensile specimen, temper rolled by 1.0% along the longitudinal direction and tensile tested. Some of the temper rolled specimen were held in a chamber of 38°C for 8 days to assess the susceptibility to aging.

2. Results and Discussion
1. Effect of the Quenching Temperature

The experimental results are shown in Fig. 2 for the recrystallization condition at 700°C for 60 sec and the over-aging at 450°C for 60 sec. The differences between the dotted curves and the corresponding solid curves indicate the aging tendencies. The restoration of the yield point elongation and the deterioration of the elongation clearly indicate that the aging susceptibility is reduced monotonically with increasing temperature. This is in agreement with the expectation that the super-saturation accelerates the precipitation.

The internal friction changes monotonically in a reasonable manner as shown in Fig. 3. It should be noted that the Snook peak height is lower than that of the batch annealed sample when the quenching temperature is 600°C or higher. The elongation and the yield stress show the maximum and the minimum, respectively, for the same temperature of 600°C.

Morphology of the carbides was examined by the direct observation of thin foils with an electron microscope. The result is shown in Photo. 1. For the samples quenched from 500°C and subsequently over-aged, tiny carbides were observed mainly at grain boundaries. Many precipitates were observed in the matrix along with the boundary carbides when the quenching temperature was raised to 600° and 700°C. The precipitates in the matrix seem to have an adverse effect on the ductility, but up to the quenching tem-

<table>
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<tr>
<th>Finishing temperature (°C)</th>
<th>Coiling temperature (°C)</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>N</th>
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<tr>
<td>850-855</td>
<td>680-700</td>
<td>0.037-0.062</td>
<td>0.27-0.32</td>
<td>0.010-0.014</td>
<td>0.017-0.024</td>
<td>0.0013-0.0026</td>
<td>0.033-0.056</td>
</tr>
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Table 1. Chemistry and hot rolling conditions
Quenching temperature: (a) 500°C; (b) 100°C; (c) 700°C

Photo. 1. Influence of quenching temperature on the carbide morphology after the over-aging treatment
(Transmission electron micrographs)

It can be concluded that the optimum quenching temperature is 600°C because for this temperature the ductility is best and at the same time the internal friction is below the level of the batch annealed products.

2. Effect of the Over-aging Condition

The samples were annealed at 700°C for 60 sec, water-quenched from 600°C and subsequently subjected to the over-aging treatment. The effect of the over-aging temperature is shown in Fig. 4 for the aging time of 60 sec. The yield point elongation restored on and the elongation deteriorated by the aging temperature of 600°C, the beneficial effect of scavenging the interstitial carbon can predominate.

Both show the minimum for the treatment at 400°C. The elongation and the yield stress change only slightly in the temperature range between 350° and 450°C. However, the elongation deteriorates considerably for the treatments at 300° or 500°C, and the yield stress is raised markedly for the treatment at 300°C. These observations imply that neither of the 300°C treatment nor of the 500°C treatment is adequate because the mobility of solute carbon is too small in the former and the solubility is too large in the latter.

The effect of the over-aging time for the treatment at 400°C is shown in Fig. 5. Prolongation of the aging time beyond 30 sec does not give any noticeable benefit. It can be surmised from this figure that the amount of the precipitates increases very radidly in
the first 10 sec, leveling off immediately after this period.

It can be concluded that the appropriate range of the aging temperature is between 350°C and 450°C and that 30 sec are long enough for the over-aging at this temperature range. Here it should be noted that the accelerated over-aging can be realized in an over-aging time of much less than 1 min, a period of time that is remarkably shorter than the previously employed duration.1,4-6

3. Effect of the Recrystallization Condition

The heating temperature dependence of the yield strength and elongation is shown in Fig. 6 for a soaking time of 60 sec, a quenching temperature of 600°C and an over-aging condition of 400°C for 120 sec. The yield stress shows the minimum for the heating at 720°C, whereas the elongation remains almost constant for temperatures below 720°C, but it lowers slightly above that.

Microstructures were examined to account for these tendencies. The result is shown in Photo. 2 for the treatments at 680°C, 720°C and 760°C each for 60 sec. The ferrite grain size increases monotonically with the temperature, but there is a distinct difference in carbide morphology for the intracritical and subcritical temperatures. Namely the higher yield stresses for temperatures lower than 720°C are due to the smaller grain size, whereas the higher yield stresses and lower elongations for temperatures higher than 720°C are attributed to the presence of the pearlite colonies which offset the favorable effect of the larger ferrite grains. The hardening due to heating at temperatures over $A_1$ point in continuous annealing of black plate has also been attributed by Mohri,13 Garber17 and Williams15 to the pearlite colonies.

As for the soaking time, the same conclusion was drawn also from the present experiment as from those by Mohri,13 Garber17 and Williams15 that 40 sec were sufficient because no substantial further grain growth could be expected by extending the time.

III. Water Quenching Technique

In order to realize the heat cycle established in the preceding section for a traveling strip, a reliable water quenching technique, one which does not impair the as-rolled strip shape, must be developed.

An industrial application of water quenching to the production of low carbon martensitic steel sheets was developed at Inland Steel Company.16 The primary object of the Inland's technique was the attainment of a high quenching rate necessary to assure the desired martensitic micro-structures. Devices such as the counter flow and the immersed spray system were contrived to increase the heat transfer rate by means of turbulent flow.

As is evident from the result of the experiments of the preceding section, in which water quenching was
conducted by a mere immersion into still water, no special devices to raised the heat transfer rate are necessary for the present purpose so far as immersion water quenching is employed. Thus the primary objective of this investigation is the attainment of a good as-quenched strip shape. This is very important since the product to come off the process should be endowed with the flatness of the ordinary cold-rolled products such as to comply with the requirement for the application, for example, to panels of electric appliances.

In this section, first, the cause of the strip shape deterioration is clarified, and secondly, counter measures are discussed.

1. Deterioration of Strip Shape

Deterioration of the strip shape, which takes place in the form of occurrence of edgewaves to general crumpling of the strip at its worst, is given rise to as a result of non-uniform distribution of thermal stress which exceeds the elastic limit. An example is illustrated in Photo. 3.

An observation of the cooling process in the course of water quenching was performed to find the predominant factors causing the shape aggravation. A 500 mm high glass vessel with a square bottom of 500 mm × 500 mm was prepared and filled with water. A steel sheet 0.8 mm thick, 300 mm wide and 400 mm long was heated to 700°C in an electric furnace, pulled out of the furnace and then immersed into the water straight away. Instantly a vapor film appeared over the strip surface, but it faded into the surrounding water, first at the edge of the strip, then irregularly at the inside of the strip width and finally the whole surface was covered directly with the water. The cooling stages are indicated schematically in Fig. 7.

The whole process occurred in only 2 sec, and the quenched strip came up with a bad shape such as shown in Photo. 3. The strip temperature was measured at the center and at the edge of the strip width. The result is shown in Fig. 8, in which the temperature differences between the center and edge are also shown together with the cooling curves for each of the locations. The arrows and attached symbols (a) to (d) indicate the cooling stages corresponding to the schematic representations with those same symbols of Fig. 7. The cooling curve for the center has a transition point at around 400°C. It has been confirmed that this is a reflection of the transition from film boiling to nucleate boiling.

In contrast to this, the cooling curve for the edge shows a monotonic change and is of a higher cooling rate from the beginning, indicating that the nucleate boiling begins at a very early stage. The temperature difference showed the maximum of over 300°C about 0.5 sec after the immersion. At this instant the temperatures for the center and edge are about 550°C and 300°C, respectively. The yield strength of the strip for these temperatures is about 8 and 14 kg/mm². If the termal stress is to obey the elementary principle that the stress be given as the product of the temperature difference (JT), thermal expansion coefficient (α) and Young's modulus (E), with JT = 350°C, α = 13.6 × 10⁻⁶ and E = 1.8 × 10⁴ kg/mm², 86 kg/mm² is the estimated thermal stress. This value is far over the yield strengths such that the shape deterioration can be accounted for.
Another cooling experiment, immersion into ethanol, was also tried. The strip shape turned out to be quite good. In this case the film boiling was maintained for over 4 sec, and the greatest temperature difference, of mere 120°C, was observed only after the temperatures of the center and edge dropped to below 200°C.

The wavy edge of the as-quenched strip results from the plastic strain caused by the thermal stress. Therefore the occurrence of the wavy edge can be prevented when the temperature difference between the center and edge of the strip is reduced to such a degree that thermal stress does not exceed the yield strength. By assuming that the yield strength is 17 kg/mm², the value approximately corresponds to the yield strength at 200°C, the condition for edge-wave prevention is given as ∆T<70°C.

However, it was assumed that substantially sound strips could still be obtained when the temperature difference was held to smaller than 100°C, because quite a good shape was realized in the ethanol quenching experiment where the maximum temperature difference was 120°C.

2. Method for the Strip Shape Improvement

On the basis of the above experiments and discussion, two measures were proposed as possible ways for the improvement of the as-quenched strip shape:

1) Maintaining the uniform film boiling as long as possible
2) Realizing the uniform nucleate boiling throughout the cooling.

It was discovered that the spraying is a possible candidate to accomplish the second way; the vapor film could be washed away by adequate spraying.

Again determination of the cooling curves was conducted, this time under the presence of the spraying. A sheet spray covering the whole width of the specimen was applied to the cooling surface, and influence of the impact pressure was investigated. The result is shown in Fig. 9. As the spray impact pressure increases the temperature difference between the edge, as measured at a quarter of the width inside from the edge, and the center decreases; and the cooling curve of the center changes its shape, gradually losing the transition point. It was confirmed that under the impact pressure in excess of 10 cm aq., the temperature difference became below 100°C, and that the corresponding strip shape was of a tolerable flatness (Photo. 4).

Thus it is concluded that the strip shape deterioration can be avoided by means of water spraying with an impact pressure of over 10 cm aq. until the strip temperature goes down below the Leidenfrost temperature, the temperature at which transition from film boiling to nucleate boiling occurs.

3. Spray Header and Spraying Conditions

A spray header was designed to study the influence of the spraying conditions. Figure 10 is a vertical section of the spray header. A baffle plate is placed between the feeding pipe and orifices to ensure uniform spraying over the width of the strip. Elongated rectangular orifices with the opening of 2 mm by 100 mm are provided so that sheets of water are directed against the strip surface.

A proper combination of the orifice spacing and the header interval was selected to assure the impact pressure to be in excess of 15 cm aq. throughout the spray zone and at the same time the pressure distribution to be fairly uniform. 17

In the following description nozzle arrangement is represented by a notation as (a) b(c) d(e). This means:

Photo. 4. As-quenched strip shape improved by virtue of the spray quenching

Fig. 9. Effect of spraying on the cooling curve and the temperature difference

Fig. 10. Vertical section of the spray header

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The top slit is inclined downwards by 45° and its opening is $a$ mm.

The interval between the top and the 2nd slit is $b$ mm.

The 2nd slit is horizontally set with an opening of $c$ mm.

The interval between the 2nd and the 3rd slits is $d$ mm.

The 3rd slit is horizontally set with an opening of $e$ mm.

A cold-rolled sheet 0.8 mm thick, 900 mm wide and 2000 mm long was heated in an electric furnace to 700°C and quenched in the spray zone formed between a pair of identical spray headers.

The strip shape was assessed in terms of an index called $SI$ (for Shape Index) defined in the appendix. Physical meaning of $SI$ is that when a strip has edge waves, the length of the edge as measured along the strip contour is increased by an amount of $SI$ as compared to the 1000 mm long dead flat sample, namely, the smaller the $SI$, the flatter, or the better, the strip. Relation between $SI$ and the conventionally defined inspection rank of edge wave is shown in Table 2. It may safely be assumed that no problem will arise in the processing if $SI$ is controlled to under 100 $\mu$. When the first water sheet was jetted perpendicularly to the strip surface, however, any increase in the header pressure tended to disturb the uniformity of the jetting, and splashing occurred at a higher header pressure on account of the reflected water, causing non-uniformity in the initial cooling along the strip width. It was then found that the non-uniformity could be alleviated by orienting the first slit downwards by 45°. By means of this device, $SI$'s of below 100 $\mu$, which could not be realized in the case of spray arrangement with the horizontal first slit, were readily achieved as shown in Fig. 11. It is also indicated in the figure that the occurrence of the splash is suppressed by orienting the first sheet jet downwards.

Figure 12 shows the influence of the nozzle interval and header pressure on the $SI$. The numerals indicate the $SI$ value, and A, B, and C are the ranks of edge waves (Table 2). The increase in $SI$ with increasing nozzle interval is accounted for by the gradual appearance of dead zone in the impact pressure distribution with increasing interval.

Figure 13 shows the effect of the water temperature. A marked shape aggravation is observed when the

<table>
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<th>Table 2. Correspondence of $SI$ and the rank of edge wave</th>
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<td>Rank of edge wave</td>
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<tr>
<td>$SI (\mu)$</td>
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<td>Range</td>
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Fig. 11. Effect of orienting the first slit 45° downwards

Fig. 12. Influence of the nozzle interval and header pressure on $SI$

Fig. 13. Effect of the water temperature on $SI$

Fig. 14. Effect of the immersion velocity on $SI$
temperature exceeds 50°C. This may indicate that a water temperature above 50°C serves to make the vapor film form so easily on the strip surface that a complete washing-away becomes no longer tenable with ordinary spraying.

Figure 14 shows the effect of strip immersion velocity, the velocity at which the strip is introduced into the spray zone. The strip shape is improved monotonically as the immersion velocity increases, regardless of whether the spray header is immersed in water or not. This can be attributed to the tendency that as the velocity increases an adverse effect of the splashing is reduced and at the same time strip feels more uniform impact pressure distribution on traveling.

IV. Summary and Conclusion

A continuous annealing cycle and a water quenching method to realize the cycle were established.

The annealing cycle comprises recrystallization treatment at just below $A_i$ point, water quenching and subsequent over-aging at a temperature range between 350° to 450°C. The recrystallization and over-aging can each be accomplished in less than 1 min. Favorable microstructure and corresponding good mechanical properties are obtained when the strip is water-quenched from around 600°C.

Deterioration of as-quenched strip shape can be avoided by spray quenching to be so applied as to minimize the thermal stress by washing away the vapor film forming on the strip surface. The shape preservation is accomplished when the temperature difference between the center and edge of the strip is held to less than 100°C. This is realized by means of sheet spray with an impact pressure in excess of 10 cm aq. Fairly good as-quenched strip shape is secured by means of proper nozzle arrangement and spray impact pressure. It has been shown that lower water temperature and higher traveling speed lead to better strip shape.

The fundamental data of this paper were applied to the construction and operation of the Nos. 1 and 2 Continuous Annealing Lines of Fukuyama Works. Further progress has been made in many aspects of the process, such as the establishment of manufacturing technique for deep drawing quality sheet and high strength sheet with unique properties.

Acknowledgements

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Appendix

Definition of SI (Shape Index):

\[ SI = \sum_{i=1}^{n} \left\{ \sqrt{(a_i - a_{i-1})^2 + (b_i - b_{i-1})^2} - (a_i - a_{i-1}) \right\} \]

where, \(a_i - a_0 = 1\) mm.

The \(a_i\) and \(b_i\) are defined in Fig. A-1.

Fig. A-1. Determination of SI