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Effects of Heat Cycle and Carbon Content on the Mechanical Properties of Continuous-annealed Low Carbon Steel Sheets

Takashi Obara, Kei Sakata, Minoru Nishida, Toshio Irie

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Effects of Heat Cycle and Carbon Content on the Mechanical Properties of Continuous-annealed Low Carbon Steel Sheets*[1]

Takashi OBARA*[2] Kei SAKATA*[3] Minoru NISHIDA*[4]
Toshio IRIE*[5]

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1 Introduction

Continuous annealing of low-carbon cold rolled steel sheets is a technology which has long been investigated. For instance, Hague and Brace[1] had already studied continuous annealing of low-carbon steel sheets in 1936, and indicated that in the annealing heat cycle, high-temperature annealing followed by overaging treatment, as shown in **Fig. 1**, was necessary. After much subsequent study, Blickwede[2] proposed an application of hot rolled high-temperature coiling, thereby establishing the metallurgical foundation for the continuous annealing of drawing quality steel sheets using low carbon steel.

Since 1972, many continuous annealing facilities have been constructed,[3-5] and detailed studies of the continuous-annealing process have been undertaken.[6-9]

This report describes studies and tests concerning the metallurgical basis of the continuous-annealing process for low carbon steel, and particularly concerns rapid-cooling and overaging processes.

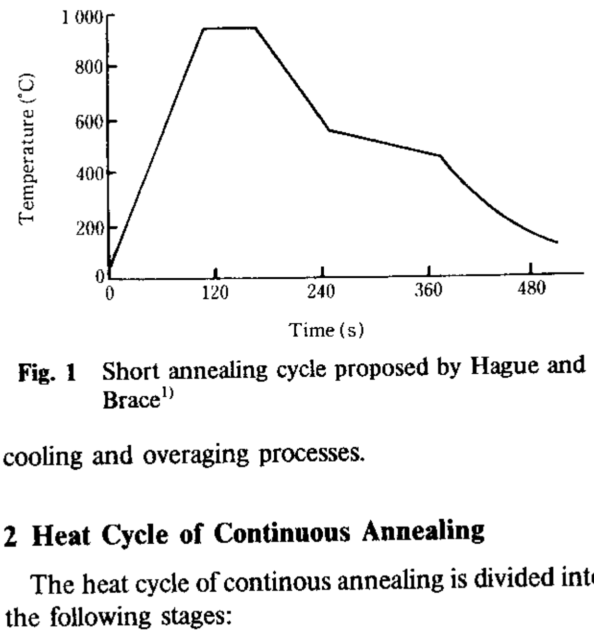

Fig. 1 Short annealing cycle proposed by Hague and Brace[1]

2 Heat Cycle of Continuous Annealing

The heat cycle of continous annealing is divided into the following stages:

*[1] Originally published in *Kawasaki Steel Giho*, 16(1984)4, 264–272

*[2] Senior Researcher, Sheet Lab., I. & S. Research Labs.
*[3] Mizushima Lab. III, Mizushima Research Dept., I. & S. Research Labs.
*[4] Staff Assistant General Manager, Hot Rolling Technology Sec. Hot Rolling Dept. Chiba Works
*[5] General Manager, Light Flat-Rolled Products Research Dept., I. & S. Research Labs.

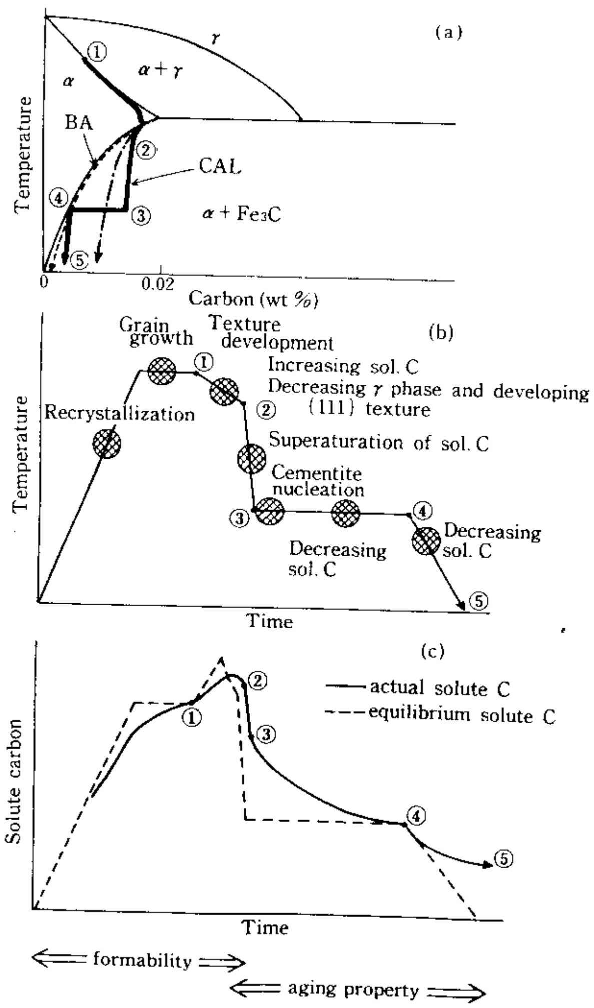

Fig. 2 Schematic illustration of (a) a change in the phase diagram, (b) a metallurgical change and (c) a change in solute C content

(1) high-temperature short-term soaking
(2) slow cooling to below A_1 temperature
(3) rapid cooling
(4) holding in the vicinity of 400°C
(5) final cooling to room temperature

Particularly important here are the stages from (3) to (5); i.e., processes from rapid cooling through overaging to final cooling. The key point in the continuous-annealing techniques is to decrease solute carbon during these processes.

Figure 2 schematically shows the structural changes and changes in solute carbon content during the continuous-annealing processes. In the stages up to the rapid-cooling starting temperature ②, texture and solute carbon content change. After stage ②, a process begins in which the grain size and texture do not change at all, while a decrease in solute carbon, namely, the precipitation of cementite, occurs. If Fig. 2 (b) "temperature change" is compared with Fig. 2 (a) "Fe-C phase diagram," changes in solute carbon content during annealing can easily be understood. Since the cooling rate is very slow in the case of box annealing, solute carbon decreases as temperature drops nearly along the solubility limit of carbon in the equilibrium phase diagram as shown by the dotted line. By contrast, in continuous annealing in which the material is simply cooled to the vicinity of room temperature, temperature changes are quick and deviate greatly from the equilibrium state of solute carbon content, as shown by the one-point chain line. In continuous annealing which uses rapid cooling and overaging, the basic processes consist of promotion of cementite nucleation due to rapid cooling from ② to ③, an efficient decrease in solute carbon content by holding the temperature at an appropriate level (③ → ④) and final cooling to room temperature (④ → ⑤). Figure 2 (c) schematically shows changes with time in solute carbon content and their deviation from the equilibrium value. These changes are examined in detail below.

2.1 Soaking Temperature

As-hot-rolled steel having the chemical composition shown in **Table 1** was used in the study. The coiling temperature was 700°C, and sheet thickness was 2.6 mm. After cold rolling the specimen to a thickness of 0.6 mm in the laboratory, it was heat-treated in various cycles by a direct resistance heating type heat-treatment simulator and its tensile properties were investigated after 0.8% skinpass rolling. For the tensile test, JIS (Japanese Industrial Standards) No. 13B in the rolling direction was used. Aging index (*AI*) was obtained by measuring the difference between the flow stress at 7.5% prestraining and the yield stress after subsequent heating at 100°C for 30 min.

The results are shown in **Fig. 3**. When the annealing temperature is raised, yield strength (*YS*) and tensile strength (*TS*) drop, and elongation (*El*) and Lankford value ($\bar{r}$ value) improve. However, when the annealing temperature rises to 875°C and exceeds the A_3 transformation temperature, *El* and the $\bar{r}$ value suddenly deteriorate. In general, however, below the A_3 transformation temperature, properties of the steel improve as the annealing temperature rises.

2.2 Effect of Slow Cooling after Soaking

When the specimen is rapid-cooled to the overaging temperature immediately after soaking, the island-shaped γ phase, which results from the dissolution of

Table 1 Chemical composition of hot bands used

(wt %)

Steel	C	Mn	P	S	sol. Al	N
A	0.035	0.26	0.014	0.014	0.020	0.0032

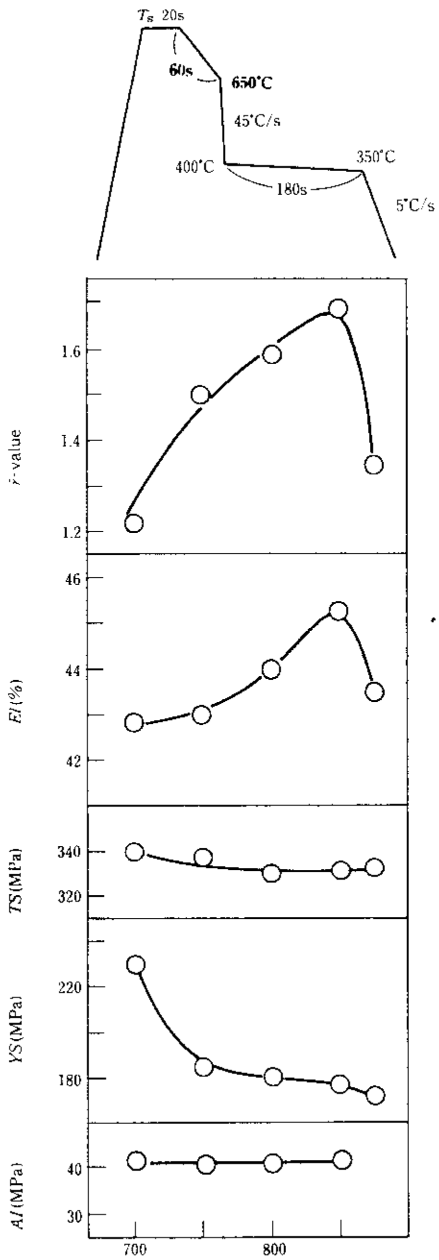

Fig. 3 Effect of soaking temperature on the mechanical properties

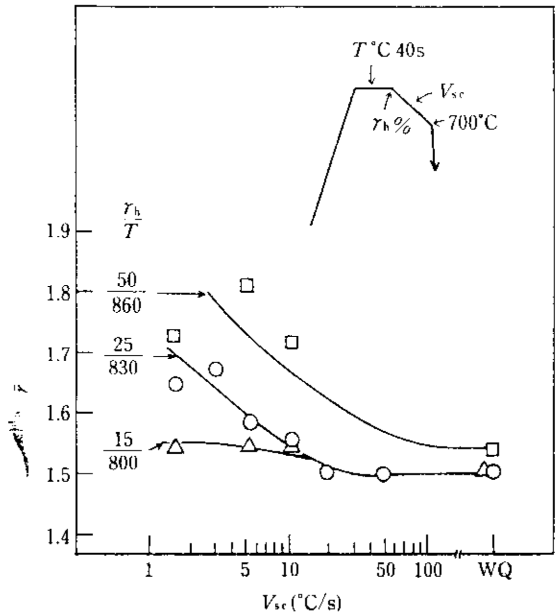

Fig. 4 Relation between V_{sc} (cooling rate during γ-α transformation) and $\bar{r}$-value of 0.018% C steel annealed at 800°, 830° or 860°C. γ_h is volume fraction of austenite at each temperature[10]

coarse cementite, becomes a hard fine pearlite structure, and therefore, it is necessary to slow-cool the specimen to the vicinity of the A_1 temperature to decrease the γ phase fraction. Slow cooling in the $\alpha + \gamma$ region can be expected to have the following two important effects.

Figure 4 shows changes in the $\bar{r}$ value when 0.018%C steel is soaked at 800 to 860°C and the cooling rate to 700°C is changed. Slow cooling at high-temperatures improves the $\bar{r}$ value.[10] This is because the region whcih has become the γ phase by high temperature soaking, preferentially transforms by slow cooling into the {111} orientation due to selective $\gamma \rightarrow \alpha$ transformation during cooling.[10]

Solute carbon content of the α phase, on the other hand, increases in the $\alpha + \gamma$ region, as the temperature decreases, as schematically shown in Fig. 2, and becomes highest at the A_1 temperature. Below that temperature, solute carbon content again decreases. In order to cause efficient cementite precipitation in the overaging process, a slow-cooling from the $\alpha + \gamma$ region to the A_1 temperature beforhand is most important as it increases solute carbon content in the α phase.

2.3 Rapid-cooling Starting Temperature

Figure 5 shows the effects of rapid-cooling starting temperatures following slow cooling, which were tested in a similar manner described in Sec. 2.1. The optimum value of the rapid-cooling starting temperature lies at or slightly below the A_1 temperature. When the specimen is rapid-cooled from a temperature higher than the A_1, the reduced supersaturation of solute carbon is insufficient to promote cementite precipitation during overaging. This will result in an increased solute carbon content at the final stage and an increase in the hard pearlite fraction, thereby increasing *YS* and *TS* as well as *AI*. When the rapid-cooling starting temperature is below 650°C, solute carbon again decreases, resulting in an insufficient supersaturation, thereby increasing *YS* and *AI*.

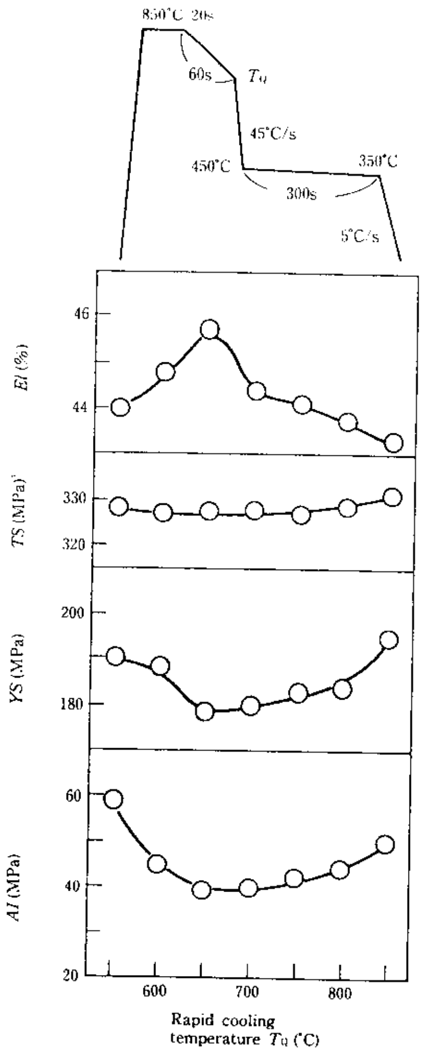

Fig. 5 Effect of rapid-cool strating temperature (T_Q) on the mechanical properties

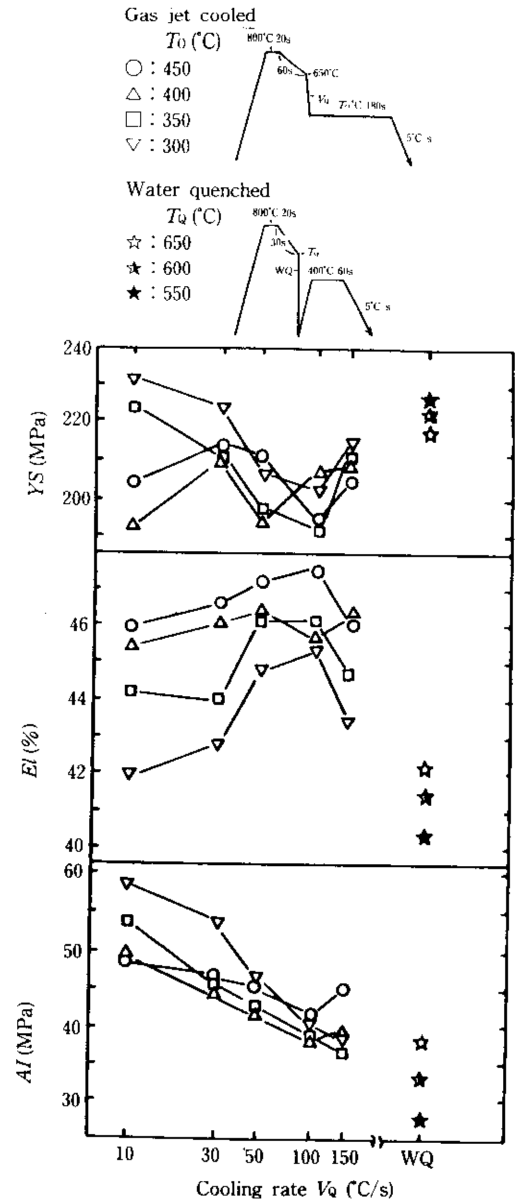

Fig. 6 Effect of cooling rate (V_Q) and overaging temperature (T_0) on the mechanical properties of 0.035% C–0.020% Al steel. The hot band used was hot-rolled by a production mill where finishing and coiling temperature were 860°C and 700°C, respectively

The optimum rapid-cooling starting temperature depends upon the slow-cooling rate and subsequent rapid-cooling rate. Thus, when the rapid-cooling rate is about 50 to 150°C/s, a temperature within the range of 650 to 700°C becomes the optimum rapid cooling starting temperature.

2.4 Rapid-cooling Rate and Overaging Conditions

As-hot-rolled sheets, 2.6 mm thick, with chemical composition as shown in Table 1 and coiled at 700°C, were cold-rolled into 0.6 mm thick sheets in the laboratory where heat treatmens were then performed. The specimens were soaked at 800°C, and then annealed under various cooling rates and rapid-cooling starting temperature conditions after slow cooling, as shown in **Fig. 6**. The tensile characteristics of these specimens were studied, with the findings shown in Fig. 6. As the rapid-cooling rate after slow cooling increases, *AI* drops at a continuous rate, but, within the range of 50 to 100°C/s, *YS* and *El* improve. Tensile properties deteriorate when the cooling rate becomes either slower or faster than this range. As the cooling rate increases and the overaging temperature becomes lower, cementite precipitates more densely and solute carbon content becomes susceptible to decrease, as shown in **Photo 1** and **Fig. 7**. As the cementite precipitates more densely, solute carbon can be effectively reduced in a shorter time, but when cementite dispersion becomes too

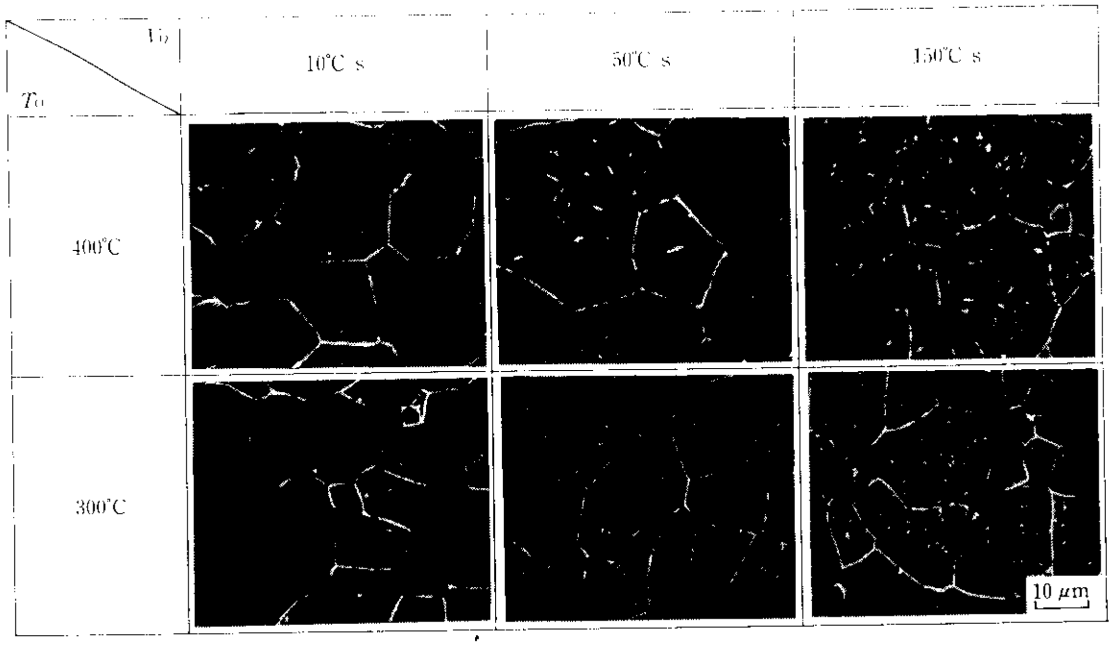

Photo 1 Scanning electron micrographs exhibiting the effect of cooling rate on the carbide microstructures of 0.035% C-0.020% Al steel sheets

dense, *El* generally deteriorates, as shown in **Fig. 8**. Consequently, there is an optimum cementite dispersion condition in which solute carbon content can be effectively reduced without causing excessive deterioration of *El* and other characteristics.

Upon detailed observation of cementite precipitation in the rapid-cooling and overaging processes, it is found that cementite is precipitating only at grain boundaries, as shown in Photo 1, when the cooling rate is as slow as 10°C/s. As the cooling rate is increased to 50°C/s, cementite begins to precipitate in the interior of relatively larger grains, but no precipitation occurs in the smaller grains. Further, when the cooling rate is increased to 150°C/s, cementite precipitation is observed in the interior of considerably smaller grain. Thus, it is difficult to bring about cementite precipitation near grain boundaries. These changes are schematically shown in **Fig. 9**.

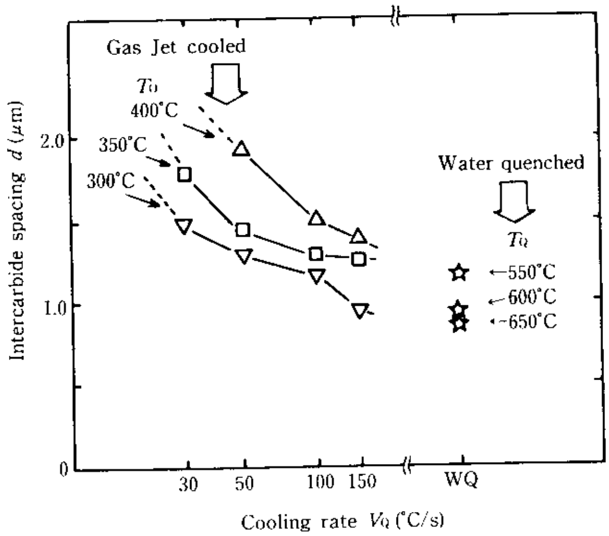

Fig. 7 Average distance between nearest-neighbor pairs of carbides plotted against cooling rate in 0.035% C-0.020% Al steel sheets

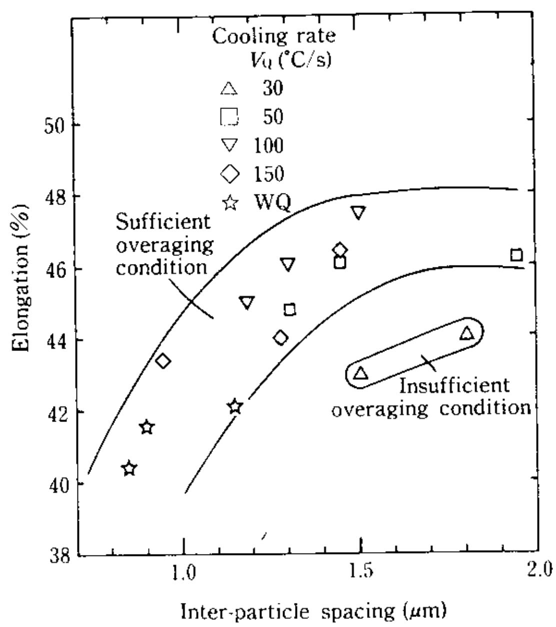

Fig. 8 Elongation plotted against intercarbide spacing

In general, grain boundaries are preferential nucleation sites of cementite. Furthermore, besides grain boundaries, various other precipitates and lattice defects such as dislocation and vacancy in the interior of the grain can be the preferential nucleation sites of cementite. However, cementite nucleation does not always occur at these latent nucleation sites in overaging treatment. It is considered, therefore, that the supersaturation of carbon is perhaps the most important factors governing the cementite nucleation rate in the grain.

Accordingly, the authors estimated changes in solute

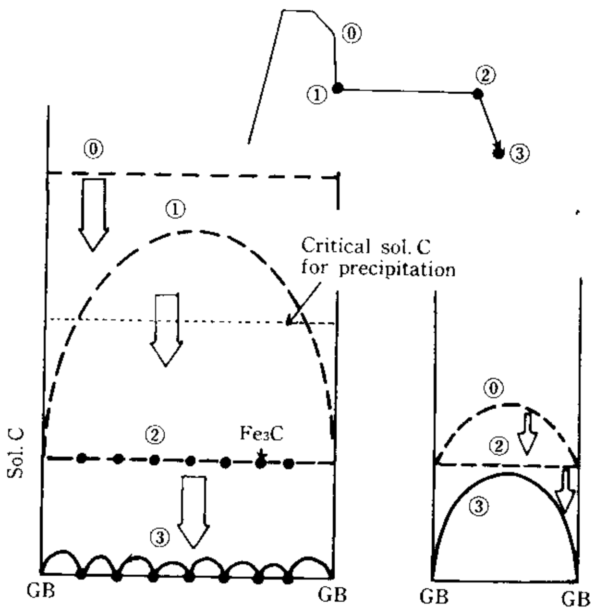

Fig. 9 Schematic illustration of a decrease in solute carbon during overaging in a grain (G B means grain boundary)

carbon content in the ferrite grain during rapid cooling by the method described below. In the estimation, it is assumed that solute carbon is nearly uniformly distributed in the grain before rapid cooling, but diffuses during rapid cooling towards the cementites which have already precipitated at grain boundaries, while cementite is precipitating at high density at grain boundaries. Consequently, it can be assumed that the carbon concentration at grain boundaries has reached approximate equilibrium. Assuming that the grain is of spherical shape, the distribution of solute carbon immediately after rapid cooling was calculated by taking 0.45 exp $(-2\,500/RT)$ cm/s as the diffusion coefficient[11] and 2.55×10^{-2} exp $(-9\,700/RT)$ wt% as the solubility of carbon.[12] **Figure 10** shows the results of the calculation when specimens were rapid-cooled from 650°C to 400°C at a rate of 10 to 150°C/s. At a grain diameter of 10 or 30 μm, supersaturation of carbon varies greatly at equal cooling rates, indicating strong dependence of the supersaturated carbon immediately after rapid cooling on grain diameters. To increase the amount of supersaturated carbon, it is necessary to increase not only the cooling rate but also the grain diameter.

In general, the precipitation process consists of a nucleation stage and a growth stage. The nucleation rate or precipitation density varies greatly depending upon

(1) the supersaturation degree of the solute element (driving force of precipitation),
(2) temperature,
(3) the presence of preferential nucleation sites such as the other precipitates which can generally decrease interfacial energy of cementite nuclei.

Further, consideration should be given to the impingement of the diffusion areas between precipitates as a factor affecting the precipitation density. In other words, since diffusion of carbon in ferrite is fast, solute carbon around cementite immediately begins to decrease, once the cementite begins to nucleate and

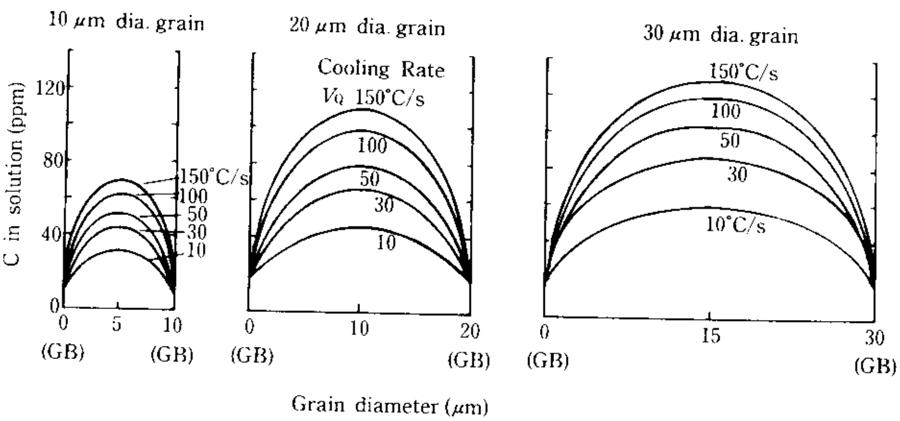

Fig. 10 Effect of cooling rate and grain diameter on the distribution of solute carbon in a grain after rapid cooling from 650°C to 400°C

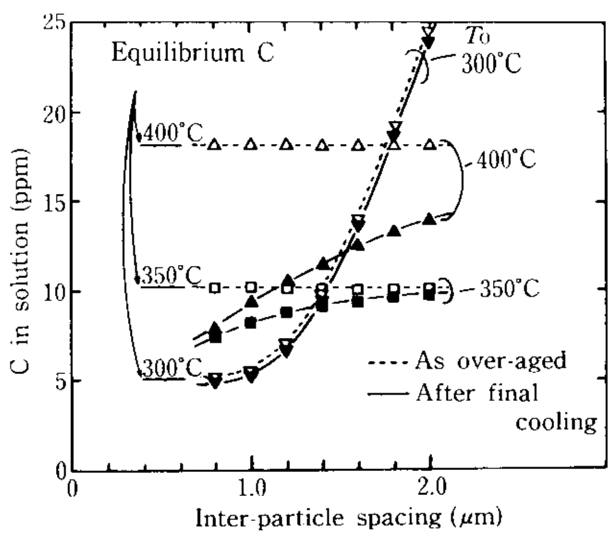

Fig. 11 Calculated maximum solute carbon between carbides after overaging and after final cooling (Equilibrium C means carbon solubility at the overaging temperature)

grow. As a result, the nucleation rate around the cementite is rapidly lowered. Further, when the overaging temperature is high, it affects precipitation density, causing it to decrease. Due to such effects, the precipitation density of cementite is fixed very early in the overaging stage, and the probability of precipitating new cementite during overaging becomes very small.

Having discussed precipitation density, that is, the nucleation rate of cementite, the decrease in solute carbon, namely, the growth of cementite can now be considered.

The diffusion process of solute carbon into cementite during the overaging and final cooling processes was simulated under the simplified assumption that cementite exists at the center of the ferrite grain. Assuming that the diameter of cementite grains is constant at 1/10 that of ferrite, calculations were made using the same method as in Fig. 10. The result is shown in **Fig. 11**. Calculations have been made of maximum solute carbon content at various temperatures after overaging treatment for 3 min at the temperatures of 300, 350, and 400°C respectively and after final cooling at a rate of 5°C/s. At the overaging temperature of 400°C, solute carbon decreases to nearly equilibrium concentration, even if the cementite spacing is as large as 2.0 μm. At 300°C, however, overaging for 3 min cannot lower solute carbon to the equilibrium concentration, unless the cementite spacing becomes as small as about 1.0 μm. Consequently, the optimum overaging temperature lies within the range of 400 to 350°C.

Lowering the overaging temperature permits the precipitation density of cementite to grow, but the diffusion rate of carbon decreases, necessitating longer overaging. This indicates that the overaging temperature should be determined by examination from two viewpoints, that is, the nucleation and the growth of cementite. Recently an attempt has been made to give a generalized expression to the above-mentioned processes.[13] To understand and express the phenomena more precisely, however, more detailed consideration will be necessary in the future.

As explained above, the basis of the technology of manufacturing deep drawing steel sheets by continuous annealing is: Use of steel sheet whose grain diameters become larger after annealing; slow-cooling to A_1 temperature after high temperature annealing; and rapid cooling with holding at about 400°C. Consequently, in terms of equipment, the following technologies are necessary:

(1) Slow cooling in the high temperature range
(2) High speed cooling
(3) Accurate temperature measurement and control

3 Influence of Carbon Content on Rapid-Cooling and Overaging Processes

The effect of rapid-cooling and overaging treatment greatly depends upon not only cooling rate but also grain diameter. It is well known that grain diameter is greatly influenced by carbon content. Therefore, study was made of the effect of carbon content on the tensile property, particularly on *AI*.

Vacuum melted ingots produced in the laboratory were hot-rolled, given simulated high-temperature coiling, and then cold-rolled into a sheet specimen 1.0 mm thick. The specimens were heat-treated by the heat cycle shown in **Fig. 12** and subjected to a 1.0% skinpass rolling, after which the quality of the sample steel was investigated. The results are shown in Fig. 12. As carbon content decreases from 0.05%, the grain diameter becomes larger and the $\bar{r}$ value also increases steadily. The *AI* reaches its minimum when carbon content is around 0.015 to 0.035%. When carbon content is about 0.02%, both *YS* and *TS* reach their minimum, and *El* reaches its maximum level. **Photo 2** (a)~(c) show optical microscopic structures of samples with carbon contents of 0.008, 0.02, and 0.048%. As carbon content increases, the grain diameter becomes smaller, and the amount of islandshaped hard pearlite also increases. As carbon content decreases from 0.048% to 0.02%, the grain diameter becomes larger and the rapid-cooling effect increases. Within this carbon range, therefore, *AI* becomes lower, as carbon content decreases. However, as carbon content decreases to below the 0.02% level, the absolute content of solute carbon becomes smaller and the rapid-cooling effect drops. As a result, when carbon content drops below 0.01%, *AI* again increases. Accompanying this increase in *AI*, increases in *YS* and

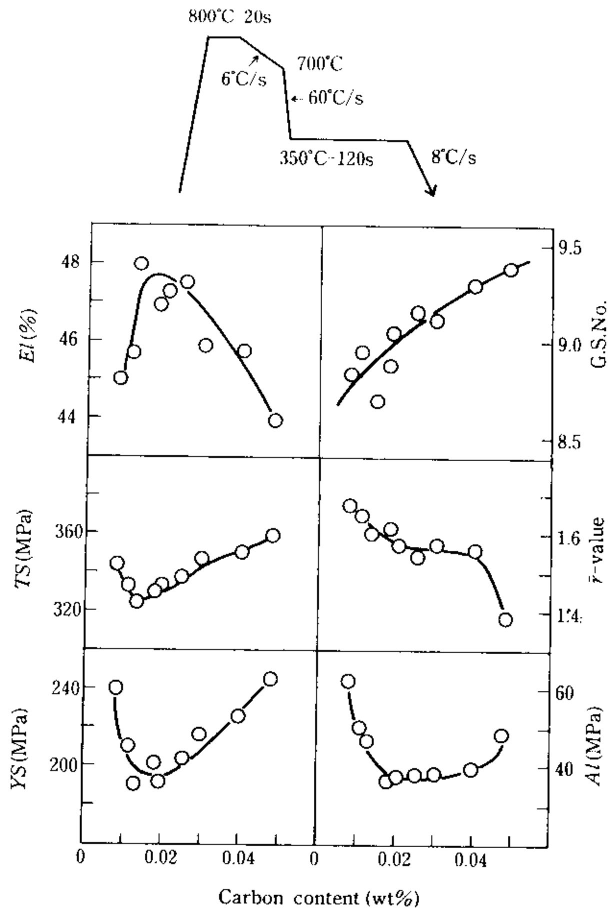

Fig. 12 Effect of carbon content on the mechanical properties of continuous-annealed sheet steels

TS and a decrease in *El* are observed.

The above results can be summarized as follows: Both *El* and *Al* become most favorable when carbon content is within the range 0.015% to 0.035%.

4 Effects of Rapid-cooling and Overaging on Continuous Annealing of Tinplate

In the past, hard tinplates with a hardness of T4 or over were manufactured on the conventional continuous annealing line (CAL), and soft (low-temper) tinplates of T3 or under were manufactured by box annealing. Similar to the case of drawing quality steel, however, the process of manufacturing low-temper tinplates of T3 or under using CAL, which is capable of rapid-cooling and overaging treatment, has now been developed and incorporated into standard operationing procedure.[14] The Metallurgical changes which occur during continuous annealing of low-temper tinplates are essentially the same as those with drawing quality steel, but are different in the following respects:

(1) High-temperature coiling at hot rolling causes coarsening of cementite grains and deterioration of the corrosion resistance (ISV, etc.) of tinplates; therefore high-temperature coiling cannot be used in tinplates as it can in drawing quality steel.
(2) Since sheet thickness is very small, cold-rolling reduction is high.
(3) Annealing temperature is low, specifically, below the A_1 temperature.

Thus, steel sheets for tinplates are coiled at low temperatures, require high cold-rolling reduction and are annealed at a low temperature; therefore the grain diameter of the annealed sheet is considerably smaller than that of drawing quality steel. The difference arising from this smaller diameter is examined below.

First, the grain diameter in tinplate is small, and consequently the supersaturation of carbon obtained even by rapid-cooling is comparatively low. Further, since the anealing temperature is below the A_1 temperature, undissolved cementites still exist in considerable amounts when carbon content is large. The undissolved

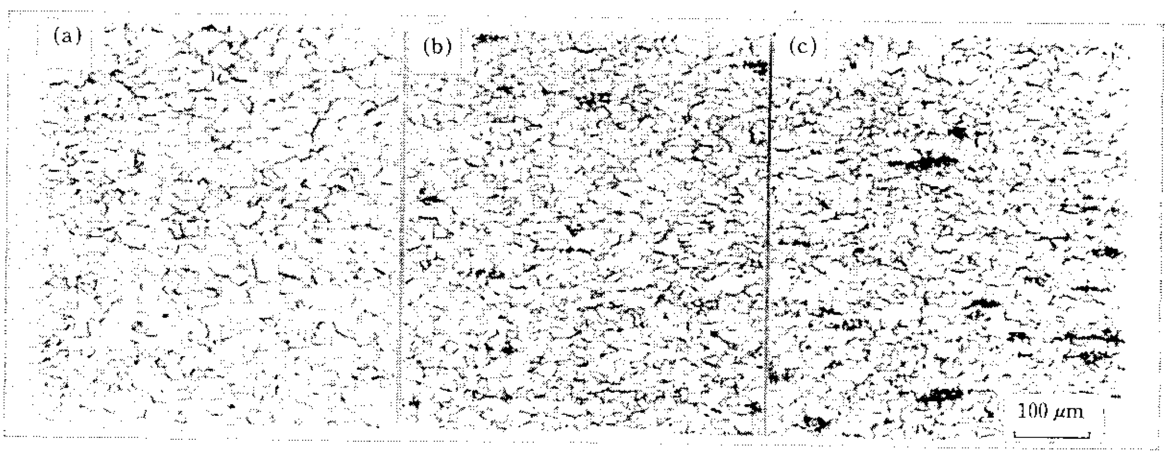

Photo 2 Optical microstructures of continuous-annealed sheet steels with carbon content of (a) 0.008%, (b) 0.020% and (c) 0.048%

Table 2 Chemical composition and hot rolling temperature of the steels used

Chemical composition (wt %)				Hot rolling temperature (°C)	
C	Mn	Al	N	FT	CT
0.011 ~0.090	0.26	0.030	0.0025	860	610

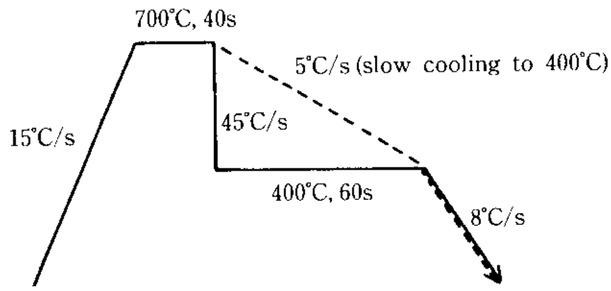

Fig. 13 Heat cycles applied (the solid line and the dotted line mean the heat cycles for low-temper tinplate and conventional hard-temper tinplate, respectively)

cementites become precipitation sites for carbon.

The effects of carbon content on hardness, solute carbon content, and grain diameters of sheets were studied in the laboratory. Chemical compositions and heat cycles are shown in **Table 2** and **Fig. 13** respectively. Specimens were 2.3 mm thick as-hot-rolled steel sheets coiled at 610°C. Specimens were cold-rolled to a 0.32 mm thickness in the laboratory and heat-treated by the direct resistance heating type heat-treating simulator. While solute carbon content was being measured using the internal friction method, the specimens were subjected to 1.5% skinpass rolling and reflow treatment at 250°C for 3 s, after which the hardness of the specimens was measured. The results are shown in **Fig. 14**. For the conventional CAL type annealing in which cooling is done in a simple manner, hardness was at its lowest when carbon content was about 0.06%; whereas for the rapid-cooling and overaging treatment, hardness was lowest at a carbon content of about 0.04%.

In general, hardness of tinplates mainly depends on grain size and solute carbon content. When carbon content decreases, grain sizes increase, which is favorable for decreasing hardness. On the other hand, it is necessary to consider the effects of solution hardening resulting from an increase in solute carbon content. Compared with the case of simple cooling, the rapid-cooling and overaging treatment considerably reduces solute carbon content. As a result, the carbon content level at which hardness is lowest in the rapid-cooling and overaging treatment is lower than the level possible with simple cooling.

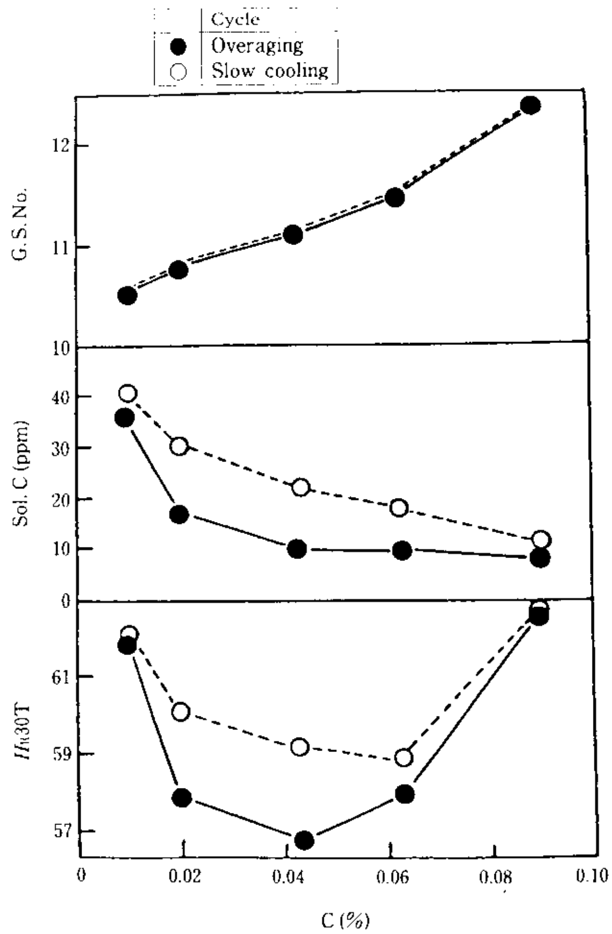

Fig. 14 Effects of carbon content and heat cycles on hardness, solute C content and grain size of tinplate

Examination was made of changes in solute carbon content when tinplates were annealed by the rapid-cooling and overaging cycle. The distribution of solute carbon in the grain, when the grain is small, that is, when no precipitation of cementite has occurred in the grain, has been estimated by the same method as that used in the preceding section. The results are shown in **Fig. 15**. The distribution of solute carbon in the grain, when the specimens were rapid-cooled from 700°C to 400°C at 45°C/s, strongly depends upon the grain diameter in the same way as shown in Fig. 10. When the grain diameter is 5 to 10 μm and the specimen is held at 400°C for 60 s, solute carbon content drops to about equilibrium concentration. It can be seen that when the grain diameter is 5 μm, further considerable decreases are possible in the solute carbon content in the final cooling process of ② to ③. In contrast, when the grain diameter is 15 μm, the distribution of solute carbon in the grain does not become uniform after the specimen is held at 400°C for 60 s, and, of course, solute carbon content after final cooling also increases. When carbon content increases

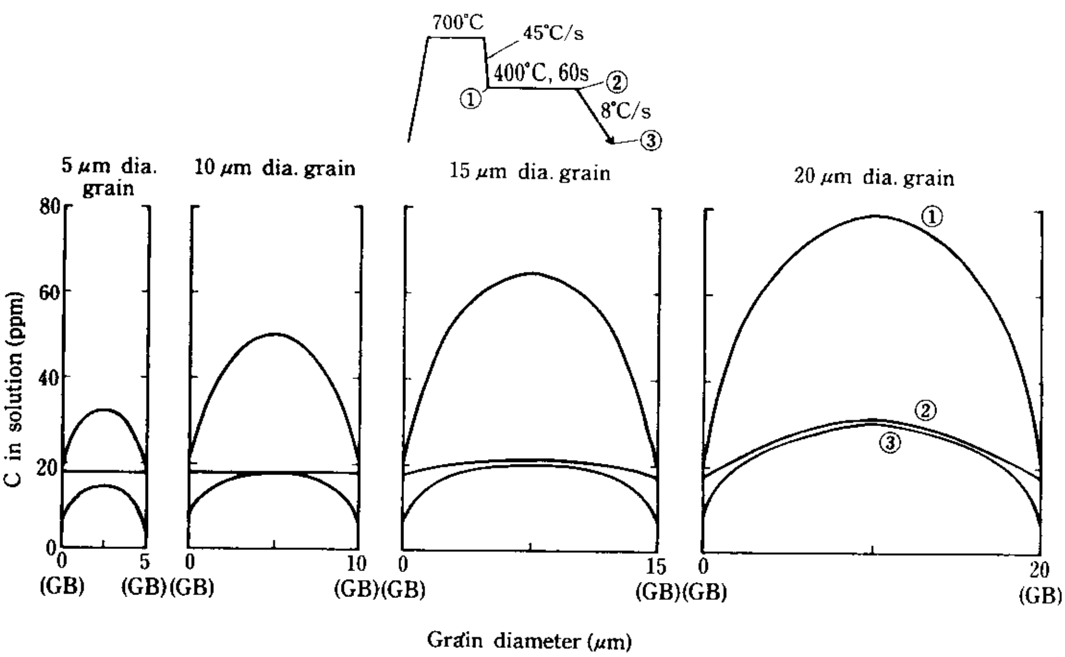

Fig. 15 Effect of grain diameter on the solute C change in a grain during overaging

and a large number of undissolved cementite grains exist at the time of soaking, the same effect is shown as that when the grain diameter becomes smaller. **Figure 16** shows the calculation results of changes in solute carbon content in overaging treatment and the effect of the grain diameter on such changes. When the grain diameter is 10 μm or below, solute carbon in the grain drops to about the equilibrium value within 30 s, but when the grain diameter is 15 μm or above, solute carbon content does not drop to the equilibrium value, even if the specimen is held for 60 s, while solute carbon content after final cooling also increases. If the grain diameter exceeds 20 μm, it is considered that cementite will precipitate in the grain, and there is a greater possibility of more effectively reducing solute carbon, in the same way as with drawing quality steel sheet. Further, the effect of the cooling rate on changes in solute carbon content during overaging treatment have been calculated. The results are shown in **Fig. 17**. The figure indicates that even if solute carbon content immediately after rapid cooling is greatly altered by changing the cooling rate, the solute carbon content after overaging treatment for 30 s will remain almost the same.

As mentioned above, the effects of rapid-cooling and overaging in the manufacture of soft tinplates are slightly different from those in the manufacture of drawing quality steel sheet. In producing drawing quality steel sheet, it is important to increase supersaturation of carbon by rapid cooling and, additionally, to cause dense cementite precipitation in the grain. However, tinplate with smaller grain diameters, even if rapid-cooling is performed, there is little cementite precipitation in the grain. Rather, holding at the overaging temperature is more important for the tinplate.

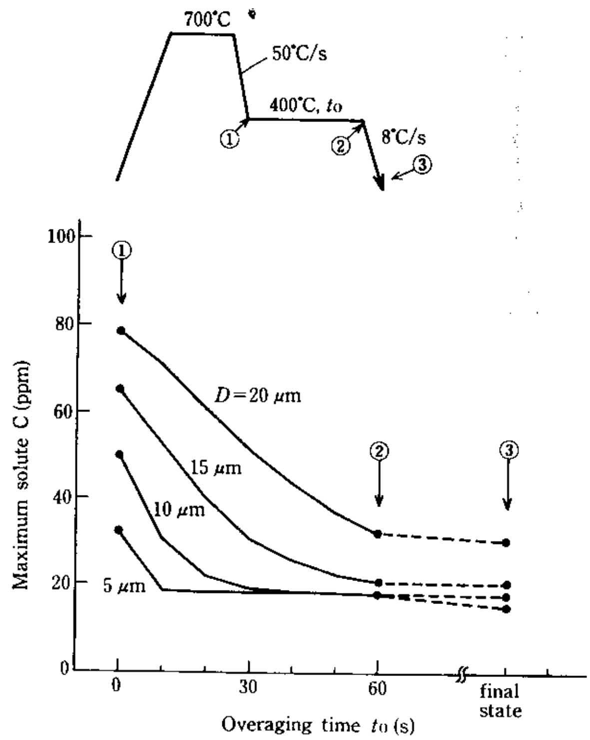

Fig. 16 Effects of ferrite grain diameter D and overaging time on the decrease in the maximum solute carbon content

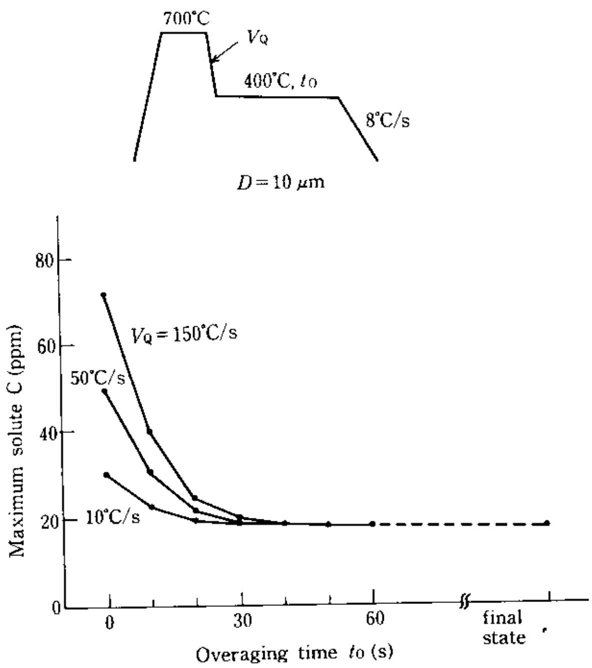

Fig. 17 Effect of the cooling rate on the decrease in the maximum solute carbon content

5 Concluding Remarks

Continuous annealing techniques for cold rolled steel sheets of low carbon steel, particularly with reference to the effects of the rapid-cooling and overaging, can be summarized as follows:

(1) The fundamental metallurigical requirements on a continuous annealing technology for producing deep drawing quality cold rolled steel sheets are the effective achievement of softening and a decrease in solute carbon content, through a combination of the following four processes:
   (a) An increase in the grain diameter by somewhat reducing the carbon content.
   (b) High-temperature annealing and slow cooling to the A_1 temperature or below.
   (c) Subsequent rapid cooling
   (d) Holding at about 400°C

(2) Effects of rapid cooling lie in enhancing supersaturation of carbon and promoting nucleation of cementite precipitation. The distribution of solute carbon during rapid-cooling can be estimated by assuming that the ferrite grain is a sphere and diffusion of carbon to grain boundaries is a rate-controlling process. The decrease in solute carbon in the overaging process can be also estimated by assuming that carbon diffusion to cementite or to grain boundaries is also a rate-controlling process.

(3) High-temperature coiling is not suitable for continuous annealing of low-temper tinplate, because it causes deterioration of corrosion resistance. It differs from deep drawing quality steel in this respect. The grain of annealed low-temper tinplate is of small diameter and contains undissolved cementite.

For tinplate, therefore, the effect of cooling rate is small and it is not a necessarily very important factor. What is important is the effective use of grain boundaries and undissolved cementite, and quick and efficient reduction of solute carbon.

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