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Development of Non-Aging Cold Rolled Steel Sheets with Deep Drawability by a Continuous Annealing Process

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Synopsis :

In order to develop a non-aging, deep drawable cold rolled steel sheet by a continuous annealing process, metallurgical factors affecting mechanical properties of extra-low carbon aluminum-killed steels were investigated. (1) In low C content less than 0.002 wt%, substantial non-aging property can be obtained without overaging treatment by continuous annealing. Improvement of deep drawability by lowering C content is relatively small because the planar anisotropy is extremely large. (2) For decreasing the planar anisotropy, Nb is the most effective alloying element among Nb, Ti, Cr, V, and W. This mainly results from the fact that the grain size of hot band effectively decreases by adding a small amount of Nb. (3) By using the steel containing 0.002wt% C and 0.005-0.010wt%Nb, non-aging cold rolled steel sheets with excellent deep drawability and low planar anisotropy were commercially produced without overaging treatment in continuous annealing line. (4) Using only this type of steel and controlling the processes after steelmaking, various grades (DQ, DDQ, and EDDQ) of non-aging cold rolled steel sheets can be manufactured by a continuous annealing process.

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Development of Non-Aging Cold Rolled Steel Sheets with Deep Drawability by a Continuous Annealing Process*

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In order to develop a non-aging, deep drawable cold rolled steel sheet by a continuous annealing process, metallurgical factors affecting mechanical properties of extra-low carbon aluminum-killed steels were investigated.

- (1) *In low C content less than 0.002 wt%, substantial non-aging property can be obtained without overaging treatment by continuous annealing. Improvement of deep drawability by lowering C content is relatively small because the planar anisotropy is extremely large.*
- (2) *For decreasing the planar anisotropy, Nb is the most effective alloying element among Nb, Ti, Cr, V, and W. This mainly results from the fact that the grain size of hot band effectively decreases by adding a small amount of Nb.*
- (3) *By using the steel containing 0.002 wt% C and 0.005–0.010 wt% Nb, non-aging cold rolled steel sheets with excellent deep drawability and low planar anisotropy were commercially produced without overaging treatment in continuous annealing line.*
- (4) *Using only this type of steel and controlling the processes after steelmaking, various grades (DQ, DDQ, and EDDQ) of non-aging cold rolled steel sheets can be manufactured by a continuous annealing process.*

1 Introduction

Continuation of the production processes is the present trend in the modern steel industry. A continuous annealing process for cold rolled steel sheets is a good example. This process greatly increases the productivity and improves the property of products comparing with a conventional box annealing process. All the cold rolled steel sheets are expected to be produced by the continuous annealing process in the near future.

Most of cold rolled steel sheets are applied to forming uses. Especially, for the use of automobile outer-panels, excellent ductility and drawability, and good resistance to aging are required. Up to now, box annealed low-C Al-killed steel sheets or decarburized and denitrogenized steel sheets by open-coil

annealing have been used for these uses. The same level of aging property as in box annealed coils is difficult to obtain by continuous annealing of a low-C Al-killed steel^{1,2)}. On the other hand, the open-coil annealing method cannot be adopted in continuous annealing because it takes a long time for decarburizing and denitrogenizing.

Extra-low carbon steel (0.005–0.010 wt% C) with more Ti³⁾ or Nb^{4,5)} added than the equivalent atomic ratio of C and N, is well known as interstitial-free steel, from which an extra deep drawing steel sheet is produced. This steel is compatible with continuous annealing process, but the manufacturing cost is high and the surface quality is poor due to a large amount of alloying elements. In contrast with this steel, Kawasaki Steel Corporation has already developed an extra-low-C Al-killed steel (0.003–0.005 wt% C) containing a small amount of Nb (atomic Nb/C ratio is about unity; 0.025–0.045 wt% Nb) for an extra deep drawing use^{6–8)}. This steel sheet can be produced by continuous annealing as well as box annealing, and

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has better ductility than that of a conventional Nb-added interstitial-free steel.

However, the steelmaking technology has progressed steadily to a point that an extra low C steel of "0.001–0.003 wt %C" can be produced with a reasonable production cost^{9,10}. Of course, it has been well known that the lower the C content, the softer the steel becomes, and a decrease in C content improves an aging property. In an extreme case, non-aging property can be achieved without the addition of alloying elements. Therefore, it is expected that the necessary amount of alloying elements can be significantly decreased with reducing carbon content, and furthermore, the role of alloying elements might be different in this extra low carbon steel.

For the purpose of utilizing this extra low carbon steel and producing non-aging and deep drawable cold rolled steels by continuous annealing, the effects of C content and the addition of alloying elements on mechanical properties have been investigated.

2 Experimental Procedures

Both laboratory and plant experiments were performed. The chemical compositions of materials used in both cases are shown in Table 1. Laboratory materials were vacuum-melted 50 kg ingots. After soaking at 1 250°C for 20 min, the forged ingots were hot rolled into 3.8 mm thick where the rolling reduction, the rolling speed, and the finishing temperature were 87%, 40 m/min and 880°C, respectively. The hot bands were heated at 700°C for 1 hr. followed by cooling at the rate of 30°C/h to simulate a coiling process in an actual hot rolling process. Then the cold rolled sheets (reduction 79%) were rapidly heated at 830°C for 40 sec. followed by cooling at the rate of 10°C/s except for the experiment described in Fig. 1.

Materials in plant were made by a bottom-blown converter and an RH-degasser. After heating at 1 220°C, slabs were hot rolled into 3.2 mm thick hot

bands where the finishing temperature and the coiling temperature were 880°C and 700°C, respectively. Then the cold rolled sheets (reduction 75%) were heated at 820°C, held for 30 sec. and cooled at the rate of 20°C/s without overaging treatment.

The annealed sheets were finally temper-rolled at a reduction of 0.5–0.8% and tested their mechanical properties using the JIS No. 5 specimen (25 mm width, 50 mm gage length). The *r*-value was calculated by measuring the widths at three points of a tensile specimen after stretching by 15%; this method is similar to specimen B of ASTM E517. Aging index (AI) was tested by measuring the difference between the flow stress at 7.5% prestraining and the yield stress

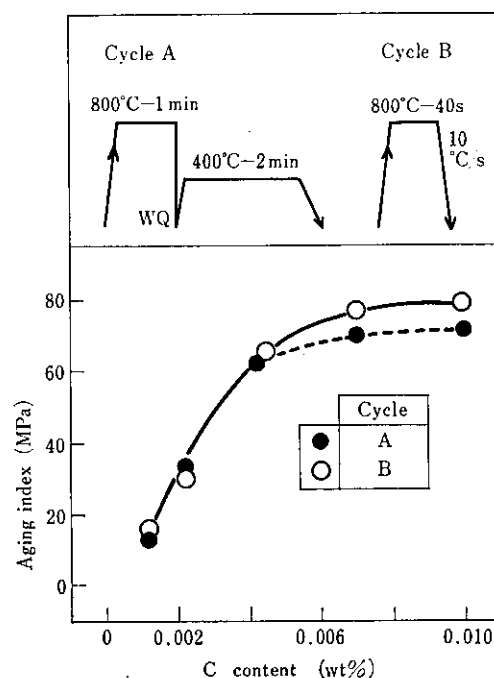


Fig. 1 Effects of C content and annealing heat cycles on aging indices of laboratory-made steels with no alloying elements

Table 1 Chemical composition of materials used

	C	Si	Mn	P	S	sol.Al	N	O	X
For laboratory test	0.001 1	0.02	0.15	0.01	0.01	0.025	0.003 1	0.003	—
	0.010 0								—
	0.001 6	0.02	0.15	0.01	0.01	0.031	0.002 0	0.002	Nb ≤ 0.026
	0.002 2								0.003 2
For plant test	0.001 7	0.01	0.13	0.01	0.01	0.028	0.001 6	0.002	V ≤ 0.060
	0.002 2								0.002 5
									Cr ≤ 0.040
									Nb ≤ 0.017

after subsequent heating at 100°C for 30 min. Non-aging property is substantially ensured when AI is lower than 30 MPa. Aging indices of hot bands were measured by the same method. Total elongation (EI) and r -value were tested in the three directions of 0° (L), 45° (D) and 90° (T) to the rolling direction. The average value (\bar{EI} , \bar{r}) and the planar anisotropy (ΔEI , Δr) were calculated by the following equations.

$$\bar{EI} = (EI_L + EI_T + 2EI_D)/4 \dots\dots\dots(1)$$

$$\bar{r} = (r_L + r_T + 2r_D)/4 \dots\dots\dots(2)$$

$$\Delta EI = (EI_L + EI_T - 2EI_D)/2 \dots\dots\dots(3)$$

$$\Delta r = (r_L + r_T - 2r_D)/2 \dots\dots\dots(4)$$

Microstructures and textures of hot bands and annealed sheets were investigated as follows.

- (1) Crystal grain size: The cross section in the rolling direction was observed by optical microscopy.
- (2) Precipitate: The morphology and dispersion were observed by transmission electron microscopy. The precipitates were electrolytically extracted and then analyzed.
- (3) Texture: (200) pole figures at the center of thickness were measured by reflective X-ray technique.

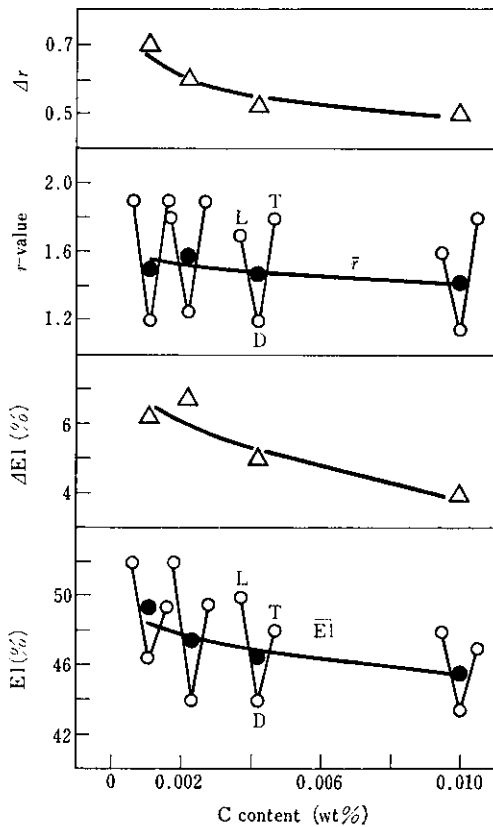


Fig. 2 Effect of C content on mechanical properties and their planar anisotropy of laboratory-made steels with no alloying elements

3 Results

3.1 Effect of Carbon Content on Mechanical Properties (Laboratory Experiments)

When N is stabilized as AlN, the aging property of cold rolled sheets is influenced only by the amount of solute C. The effect of C content on the aging indices of the steel sheets having no alloying elements in **Table 1** is shown in **Fig. 1**. The heat cycle A shown in **Fig. 1** includes overaging treatment. On the other hand, the heat cycle B is a simple one. As for the steels with C content less than 0.002 wt%, the aging indices become smaller than 30 MPa irrespective of the heat cycles. Thus, substantial non-aging property can be achieved, when C content is less than 0.002 wt%. The effect of overaging treatment on the aging indices was small in the range of C content of the steels used in this experiment.

Figure 2 shows the relationship between C content and the mechanical properties of the extra low C steels with no alloying elements. EI and r -value increase with decreasing C content. Those changes, however, are relatively small, considering the large

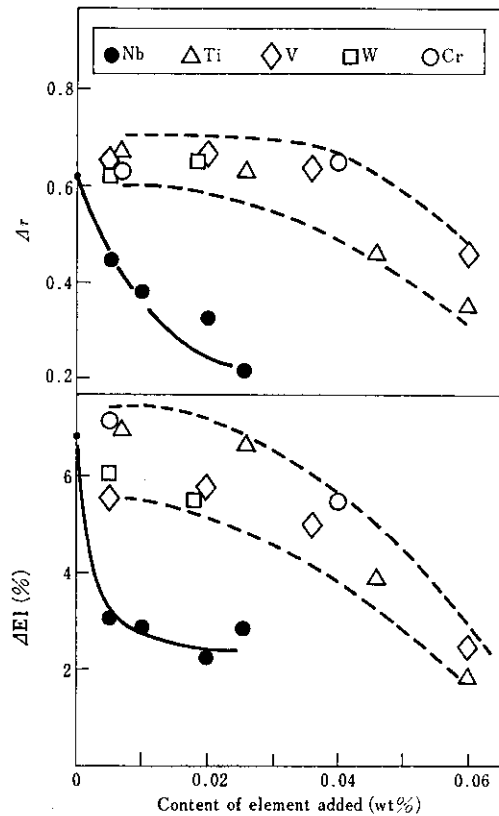


Fig. 3 The planar anisotropy of total elongations and r -values in laboratory-made extra low C steels (0.002 wt% C) plotted against the content of alloying elements

variation of C content. Total elongations and r -values in the longitudinal (L) and transverse (T) directions increase with decreasing C content. On the contrary, EI and r -value in the diagonal direction (D) are lower than those in other directions irrespective of C content.

From the above results, the followings have been clarified.

- (1) Non-aging cold rolled steel sheet can be produced through a continuous annealing process without overaging treatment when the steels with C content lower than 0.002 wt% are used.
- (2) However, the planar anisotropy of the mechanical properties in the steels with C content lower than 0.003 wt% is so large that the improvement of average mechanical properties is relatively small.

Figure 3 illustrates the planar anisotropy of total elongations and r -values in the extra low C steels (0.002 wt% C) with Nb, Ti, V, W, and Cr added. Nb is the most effective element among these carbide forming elements for lowering the planar anisotropy of the mechanical properties.

The superiority of Nb is invariable even if Fig. 3 is arranged by atomic percent or effective amount, for

example, effective Ti defined as (total Ti)—(Ti as TiS)—(Ti as TiN).

3.2 Commercial-scale Experiments

Based on the above results, commercial-scale experiments were performed. The effect of Nb content on the mechanical properties of extra low C steel sheets (0.002 wt% C) is demonstrated in Figs. 4 and 5. The addition of a small amount of Nb (0.005–0.010 wt%) to extra low C steels exceedingly improved total elongations and r -values in the diagonal direction. Consequently, the planar anisotropy (ΔEI , Δr) markedly decreases. In addition, the average mechanical properties (\bar{EI} , \bar{r}) increase with an addition of a very small amount of Nb. In case of Nb content over 0.010 wt%, \bar{EI} slightly decreases and the increase of \bar{r} -value is saturated.

Figure 6 shows the influence of Nb content on aging indices (AI) of extra low C steel sheets (0.002 wt% C). Substantial non-aging property can be obtained even in the Nb-free steel. However, if Nb is added to the steels, the aging indices decrease furthermore. Thus, non-aging property is ensured by using a small amount of Nb.

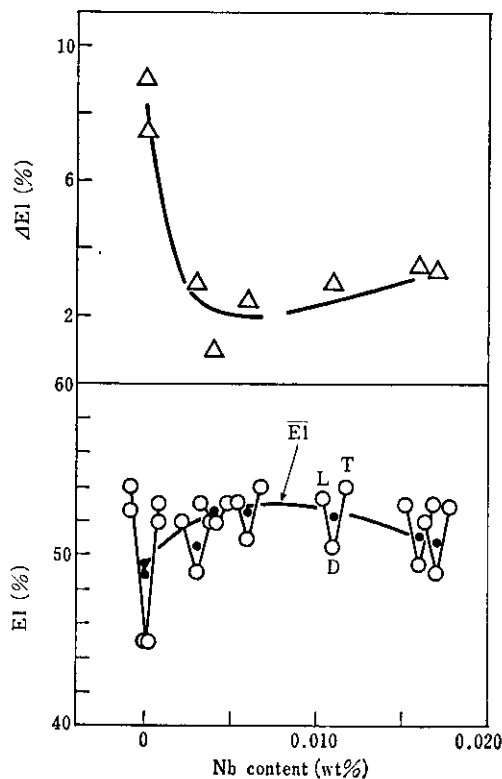


Fig. 4 Effect of Nb content on total elongations and their planar anisotropy of commercially produced extra low C steels (0.002 wt%)

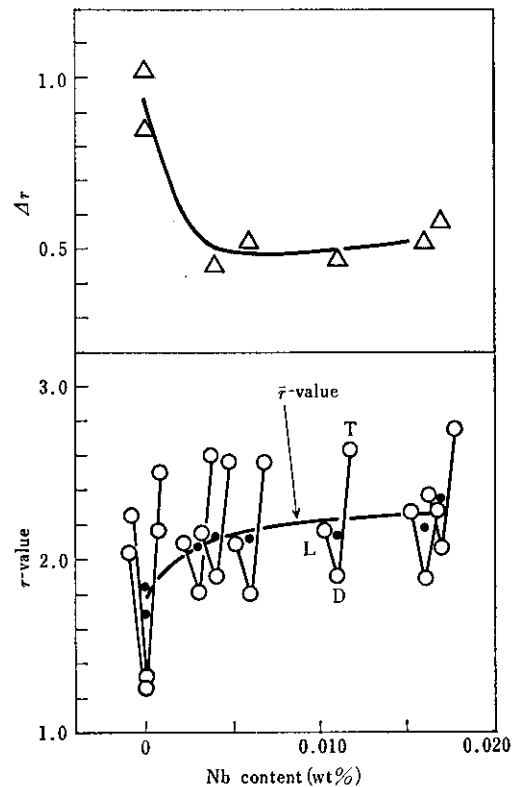


Fig. 5 Effect of Nb content on r -values and their planar anisotropy of commercially produced extra low C steels (0.002 wt%)

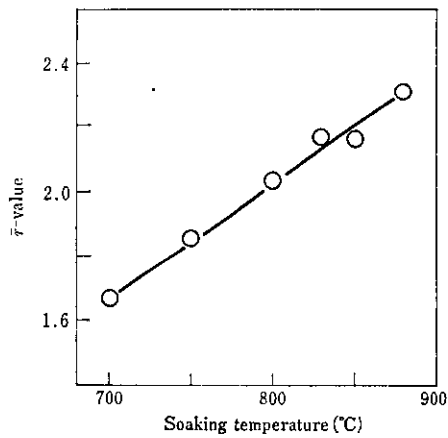


Fig. 6 Relationship between Nb content and aging indices of commercially produced extra low C steels (0.002 wt%)

3.3 Microstructure Changes with Nb Addition

The effect of Nb addition on the microstructures of hot bands and annealed sheets was examined. The chemical composition of hot bands used is denoted in Table 2. Most of N was stabilized as AlN. The production conditions in plant were the same as those described in Chapter 2.

Figure 7 shows (200) pole figures of cold rolled and annealed sheets in the steels C and E. The steel C, Nb-free steel, has strong textures from $\{110\}\langle 001\rangle$ to $\{210\}\langle 001\rangle$ besides a texture of $\{111\}\langle 1\bar{1}0\rangle$. As for the steel E, the intensity of the former orientations is very weak. On the contrary, steel E has stronger textures of $\{111\}$ //ND than that of the steel C.

This result suggests that the planar anisotropy of mechanical properties has connection with recrystallization texture. Hence, factors affecting recrystallization texture were investigated below.

Table 2 Chemical composition of hot bands produced in plant

Steel	C	Si	Mn	P	S	sol.Al	total N	N as AlN	O	Nb
C	0.0017	0.01	0.13	0.01	0.009	0.032	0.0018	0.0017	0.0024	—
E	0.0019	0.01	0.13	0.01	0.010	0.032	0.0021	0.0018	0.0026	0.006
G	0.0020	0.01	0.13	0.01	0.009	0.029	0.0018	0.0015	0.0024	0.011

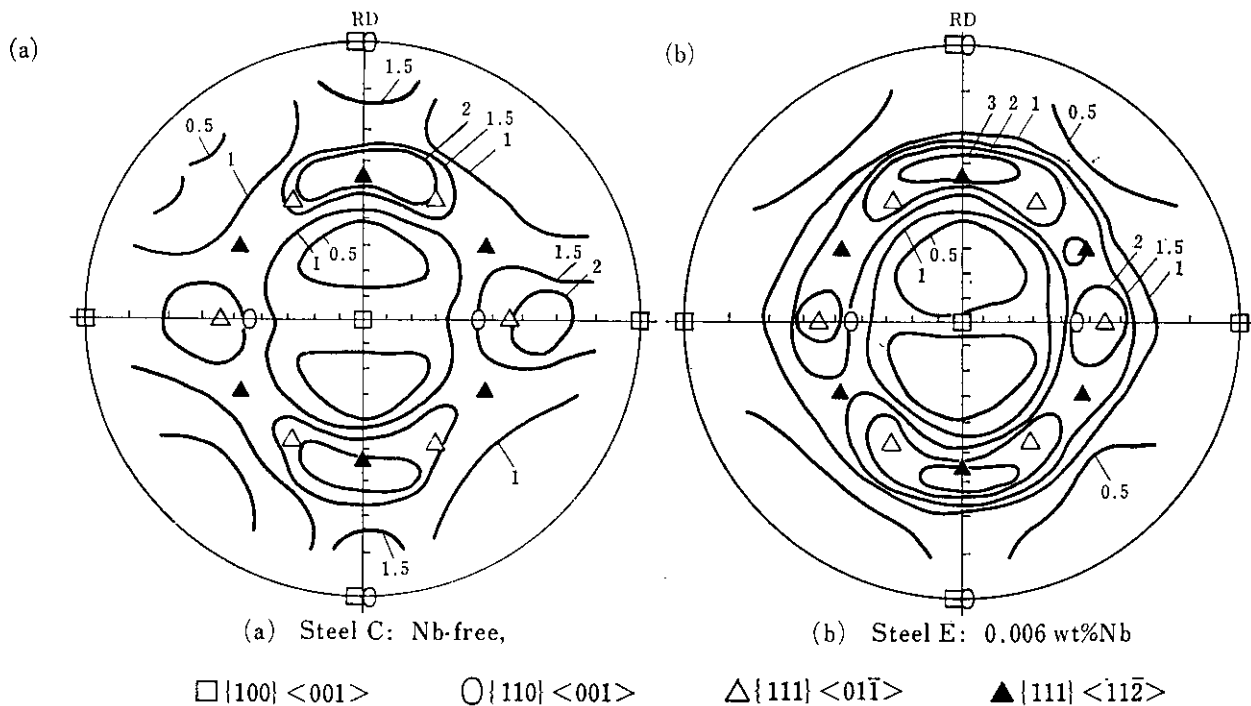


Fig. 7 (200) pole figures showing the effect of Nb content on recrystallization textures of steel sheets annealed in a continuous annealing line

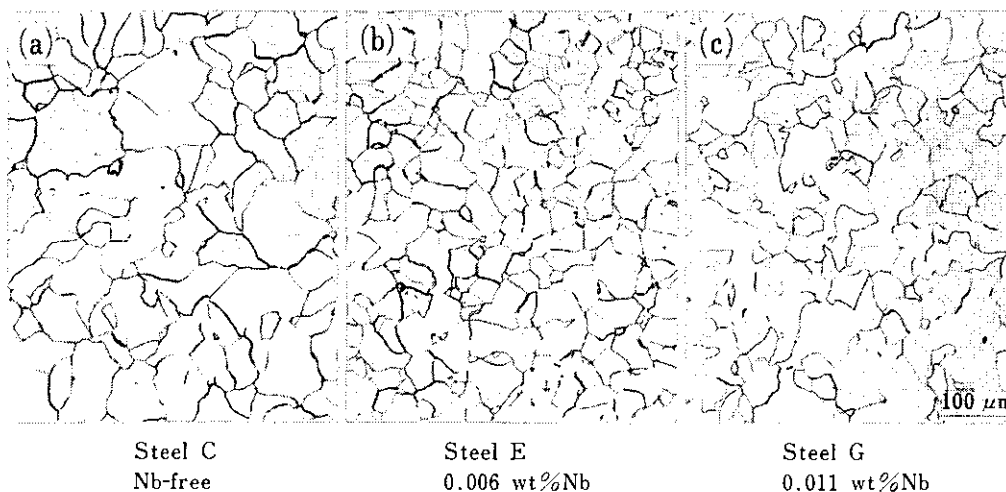


Photo 1 Optical micrographs showing the effect of Nb content on the grain size of commercially produced hot bands

Optical microstructures of the hot bands in the steels C, E and G are shown in **Photo 1**. The Nb addition of 0.006 wt% exceedingly decreases the grain size of hot band. **Figure 8** denotes the effect of Nb content on grain size numbers and aging indices of the hot bands. The grain size becomes fine and the aging index decreases with an increase in Nb content. However, the change of the grain size is larger than that of aging index in the range of low Nb content.

Transmission electron micrographs of hot bands in the steels C and E are demonstrated in **Photo 2**. The density of precipitates increased with Nb addition. However, difference between steels C and E was not so large concerning morphology and dispersion of precipitates. According to the X-ray analysis of the electrolytically extracted precipitates, most of the precipitates in both steels were detected as AlN and MnS. The amount of Nb-carbonitrides was extremely small in the steel E.

There was no large distinction with regard to texture of hot bands and cold rolled sheets (before annealing) in all steels.

4 Discussion

Non-aging cold rolled steel sheet can be produced without overaging in a continuous annealing process by the use of extra low C steel. The planar anisotropy of the mechanical properties, however, is extremely large in a conventional extra low C steel. The planar anisotropy noticeably decreases by a very small amount of Nb addition. Addition of Nb leads to changes in ① grain size, ② aging index, and ③ precipitates of hot band. According to the results described in Paragraph 3.3, the change of grain size was most important factor among others.

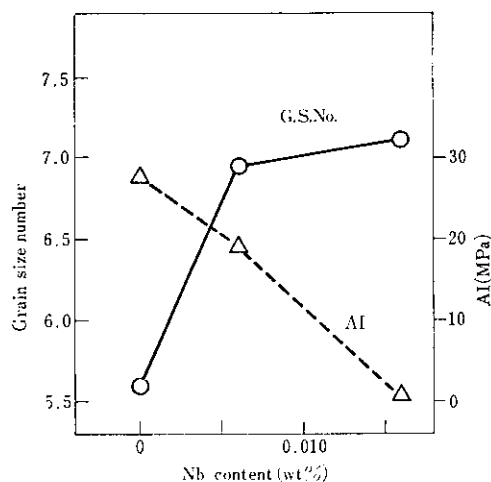
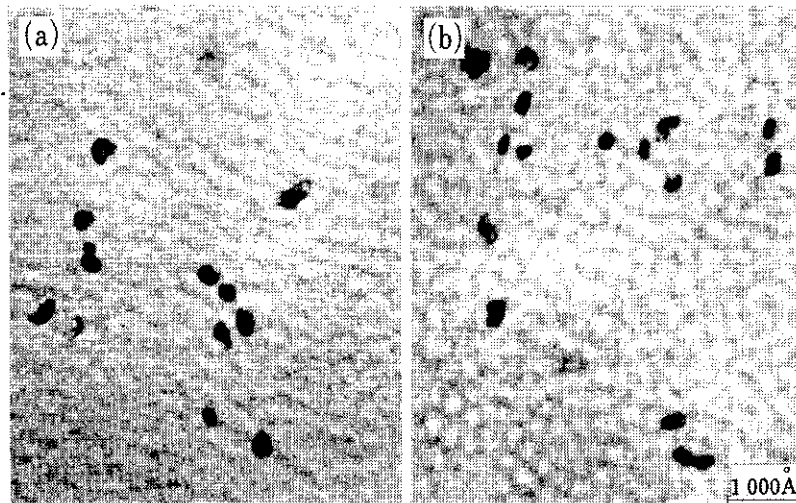


Fig. 8 Grain size number and aging indices of hot bands with composition shown in Table 2 plotted against Nb content

4.1 Effect of Nb Addition on Grain Size of Hot Band

It has been well known that Nb has an extremely strong grain-refining effect. Recrystallization of austenite (γ) phase during hot rolling is restrained in Nb-added steel¹¹⁻¹⁴. The suppression of recrystallization results from ① dragging of solute Nb¹¹ or ② precipitating of fine NbC(N)¹². The latter effect is important to ordinary carbon steels containing C over 0.05 wt%¹⁴.

Content of Nb as NbC calculated from the solubility products¹⁵⁻¹⁸ at 900°C is shown in **Table 3** in case of the steel E (0.0019 wt% C, 0.006 wt% Nb). A γ to α transformation temperature (A_{T3}) of this steel was about 860°C. An amount of Nb as NbC is zero even



Steel C
Nb-free

Steel E
0.006 wt% Nb

Photo 2 Transmission electron micrographs showing the effect of Nb content on precipitate morphology of commercially produced hot bands

Table 3 Nb content as NbC calculated on solubility products at 900°C (Steel E; 0.0019 wt% C-0.006 wt% Nb)

Solubility products	Nb as NbC (wt%)
$\log [\text{Nb}][\text{C}] = -7900/T + 3.42^{(15)}$	0
$\log [\text{Nb}][\text{C}] = -6770/T + 2.26^{(16)}$	0
$\log [\text{Nb}][\text{C}] = -7290/T + 3.04^{(17)}$	0
$\log [\text{Nb}][\text{C}] = -9100/T + 3.7^{(18)}$	0

if Smith's formula¹⁸⁾, which offers the lowest equilibrium solubility product at 900°C and is the most reliable one of four formulas⁷⁾, is adopted. The influence of NbC precipitating after hot rolling upon grain size of hot band is negligibly small because NbC content in hot band of the steel E was low as described in Paragraph 3.3.

Thus, it is deduced that grain-refinement of the Nb-added hot band results from restraint of recrystallization by solute Nb dragging.

4.2 Effect of Nb Addition on Planar Anisotropy of Mechanical Properties

The r -value and its planar anisotropy closely relate to the texture of a steel sheet. As for elongation, Itoh et al.¹⁹⁾ reported that total elongation exhibited high value in the tensile direction where r -value was high, and nonuniform elongation, especially, had strong interrelation with r -value. Therefore, the planar anisotropy of El and r -value essentially depends on the texture of a steel sheet.

Nb-free steel, which had large planar anisotropy of mechanical properties, exhibited strong textures with

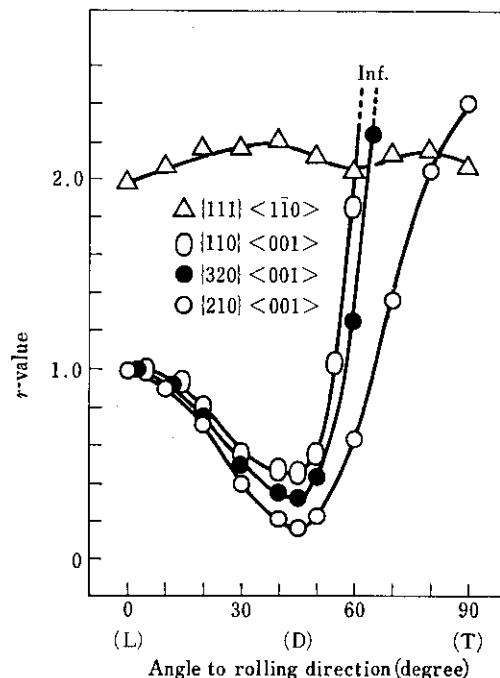


Fig. 9 Relationship between angle to rolling direction and r -values calculated on the assumption that slip systems with Schmid factor higher than 0.1 are activated²¹⁾

{110}<001> and neighboring orientations (<001>//RD). It has been theoretically proved that {110}<001> orientation reduces r -value in the diagonal direction^{20,21)}. Figure 9 illustrates r -values calculated by the method of Kitagawa et al.²¹⁾ concerning {110}

$\langle 001 \rangle$ to $\{210\}\langle 001 \rangle$ orientations. The r -values in the diagonal direction are extremely low in these orientations compared with $\{111\}\langle 1\bar{1}0 \rangle$ orientation. The large planar anisotropy of mechanical properties in Nb-free steel results from the strong texture with $\{110\}\langle 001 \rangle$ and neighboring orientations.

With a small amount of Nb added, the textures with $\{110\}\langle 001 \rangle$ and neighboring orientations became weak and $\{111\}$ //ND texture developed strongly. The reason is discussed below.

Nb addition caused ① a refinement of grain size, ② a decrease in aging index, and ③ an increase in precipitate density of hot band. On the other hand, the effect of Nb addition on the textures of hot band and cold rolled sheets (before annealing) was very small.

Matsuo et al.²²⁾ studied the effect of grain size of mother plate before cold rolling on recrystallization behavior of pure iron. The recrystallization texture depended on the grain size of mother plate, while the grain size hardly affected the cold rolled texture. Fine grain specimen exhibited strong $\{111\}\langle 01\bar{1} \rangle$ recrystallization texture. As for the coarse grain specimen, recrystallization texture with $\{110\}\langle 001 \rangle$ orientation noticeably developed. When the grain size of mother plate decreased, the frequency of $\{111\}\langle 01\bar{1} \rangle$ orientation grains nucleated around grainboundary increased, in contrast with a decrease in the nucleation of $\{110\}\langle 001 \rangle$ orientation grains far from grainboundary. Based on these results, they concluded that mother plate with fine grain was advantageous for the development of $\{111\}$ recrystallization texture because inhomogeneously deformed region around grainboundary was advantageous to the development of $\{111\}$ recrystallized grains.

Takahashi et al.²³⁾ investigated the effect of content of solute N on the recrystallization texture in extra low C rimmed steel sheets. With an increase in the solute N content, $\{110\}\langle 001 \rangle$ component and neighboring orientations increased, while $\{111\}\langle 11\bar{2} \rangle$ component weakened. They insisted that the recrystallization accompanied with the motion of high-angle grainboundary easily occurred because solute N suppressed the motion and the annihilation of dislocations, from the result that $\{110\}$ orientation grains, which could recover rapidly, preferentially grew in the specimen containing high content of solute N.

Terasaki et al.²⁴⁾ studied the effect of precipitates on recrystallization texture using Ti-bearing extra low C steel sheets. They insisted that a distinct restraint of development of $\{110\}$ orientation grains during recrystallization resulted from fine TiC precipitates.

In summarizing the previous studies, $\{110\}\langle 001 \rangle$ and neighboring textures are restrained when ① mother plates (before cold rolling) have a small grain

size, ② content of interstitial atoms (C, N) is low, and ③ fine precipitates exist in the recrystallization stage. In the Nb-added steel, the reason for definite decrease in intensities of $\{110\}\langle 001 \rangle$ and neighboring orientations can be explained by the overlapping effect of the above three factors. However, considering the results described in Paragraph 3.3, the change of grain size of hot band caused by Nb addition is the most important factor for decreasing components of $\{110\}\langle 001 \rangle$ and neighboring orientations, that is, lowering the planar anisotropy of mechanical properties.

5 Application

In order to improve the steel manufacturing productivity, simplification of production processes is as important as their continuation. Mechanical properties required for cold rolled steel sheets strongly differ by its usage. The usage of formable cold rolled steel sheets is very wide. The usage ranges from the parts simply bended to the ones of automobiles demanding extra deep drawability such as fender and high roof.

The production of many kinds of cold rolled steel sheets by the use of only one type of steel will exceedingly contribute to increasing the productivity. The new method for production of various grades of deep drawable cold rolled steel sheets by using only one type of steel has been developed by Kawasaki Steel. It is realized by adoption of a Nb-added extra low C steel as the base steel and control of the production conditions, mainly hot rolling and continuous annealing conditions. Slab-reheating temperature and finishing temperature are especially important among hot rolling conditions^{25,26)}. As for annealing conditions, soaking temperature has a large effect for controlling the mechanical properties. Relationship between soaking temperature (soaking time 40 sec.) and \bar{r} -value of commercially produced steel (0.002 0 wt% C, 0.011 wt% Nb) is shown in Fig. 10. The \bar{r} -values linearly increase with an increase in soaking temperature. Soaking temperature is effective for controlling the \bar{r} -values.

Table 4 demonstrates the average mechanical properties of DQ(SPCD; JIS), DDQ(SPCE; JIS), and EDDQ(Extra Deep Drawing Quality) grade steel sheets produced by the above-described method (C and Nb contents are about 0.002 wt% and 0.010 wt%, respectively). All sheets were continuous annealed by a simple heat cycle without overaging treatment.

On the other hand, concentration of alloying elements to surface layer in these steel sheets hardly occurs because ① annealing time is very short, and ②

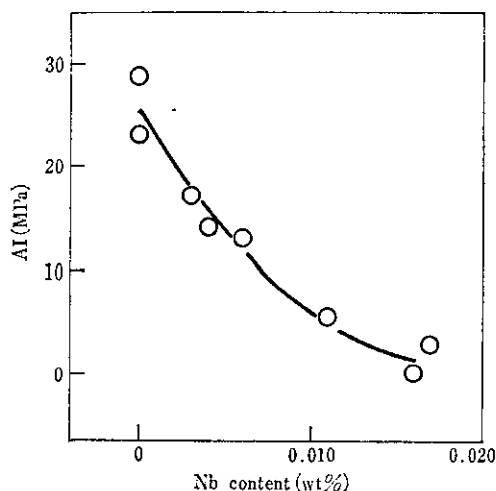


Fig. 10 Relationship between soaking temperature (soaking time 40 s) and \bar{r} -values of commercially produced steel (0.002 0 wt% C, 0.011 wt% Nb)

Table 4 Average mechanical properties of three kinds of steel sheets commercially produced by controlling the manufacturing conditions with using a kind of Nb-added extra low carbon steel

Grade	YS (MPa)	TS (MPa)	El (%)	\bar{r}	AI (MPa)
DQ	176	310	48	1.59	5
DDQ	165	300	49.2	1.76	5
EDDQ*	143	290	52.0	2.10	1

*Extra Deep Drawing Quality

the content of alloying elements such as Nb is extremely low. Thus, these steel sheets have excellent phosphatability.

Moreover, this steel has so excellent adhesion property of zinc-coating that it can be applied to producing formable galvanized and galvanized (including one-side coating) steel sheets by hot-dip galvanizing line as well as electro-zinc coating method.

6 Conclusions

In order to develop non-aging and deep drawable cold rolled steel sheet by continuous annealing process, metallurgical factors affecting mechanical properties of extra low C steels were investigated.

- (1) In low C content steel less than 0.002 wt%, substantial non-aging property can be obtained by

- continuous annealing without overaging treatment.
- (2) Improvement of deep drawability by lowering C content is relatively small because the planar anisotropy is extremely large.
- (3) For the purpose of decreasing the planar anisotropy of the mechanical properties, Nb is the most effective element among Nb, Ti, Cr, V, and W.
- (4) A small addition of Nb rapidly reduces the grain size of a hot band and retards the development of $\{110\}\langle 001 \rangle$ and neighboring recrystallization textures ($\langle 001 \rangle // RD$) which enhance planar anisotropy.
- (5) By using the steel containing 0.002 wt% C and 0.005–0.010 wt% Nb, non-aging cold rolled steel sheets with excellent deep drawability and low planar anisotropy can be commercially produced in a continuous annealing line without overaging treatment.
- (6) Using only this type of steel and controlling the processes after steelmaking, various grades (DQ, DDQ, EDDQ) of non-aging cold rolled steel sheets can be manufactured by continuous annealing.

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