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1 Introduction

Because of their excellent formability, superior surface quality and high dimensional accuracy, cold-rolled sheet steels have extensive applications for automotives body outer and many others. The progress of automotive industry has highlighted the role of the cold-rolled sheet steels as one of major products in the modern steel industry.

Cold-rolled sheet steels, when mainly used for press-forming, are required to possess (1) ductility, (2) drawability, and (3) anti-aging as mechanical properties. The higher the elogation (El) and the lower the yield strength (YS), the sheet steel exhibits the better ductility. The drawability is governed by the crystal orientation of the sheet steel; the higher the {111} intensity normal to sheet steel surface, the more excellent the drawability. The drawability is evaluated by Lankford-value (r-value). The r-values of deep drawable grade DDQ (JIS SPCE) and extra-deep drawing grade EDDQ are approximately 1.8 and 2.0, respectively. Anti-aging property represents resistance to the deterioration of mechanical properties caused by age hardening due to the diffusion of solute C and N remaining in a sheet steel. Non-aging sheet steels with negligible aging deterioration of mechanical properties can be produced by reducing to less than 1 ppm the total solute C and N.

As a notable trend in recent years, automotive parts press-formed with sheet steel have become larger in size and complex in design. Integration of press-formed parts is especially effective in (1) omitting some manufacturing processes such as press-forming and welding, (2) reducing car weight, and (3) preventing corrosion

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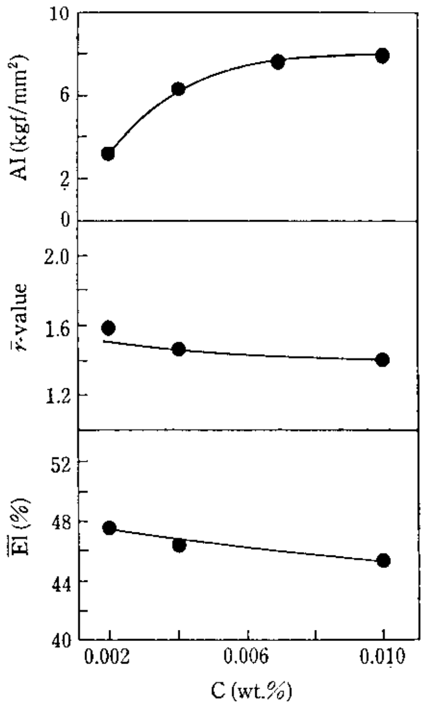

Fig. 1 Effect of C content on mechanical properties of annealed sheet steels with no alloying elements (Annealing temperature: 830°C)

around jointed portions. A grade higher than the conventional EDDQ sheet steel is often required for the integrated press-forming.

In order to improve the mechanical properties of cold-rolled sheet steels, reducing C content in sheet steels is efficient. A typical example is a decarburized EDDQ sheet steel produced by open-coil box annealing. On the other hand, the recent advanced steelmaking technology has made it possible to produce an extra-low C steel (C < 50 ppm) at a reasonable cost.[1,2] With the extra-low C steel, EDDQ sheet steel can be produced by tight-coil box annealing and continuous annealing. However, it is difficult to manufacture an EDDQ sheet steel by merely ruducing C content to less than 20 ppm, as described in **Fig. 1**. Interstitial-free (IF) steels were developed by stabilizing C in extra-low C steels with strong carbide forming elements such as Ti and Nb.[3~7]

This report introduces methods of improving mechanical properties of these IF steels in order to develop new EDDQ steels available for integrated press-forming.

2 Experimental Procedures

The chemical compositions of steels used in the laboratory test are shown in **Table 1**. The starting materials were 30 kg vacuum-melted ingots. The basic chemistry is an aluminum-killed extra-low C steel with the C content of approximately 20 ppm (steel F). Steels T and N are Ti- and Nb-bearing steels, respectively, where alloying elements are added enough to stabilize C as carbides. Steels TN1 to TN5 are Nb co-added steels to a Ti-stabilized steel as the base steel. Table 1 represents the atomic ratios of effective Ti and Nb to the C content. Effective amount of Ti (Ti*) is calculated with the following equation since Ti-sulfide and Ti-nitride are preferentially formed in Ti-added steels, compared with Ti-carbide:

Table 1 Chemical compositions of steels used

Steel*1	C (ppm)	Ti (wt.%)	Ti*(at.%) / C (at.%)	Nb (wt.%)	Nb (at.%) / C (at.%)
F	32	—	—	—	—
T	24	0.045	3.6	—	—
N	22	—	—	0.065	3.8
TN1	20	0.030	2.4	0.004	—
TN2	21	0.026	2.0	0.006	—
TN3	24	0.028	2.0	0.007	—
TN4	18	0.029	2.8	0.016	—
TN5	18	0.029	2.6	0.031	—

*1 Si 0.01, Mn 0.1, P 0.01, S 0.005, N 0.002, Al 0.04 (wt.%)

$$\mathrm{Ti^*}(\%) = \mathrm{Ti}(\%) - \frac{48}{32}\mathrm{S}(\%) - \frac{48}{14}\mathrm{N}(\%) \quad \cdots\cdots(1)$$

The effective amount of Nb is defined as the total Nb content in case of Nb-added steels. As for Ti and Nb co-added steels, only Ti*/C is denoted in Table 1, assuming that Ti has stronger affinity to C than Nb.

These ingots were hot rolled to 30 mm thickness sheet-bars after heating to 1 250°C, and then air-cooled to ambient temperature. These sheet bars were reheated to 1 250°C and hot rolled to 3.3 mm thickness hot-bands (pass number, 3; total reduction, 89%; rolling speed, 40 m/min; finishing temperature, 860°C above Ar_3). The hot-bands were first air-cooled to ambient temperature (average cooling rate until 500°C: 3°C/s). They were soaked at 500°C for 1h, followed by slow cooling at the rate of 10°C/h.

Pickled hot-bands were cold-rolled to 0.7 mm thickness (reduction: 79%). The annealing was performed in the following two methods. As for the testing of mechanical properties, specimens were heated at the rate of 10°C/s, soaked for 20s, then cooled at the rate of 20°C/s by using an alumina-fluidized furnace. The specimens for investigating the recrystallization temperature were heated to specified temperatures at the rate of 10°C/s and directly cooled at the rate of 10°C/s.

Temper-rolling of 0.7% reduction was conducted to annealed specimens for testing mechanical properties. The mechanical properties were determined using the JIS No. 5 specimen (25 mm width, 50 mm gauge length). Total elongation (El) and r-value were examined in the three directions; 0°(L), 45°(D) and 90°(T) with respect to the rolling direction. The average value ($\bar{El}$, $\bar{r}$) and the planar anisotropy (ΔEl, Δr) were calculated by the following equations:

$$\bar{M} = (M_L + 2M_D + M_T)/4 \quad \cdots\cdots\cdots\cdots (2)$$

$$\Delta M = (M_L - 2M_D + M_T)/2 \quad \cdots\cdots\cdots\cdots (3)$$

where, M denotes the value of El or r-value measured by each direction test piece. Aging index (AI) was evaluated by measuring the stress difference between the flow stress at 7.5% prestrain and the lower yield stress after aging 100°C for 30 min.

Microstructure and precipitate dispersion of hot bands were observed by optical microscopy and transmission electron microscopy.

3 Results and Discussion

3.1 Effect of Ti and Nb Addition on Mechanical Properties

The relationship between soaking temperature at annealing and mechanical properties of steels T and N is shown in **Fig. 2**. Both sheet steels exhibited non-aging property with AI less than 1 kgf/mm^2 since they contained enough amount of alloying elements to the C content. The El and r-value strongly depended on soaking temperature at annealing. Nb-added steel, especially, showed lower El at low temperature soaking than Ti-added steel. Ti-bearing steel provided higher El and r-value than Nb-bearing steel even at soaking temperatures higher than 800°C.

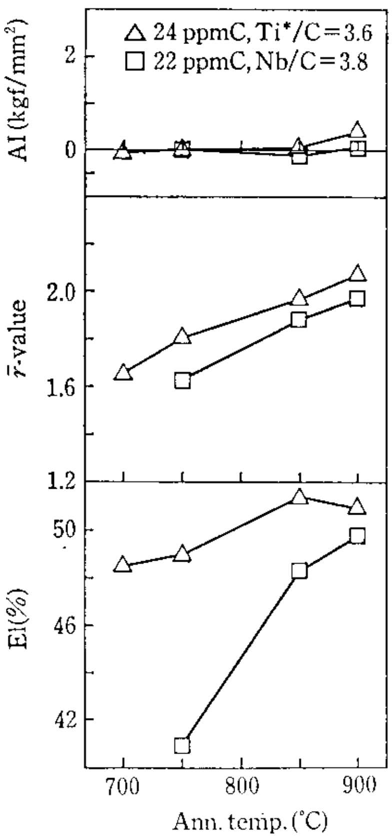

Fig. 2 Comparison of mechanical properties between Ti- (steel T) and Nb-bearing (Steel N) sheet steels

Figure 3 illustrates the recrystallization behavior obtained by the hardness change of steels, F, T, and N. The finishing temperature of recrystallization of steels T and N was higher than 750°C while steel F completed recrystallization at approximately 650°C. Especially, Nb-added steel had higher recrystallization temperatures by about 30°C than Ti-added steel.

Precipitate dispersion in hot bands were observed to clarify the difference in mechanical properties and recrystallization behavior between Ti- and Nb-added steels, as described in Figs. 2 and 3. **Photo 1** demon-

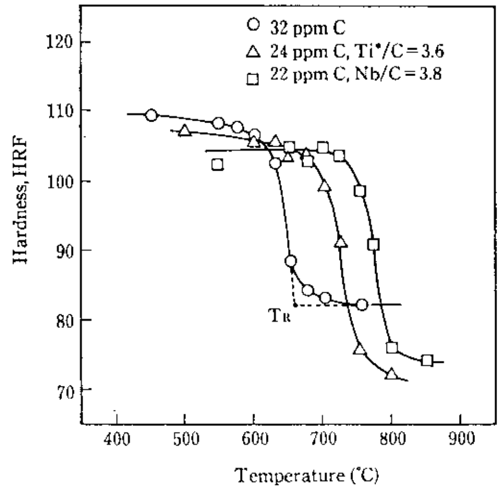

Fig. 3 Effect of steel chemistry on finishing temperature of recrystallization, T_R (steels F, T, and N)

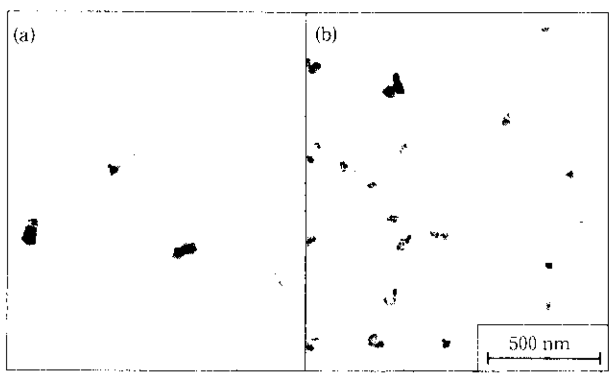

Photo 1 Transmission electron micrographs of (a) Ti- (steel T) and (b) Nb-bearing (steel N) hot bands

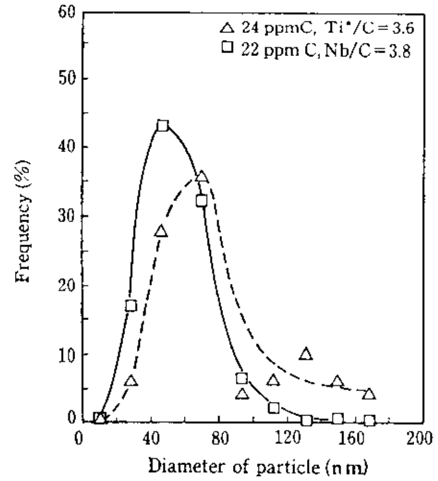

Fig. 4 Distribution of precipitate diameter of Ti- (steel T) and Nb-bearing (steel N) hot bands

strates transmission electron micrographs of replicas extracted from hot bands of steels T and N. Ti-bearing steel showed coarser dispersion of precipitates than in Nb-bearing steel. **Figure 4** shows the precipitate frequency plotted against the particle diameter obtained by image analysis of transmission electron micrographs. Compared with Ti-bearing steel, Nb-bearing steel contained a greater number of fine precipitates with diameters less than 50 nm. In other words, compared with Nb-bearing steel, Ti-bearing steel had higher frequency of precipitates with diameters larger than 100 nm.

Precipitates of hot bands, as shown in Photo 1 and Fig. 4, consists of three kinds of particle, namely carbide, nitride and sulfide. There might exist Fe_3C as a carbide in steel F which contains no alloying elements. However, Fe_3C dissolves within a range from 600°C to the recrystallization temperature. It is thus inferred that the stability of carbides results in the difference in recrystallization behaviors between steel F and stabilized steels (steels T and N). Pinning effect due to Ti- and Nb-carbides might suppress the grain growth during recrystallization since these carbides are stable at a high temperature above the recrystallization temperature. The following favorable effects of the stable carbides are worth noticing. Stabilized steels, where the carbides are stable during annealing, provide a high intensity of $\{111\}$ recrystallization texture favorable for high r-value because of the scavenging effect (very low content of solute C) and/or the precipitate effect. Non-aging property is also obtained after annealing.

In comparing Ti- and Nb-added steels, the following reasons are considered attributable to the difference in the recrystallization temperature between both steels:
(1) Difference in the precipitation dispersion
(2) Difference in the effect of solution Ti and solute Nb
The latter difference might be negligible since no distinct difference in the increment of recrystallization temperature associated with the increase of solute Ti and solute Nb was recognized in other work.[8] Consequently, the difference of precipitate dispersion is considered important to understand the recrystallization behaviors between Ti- and Nb-added steels.

The main precipitates in Ti-added steel are TiN, TiS, and TiC. Nb-added steel contains MnS, AlN, and NbC. These particles start to precipitate at a high temperature in the above order in each steel. Each kind of Ti-precipitate, for example, a nitride, starts precipitation at a temperature higher than that in Nb-added steel.[9–12] Generally speaking, precipitates depositing at higher temperatures are coarsened faster due to high diffusion rate of precipitate elements. The above finding is useful in understanding the difference in precipitate dispersion between Ti- and Nb-added steels, as demonstrated in Photo 1 and Fig. 4.

The pinning force of precipitates against grain growth of recrystallized grains in a deformed structure is described in the following equation.[13]

$$K \cdot \sigma f / r_p \quad \cdots\cdots (4)$$

where K, σ, f, and r_p are a constant, interface energy of recrystallized grains, volume fraction of precipitates, and average radius of precipitates, respectively. Equation (4) represents that, as the volume fraction of precipitates increases and the radius of precipitates decreases, the pinning force of precipitates against the grain growth of recrystallized grains in a deformed structure becomes large, resulting in suppressing the grain growth. As for the latter stage of recrystallization, the pinning force of precipitates against the eating process between recrystallized grains is also described as the similar equation to Eq. (4).[14] The above discussion helps to understand the reason for the higher recrystallization temperature of Nb-added steel containing fine and dense precipitates than that of Ti-added steel. In other words, Ti-bearing steel with coarse precipitates provides faster growth of recrystallized grains than in Nb-bearing steel, resulting in better mechanical properties compared at the same annealing temperature. It is possible to improve the mechanical properties of Nb-added steel by ripening precipitates, for example, through high temperature coiling at hot-rolling.

3.2 Effect of Nb Co-addition to Ti-stabilized Steel on Planar Anisotropy of Mechanical Properties

Small planar anisotropy of mechanical properties of sheet steels is favorable for an actual press-forming since the limit of formability is often determined by the minimum value of each planar mechanical property instead of their average values. The small planar anisotropy of mechanical properties decreases the volume of earing at press-forming, resulting in economical advantage. **Figure 5** shows the average and the planar anisotropy of El and r-value when Nb was co-added to the

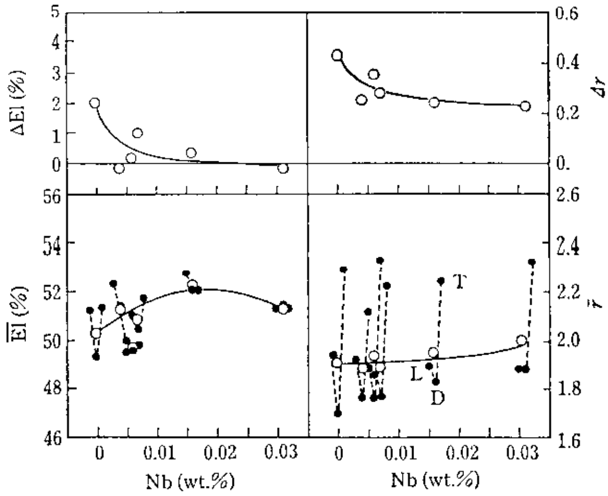

Fig. 5 Effect of Nb addition to Ti-based sheet steels on planar anisotropy of elongation and r-value (steels T and TN1 to TN5, annealing temperature 850°C)

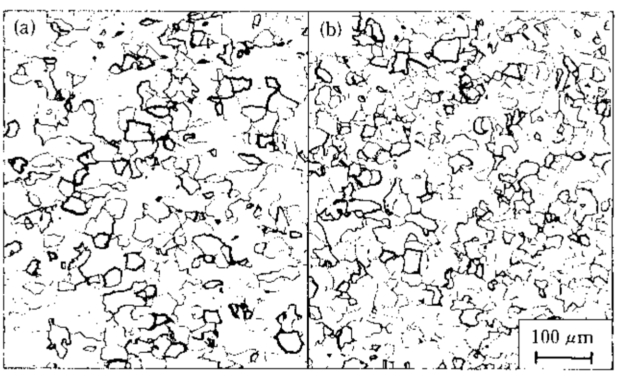

Photo 2 Optical micrographs of (a) Ti-bearing (steel T) and (b) Ti-Nb co-added (steel TN3) hot bands

basic Ti-stabilized steel. A small amount of Nb (about 0.010%) co-addition provides distinct decrement of the planar anisotropy of El and r-value, thereby maintaining high average values of El and r.

Hot band microstructures (cross section in the rolling direction) of Ti steel (steel T) and Ti-Nb steel (steel TN3) are shown in **Photo 2**. A small amount of Nb co-addition results in fine microstructure in the hot band.

It is well known that (1) texture, (2) solute C and N contents, and (3) grain size of a hot band affect the planar anisotropy of mechanical properties of cold-rolled and annealed sheet steels.[15] As for the item (1), virtually no difference was detected in hot band texture within the range of Nb addition in this experiment. The item (2) is also negligible since the enough amount of Ti is added to stabilize C and N in the hot band. In fact, the aging indices (AI) of hot bands of steels T and TN3 were zero. On the other hand, development of

Table 2 Comparison of recrystallization temperature and mechanical properties between Ti, Nb, and Ti-Nb steels

	Ti	Nb	Ti-Nb (Ti>Nb)
Recrystallization temperature	Low	High	Low
Mechanical properties	Excellent	Good	Excellent
Planar anisotropy of mechanical properties	Large	Small	Small

recrystallization texture is greatly affected by the grain size of hot bands. At the nucleation process of recrystallized grains from cold-deformed structure, {111} and {110} oriented grains are preferentially developed at the grain boundaries and inside the grains of mother hot band, respectively.[16] Thus the hot band with fine grain size suppresses the development of {110} recrystallization texture because of an increase in boundary area per unit volume. The texture {110} and its neighboring orientations decrease El and r-value of the diagonal direction (D) with respect to the rolling direction, resulting in increasing the planar anisotropy of mechanical properties[15]. Thus Nb co-addition to Ti-stabilized steel provides a fine grain hot band, with the result of retarding the development of {110} and neighboring orientations which increase the planar anisotropy of mechanical properties. It is inferred that pinning effect of solute Nb against γ grain growth during hot rolling leads to fine microstructure of hot bands.[17]

Based on the results obtained in the laboratory, effect of Ti and Nb addition on the mechanical properties of extra-low C steel (about 20 ppm C) is summarized in **Table 2**. Ti-bearing steel exhibits lower recrystallization temperature than in Nb-bearing steel, resulting in better mechanical properties when compared under the same processing conditions. However, a small amount of Nb addition to Ti-stabilized steel is effective in reducing the planar anisotropy of mechanical properties. Thus addition of both Ti and Nb ensures well balanced properties.

4 Experimental Results in Mill

Based on the above mentioned results obtained in the laboratory, the effect of processing conditions in the mill on the mechanical properties of Ti and Nb co-added extra-low C steels has been investigated.

Figure 6 shows the effect of C content on the total elongation (El) of continuously annealed (soaking temperature: 850°C) Ti-Nb co-added cold-rolled sheet steels (Ti = 0.03%, Nb = 0.008%). Reducing the C content, especially below 20 ppm, provides very high El. This is attributable to a decrease in the volume fraction of car-

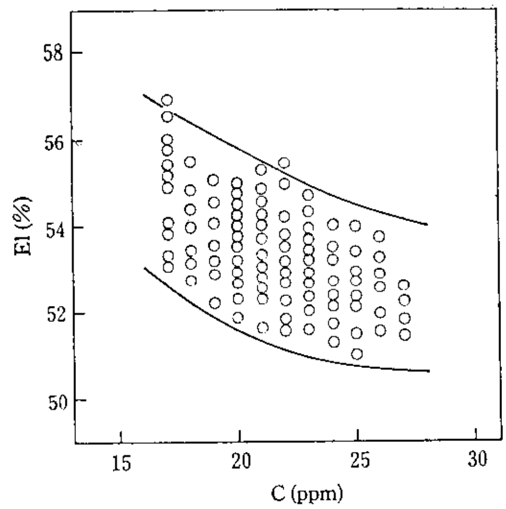

Fig. 6 Effect of content on elongation of continuously annealed sheet steels (Ti 0.028 wt.%, Nb 0.008 wt.%, annealing temperature 850°C)

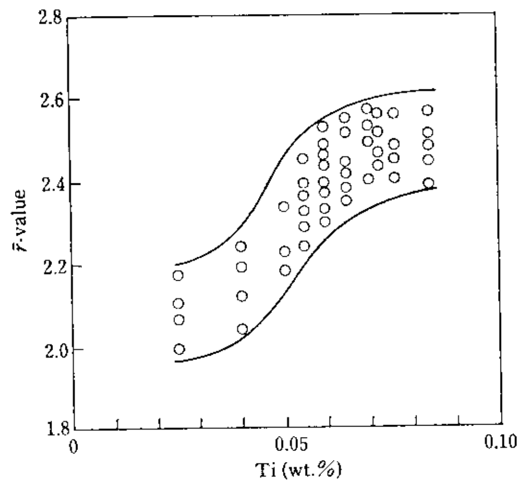

Fig. 7 Effect of Ti content on r-value of continuously annealed sheet steels (C + N 0.0045 wt.%, Nb 0.003 wt.%, annealing temperature 880°C)

bides by a reduction in the C content, resulting in an improved grain growth during recrystallization annealing.

Figure 7 demonstrates the effect of Ti content on the r-value of 20~25 ppm C-0.003% Nb sheet steels. The continuous annealing temperature was 880°C. Ti addition up to 0.06% is distinctly effective in improving r-value, while the improvement is saturated at the amount of Ti addition higher than 0.06%. In this case, Ti content is more excessive (by approximately 0.04%) than that needed for stabilizing C, N, and S. Therefore, the following reasons are considered attributable to the improvement of r-value in high Ti content steels: (1) Stabilizing C and N in a more assured manner, (2) coarsening of precipitates nucleated at higher temperatures, (3) change of recrystallization texture due to solute Ti. Among them, (2) and (3) are more accountable for explaining the results described in Fig. 7.

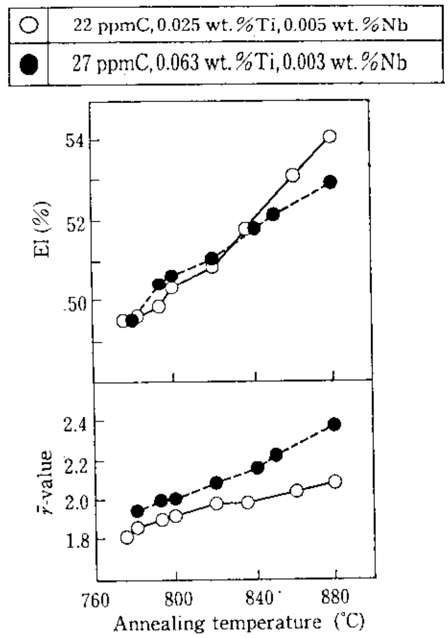

Fig. 8 Effect of continuous annealing temperature on elongation and r-value

Figure 8 shows the r-value and El of two types of Ti-Nb co-added steels plotted against the soaking temperature of continuous annealing. Both properties were improved by an increase in soaking temperature up to 880°C. Remarkably high El and r-value were obtained in the low C-low Ti steel and the high Ti steel, respectively.

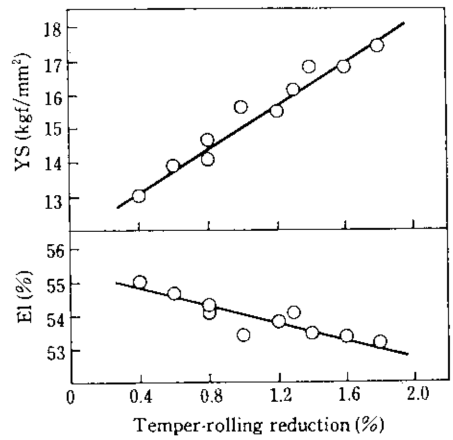

Fig. 9 Effect of temper-rolling reduction on eleongation and yield strength of continuously annealed sheet steels (C 0.0025 wt.%, Ti 0.030 wt.%, Nb 0.006 wt.%, annealing temperature 850°C)

Figure 9 shows the influence of temper-rolling reduc-

Table 3 Manufacturing conditions and mechanical properties of newly developed EDDQ steels

Steel Type (Thickness)	Chemical Composition C (ppm)	Ti (wt.%)	Nb (wt.%)	Annealing temp. (°C)	Temper-R. reduction (%)	YS (kgf/mm²)	TS (kgf/mm²)	El (%)	$\bar{r}$	Δr
High ductility (0.7 mm)	18	0.026	0.008	850	0.5	13.2	27.8	55	2.15	0.43
High drawability (1.2 mm)	23	0.067	0.003	880	0.5	14.2	27.5	55	2.50	0.36
Conventional EDDQ (0.7 mm)						14.9	29.2	52	2.10	0.56

tion on YS and El of a Ti-Nb co-added steel. Temper rolling with about 0.5% reduction, which is lower than the conventional temper-rolling reduction, provides high ductility.

Table 3 summarizes the processing conditions and the mechanical properties of newly developed sheet steels. The feature of steel chemistry is very low C (below 20 ppm) for the high ductility type and high Ti content for the high r-value type. Both types of steels are continuously annealed above 850°C and temper-rolled lightly at 0.5% reduction. The high ductility sheet steel and the high r-value sheet steel have been applied to drawing-stretching complex forming such as for an integrated large panel, and deep-drawing such as for a complex shape oil pan, respectively.

5 Conclusions

Effects of steel chemistry and processing conditions on the mechanical properties of Ti- and Nb-added extra-low C steels have been investigated in the laboratory and the mill in order to develop extra-deep drawing (EDDQ) cold-rolled sheet steels for integrated automotive parts.

(1) When enough amounts of Ti or Nb to stabilize C were added to an extra-low C steel (20 ppm C), Ti-added steel provided better El and r-value than in Nb-added steel, expecially in lower annealing temperature below 800°C.

(2) The grain growth at recrystallization in Ti-bearing steel was faster than in Nb-bearing steel, since Ti-bearing steel contained a lesser amount of fine precipitates with diameters below 50 nm in the hot band than in Nb-bearing steel.

(3) A small amount of Nb addition (approximately 0.01%) to Ti-stabilized steel is effective in decreasing the planar anisotropy of mechanical properties, thereby keeping high El and high r-value.

(4) High temperature continuous annealing (850–880°C) and low reduction temper-rolling (about 0.5%) using Ti-Nb co-added steels have provided new products of their mechanical property superior to that of the conventional EDDQ steel.

(5) These products have been applied to complex and large automotive parts such as side outer panels and oil pans.

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