On the Combination of Tensile Strength and Ductility in High Strength Cold Rolled Sheet Steels and the Metallurgical Factors Responsible Therefor*

By Hiroaki MASUI** and Hiroshi TAKECHI***

Synopsis
The strengthening mechanisms in high strength cold rolled sheet steels for uses as the inner and outer steel panels of safety automobiles have been examined with regard to the press formability, namely in terms of the combination of strength and ductility (n-value and total elongation).

In view of the manufacturing process, which includes the recrystallization anneal as a necessary step, only three hardening mechanisms, the substitutional hardening, the precipitation hardening, and the grain boundary (grain refinement) hardening, are possible.

The results obtained are as follows:
1. The combination of the strength and ductility can be ranked in a worsening order as:
   - Substitutional solution hardening > Grain boundary hardening > Precipitation hardening
2. In the substitutional solution hardening type, collinear arrays of dislocation are dominant, and the work hardening still continues on at a large tensile strain (the continually hardening type).
3. In the grain boundary hardening type, the deformation behavior is essentially the same as in "pure" iron both in the cell substructure formation and in the type of strain hardening (the ordinary hardening type).
4. In the precipitation hardening type, most of the dislocations interact with the fine precipitates uniformly distributed in the matrix, and the strain increases forming dislocation loops at an early stage of tensile strain, whereupon the work hardening is large at early stage of straining but soon saturated without forming clear cell substructures at late stage (the early stage hardening type).
5. The poor combination of strength and ductility of the precipitation hardened steels can be improved up to the level of the grain boundary hardening type by causing the precipitating particles to coalesce and the distributed heterogeneously, e.g., by austenitizing before cold rolling, whereby changing the deformation behavior over to that of the grain boundary hardening type.

I. Introduction
To improve the safety of riders, high strength cold rolled sheet steels are being introduced to automobiles. For example, in recent ESV's (Experimental Safety Vehicles) cold rolled sheet steels which have both the tensile strength (TS) of about 40 to 60 kg/mm² and the good press formability have been used for several inner panel parts (e.g., center pillar), outer panel parts, and reinforcements instead of the usual mild steels of about 30 kg/mm² TS. It will be appreciated that, in this application, the press formability is as important as the strength, and, in this case, it calls for, besides good drawability, good stretchability.

The relation between stretchability and n-value is well known,[1,2] and the relation between stretchability and total elongation has been found by Okamoto and Hayashi[3] in terms of the bulge height, which was, in a sense, a combination of n-value and local elongation.

Therefore, for the high strength cold rolled sheet steels as structural parts of safety automobiles, both the strength and the ductility (especially the n-value or the total elongation, in that the larger the values, the better the press formability) need be excellent. In this paper, this will be expressed as the "good combination of strength and ductility".

Because those cold rolled sheet steels must go through the recrystallization annealing in their manufacture, however, of several metallurgical means of attaining good combinations of strength and ductility, only the substitutional solid solution hardening and the grain boundary (grain refinement) hardening are possible, while the interstitial solid solution hardening will scarcely be effective. Further, by the same reason, the precipitates will be small and incoherent with the ferrite matrix.

II. Steels and Experimental Procedures
A part of the steels was melted in a 100 kg Balzer vacuum melting pot using electrolytic iron (Samples C, D and E), while the other in a 1 t electric furnace (Samples A and B). The ingots were 100 and 250 kg, respectively.

The samples were classified according to the intended hardening mechanism into the precipitation hardening type, the grain boundary hardening type and the substitutional solution hardening type as shown in Table 1. Because the grain boundary hardening is common to all, those steels in which neither effective precipitation hardening nor effective solution hardening are to be expected are put in the grain boundary hardening type.

The ingots were heated for 1 hr at 1250°C and hot rolled to 20 mm (100 kg ingots) or 40 mm (250 kg ingots) with the finishing temperature of 900°C. They were heated again at 1250°C for 1 hr and hot rolled to 2.7 mm thickness with the finishing temperature of 900°C followed by air cooling. The hot rolled steel sheets were pickled in a hydrochloric acid, then cold rolled to 0.8 mm with a reduction of 70%. The specimens were annealed with a heating rate of 100°C/hr to 700°C and kept for 4 hr followed by

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furnace cooling. This procedure will be called the \( N \) treatment (Table 1).

With the Sample B, however, the hot rolled sheets were reheated at 950°C for 1 hr before the cold rolling and annealing. This will be called the \( P \) treatment (Table 2).

Two kinds of tensile test specimens were used. One, to determine mechanical properties such as tensile strength, \( n \)-value (at 10 or 20\% elongation) and total elongation, JIS No. 5 test pieces were used. Specimens were machined from annealed sheets generally parallel to the rolling direction, but for some samples, specimens along 45° (diagonal) direction and 90° (cross) direction were also taken. Two specimens for observation in a transmission electron microscope (100 kV) were prepared from the tensile test pieces having a 30 mm wide parallel part as deformed to 6 or 15\% elongation (some to 2\%).

All the test was conducted in a 5 t Instron machine at room temperature and with a cross head velocity of 10 mm/min.

### III. Experimental Results

#### 1. Combination of Tensile Strength and Ductility

It has been shown that the relationship between tensile strength and total elongation in commercial cold rolled steel sheets (thickness: 0.8 mm) is as shown in Fig. 1. A similar relationship was found between the strength and the \( n \)-value.\(^5\)

With the steels of this investigation, as annealed at 700°C for 4 hr (Photo. 1), the relation between tensile strength and total elongation was as shown in Fig. 2 against the curve of Fig. 1, which may be taken as the standard. It will be observed in the figure that the substitutional solution hardening type steels lie above the standard line, the grain boundary hardening type steels right on the line, and the precipitation hardening type steels below the line. Therefore, it may be concluded that the combination of tensile strength and total elongation is worse in the order of substitutional solution hardening type > grain boundary hardening type > precipitation hardening type. This was the same for the combination of tensile strength and \( n \)-value as shown in Fig. 3.

In the true stress–true strain curves (stress–strain curves, hereafter) shown in Fig. 4, differences in the strain hardening behavior among those three types are recognized. That is to say, as compared with the grain boundary hardening type Sample C, the precipitation hardening type Samples A and B harden more in the early stage of straining, while the substitutional solid solution type Samples D and E harden continuously even in a later stage.

In order to emphasize these characteristics more clearly, the flow stresses of Sample C were subtracted from respective stresses of A, B, D and E in Fig. 4 and shown in Fig. 5. Though, strictly speaking, correct comparison of work hardening behaviour among these samples cannot be made because grain sizes of the annealed samples are not the same (Photo. 1), the fact that the precipitation hardening type is the early stage

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</thead>
<tbody>
<tr>
<td>A</td>
<td>0.031</td>
<td>0.044</td>
<td>0.30</td>
<td>0.003</td>
<td>0.013</td>
<td>0.0003</td>
<td>0.0087</td>
<td>0.023</td>
<td>0.179</td>
<td>Precipitation hardening</td>
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<tr>
<td>B</td>
<td>0.050</td>
<td>0.050</td>
<td>0.32</td>
<td>0.005</td>
<td>0.012</td>
<td>0.0007</td>
<td>0.0096</td>
<td>0.055</td>
<td>0.396</td>
<td>Grain boundary hardening</td>
</tr>
<tr>
<td>C</td>
<td>0.005</td>
<td>0.009</td>
<td>0.17</td>
<td>0.009</td>
<td>0.017</td>
<td>0.0009</td>
<td>0.0036</td>
<td>0.003</td>
<td>0.096</td>
<td>Substitutional solution hardening</td>
</tr>
<tr>
<td>D</td>
<td>0.006</td>
<td>0.034</td>
<td>2.80</td>
<td>0.005</td>
<td>0.009</td>
<td>0.0005</td>
<td>0.0036</td>
<td>0.002</td>
<td>0.062</td>
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<tr>
<td>E</td>
<td>0.004</td>
<td>0.030</td>
<td>0.29</td>
<td>0.008</td>
<td>0.014</td>
<td>0.0008</td>
<td>0.0024</td>
<td>0.003</td>
<td>0.109</td>
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### Table 2. Treatment of samples

(The \( P \) treatment has been done only with Sample B)

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<th>Treatment</th>
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<tr>
<td>( N )</td>
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<tr>
<td>( 1250°C \times 1 \text{ hr} \rightarrow \text{hot rolling (900°C finishing)} \rightarrow 70% \text{ cold rolling} \rightarrow 700°C \times 4 \text{ hr annealing}</td>
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<tr>
<td>( P )</td>
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<tr>
<td>( 1250°C \times 1 \text{ hr} \rightarrow \text{hot rolling (900°C finishing)} \rightarrow 950°C \times 1 \text{ hr} \rightarrow 70% \text{ cold rolling} \rightarrow 700°C \times 4 \text{ hr annealing}</td>
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(a) Sample A (0.03% C + 0.18% Ti)
(b) Sample B (0.05% C + 0.40% Ti)
(c) Sample C (Trace C−Ti-killed)
(d) Sample D (Trace C−Ti-killed + 2.8% Mn)
(e) Sample E (Trace C−Ti-killed + 0.8% Si)

Photo. 1. Microstructure of specimens as annealed at 700°C for 4 hr (×100) (11/20)

Fig. 2. The combination of tensile strength and total elongation of the three different hardening type steels with respect to those of commercial steels

Fig. 3. The combinations of tensile strength and n-value of the three different hardening type steels

Fig. 4. The true stress-true strain curves of the three different hardening type steels

Fig. 5. The differences in the flow stress between Samples A, B, D, E and Sample C (grain boundary hardening type)

It is to be noted that these characteristics are reflected in the order of superiority with regard to combination of tensile strength and n-value in Fig. 3 and...
also to the combination of tensile strength and total elongation Fig. 2.

2. Metallurgical Mechanisms behind the Combination of 
   Strength and Ductility

As mentioned above, the order of superiority in the 
combination of strength and ductility is closely related 
with the work hardening behavior. In this section, 
metallurgical mechanisms responsible for this relation-
ship are examined.

Observations in transmission electron microscopy of 
the specimens strained in tension were as follows.

1. The Grain Boundary Hardening Type Sample C

The structural changes in this sample were essen-
tially the same as often reported for various “pure” 
irons. Namely, at 6% elongation, dislocations were 
entangled as shown in Photo. 2, then at 15% elonga-
tion work cell substructure was formed (Photo. 3).

2. The Substitutional Solid Solution Hardening Type 
   Sample D

Different from the case of grain boundary hardening 
type, at 6% elongation, collinear arrays of dislocations 
were found predominantly (Photo. 4). Although the 
work cell substructure was formed at 15%, the collinear 
dislocation arrays still remained, and on the whole, 
the cells were not so clearly formed (Photo. 5) as in 
Sample C (Photo. 3).

3. The Precipitation Hardening Type Sample B

In the transmission electron micrograph of the an-
ealed state (Photo. 6), it is to be recognized that most 
of the small TiC precipitates, about 10^2 Å in diameter, 
were incoherent with matrix. At 2% elongation, dislo-
cations started already to tangle around the small 
precipitates (Photo. 7), and at 6% the tangling was 
quite intensive, where some dislocations had formed 
loops around precipitates (Photo. 8). At 15%, how-
ever, as shown in Photo. 9, the microstructure was not 
so much different as compared with that at 6%, but 
there was a distinctive feature that only a few work 
cell substructures were formed. That the work hard-
ening had become saturated in so early a stage of de-
formation as 10% elongation is shown also by the

![Photo 2](image2)

1 μ

![Photo 3](image3)

1 μ

![Photo 4](image4)

1 μ

![Photo 5](image5)

1 μ

![Photo 6](image6)

1 μ

![Photo 7](image7)

1 μ

![Photo 8](image8)

1 μ

![Photo 9](image9)

1 μ
stress–strain curve in Fig. 4 or 5.

Another feature of this sample was that, although precipitation hardening type alloys usually have unrecrystallized regions as annealed, no unrecrystallized regions were found at least within the fields examined. This may have certain bearing on the particular deformation behavior this steel showed.

3. Change-over from the Precipitation Hardening Type to Grain Boundary Hardening Type

When Sample B was subjected to the P treatment, the steel came up with a finer grain size (Photo. 10, to be compared with Photo. 1(b)) and greatly coalesed TiC (now of the order of 10³Å in diameter) aligned along the austenite grain boundaries (Photo. 11, to be compared with Photo. 6).

The combination of tensile strength and ductility of Sample B thus treated (now renamed as B') is shown in Figs. 6 and 7 in terms of n-value and total elongation, respectively.
In Fig. 8, the stress–strain curve of B' is compared with those of other samples, particularly with that of Sample B. It will be seen that the work hardening at 15% elongation of B' is somewhat larger than that of B, that the deformation behavior is more like that of the grain boundary hardening type Sample C.

These observations were substantiated by the transmission electron microscopy as shown in Photos. 11 to 13. Namely, in a comparison of B' against B, at 2% elongation, dislocations in B' are already in an appreciable number and entrapped by large precipitates, whereas no such phenomena are seen in Sample B (Photo. 11 vs. Photo. 7). Here, it is reasonable to suppose that, because these particles can hardly be coherent with matrix, those dislocations had been generated elsewhere. At 6%, the dislocations are being entangled each other in B' to start the formation of work cell substructure (Photo. 12 vs. Photo. 8). At 15%, the work cell substructures have been developed well and clearly in B', whereas they are unclear in Sample B (Photo. 13 vs. Photo. 9). It will be seen that those microstructures of B' are thus closer to those of the grain boundary hardening type Sample C (Photos. 2 and 3) than to the original B, the precipitation hardening type.

As all the steels used in this investigation are titanium-killed, such interstitial solution elements as carbon and nitrogen are fully stabilized, as may be seen in the stress-strain curves in Fig. 4. Therefore, whatever the hardening type, the effects of solute carbon and nitrogen, which act additionally, can be neglected in this study.

First, in the grain boundary hardening type, (Sample C, trace carbon, titanium-killed), dislocations start to tangle early at 6% elongation. It is thought that this is a tangle of dislocations that contains many edge dislocation components, and that its sources are the drag of edge dislocations by screw dislocations or edge dislocation dipoles. At 15% elongation the work cell substructures are formed, and this may be caused by linking of the tangles. The work cell substructures are obstacles to dislocation movement in such a way as the smaller the average distance that dislocations glide, the larger the stress to give rise to a given quantity of strain. Therefore, it is evident that the cell substructures contribute to work hardening and can explain the observation that the work hardening is operating still in the neighborhood of 15% elongation. In this case of depending on only the work cell substructure, however, the work hardening at 15% elongation cannot be expected to be as large as that of substitutional solution hardening type, because in the former, dislocations can escape by cross slipping whereas in the latter cross slipping of dislocation is restrained.
Next, in the substitutional solution hardening type, limitation of slip system by the substitutional solution elements is added, and at 6% elongation, as has been often said,\(^\text{10,11}\) collinear distribution of dislocation is observed everywhere in the microstructure. Besides those collinearly distributed dislocations, edge dislocation dipoles partially forming dislocation tangles are observed. This may be a cause of work cell substructure observed at 15% elongation. Here, it is worth noting in the microstructure of 15% elongation (Photo. 5) that, typically developed work cell substructures are not observed so much as in the case of the grain boundary hardening type, while many collinear arrays of dislocations that are probably screw\(^\text{8,10,11}\) are present on the whole.

This means that, where in the grain boundary hardening type the increase of work hardening rate due only to work cell substructures can be expected, in the substitutional solution hardening type, the limitation of slip system and the restraining of cross slip by the substitutional solid solution elements will enhance the work hardening in the neighborhood of 15% elongation. This phenomenon has been observed not only in Sample D (3% manganese) but also in a phosphorus-bearing \((0.1\% \text{ phosphorus})\) aluminum killed high strength cold rolled sheet steel. Namely, as shown in Photo. 14, when strained, great many collinear screw dislocations are generated due undoubtedly to the effect of phosphorus which has an enormous solution hardenability. Therefore, the combination of tensile strength and total elongation of the phosphorus-bearing sample, a typical substitutional solution hardening type steel, is excellent as shown in Fig. 9.

Lastly, in the precipitation hardening type, most of the small precipitates are incoherent with matrix as shown in Photo. 6, because the steel is necessarily subjected to the recrystallized annealing for a long time. When such a steel undergoes tensile deformation, the small precipitates entrap many dislocations and restrain them from moving freely even at 2% elongation. Moreover, at 6% elongation, may dislocations form loops around the small precipitates, where a kind of Fisher-Hart-Pry mechanism would operate locally,\(^\text{14}\) This corresponds to the fact that the work hardening rate in an early stage of straining, till about 6 to 8% elongation, is very large as shown in Fig. 5. Meanwhile, a phenomenon that straining progresses while forming dislocation loops by the cross slip mechanism is seen. As these mechanisms are observed to continually operate even at 15% elongation, and besides there is no gross heterogenization of dislocation distribution to lead to formation of the work cell substructure, the work hardening tends to saturate easily, which is a typical early stage work hardening type behavior, this, then can be considered as the main cause to deteriorate the combination of strength and ductility.

On the other hand, if the P treatment is given to the same sample to make the precipitates coalesce and enlarge, dislocations are accumulated preferentially in the neighborhood of every large precipitate particle in deformation. These heterogeneously accumulated dislocations form tangles, and linking each other, form the work cell substructure. In this case, the obstacle to restrain the movement of dislocations is the work cell substructure only, and the work hardening type changes from the precipitation hardening type, which is the early stage work hardening, over to the grain boundary hardening type, whereby the combination of strength and ductility is improved as shown in Figs. 6 and 7. From this experience, it is to be suspected that, besides these small incoherent precipitates, such small coherent particles as Fe\(_3\)C or Fe\(_4\)N that precipitate in the low temperature quench-aged steels

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**Photo. 14.** Transmission electron micrograph of an aluminum-killed steel containing 0.1% phosphorus \((\times 40000) (1/2)\)

**Fig. 9.** The combination of tensile strength and total elongation of an aluminum-killed steel containing phosphorus

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may deteriorate the combination of strength and ductility by the early stage hardening mechanism. A study on this is being planned.

IV. Conclusion

To give a guide in developing high strength cold rolled sheet steels for uses in safety automobile, a study has been carried out to find what hardening type is the best for the combination of strength and ductility (to be evaluated in terms of the $n$-value and the total elongation, factors important in press forming) and to investigate the metallurgical mechanism underlying each type.

The following results are obtained:

(1) From the viewpoint of hardening type, better combination of the strength and ductility is resulted by

Substitutional solution hardening $>$ Grain boundary hardening $>$ Precipitation hardening.

This ranking corresponds well to the three work hardening behaviors shown in the true stress–true strain curve of the continually hardening type, the ordinary hardening type, and the early stage hardening type.

(2) In the substitutional solution hardening type steel, which is the continually hardening type, the collinear array is the dominant dislocation structure.

(3) In the grain boundary hardening type steel, more cell substructures, which give rise to work hardening to a certain extent, and fewer collinear arrays of dislocations are formed than in the substitutional solution hardening type.

(4) In the precipitation hardening type steel, which is the early stage hardening type, most of the dislocations are accumulated in the neighborhood of uniformly distributed fine precipitates forming loops around them, and the strain increases rapidly in an early stage of tensile straining. Thus, work hardening is fairly large at early stage but is soon saturated without forming clear cell substructions in the late stage.

(5) The poor combination of strength and ductility of the precipitation hardening type steels can be improved up to the level of the grain boundary hardening type by causing the precipitating particles to coalesce and be distributed heterogeneously, e.g., by austenizing before cold rolling, whereby changing the deformation behavior over to that of the grain boundary hardening type.

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