A New Type of High Strength Steel for Exposed Panels —High-Strength Steel with Excellent Formability, Superior Surface Precision after Press Forming, and Uniform Surface Appearance—[†]

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Abstract:

SFGHITEN® (super fine grain, high strength steel sheet), which was developed recently by JFE Steel, is strengthened by fine Nb(C, N) precipitates and grain refinement, giving it excellent press formability suitable for automotive exposed panels. The Nb(C, N) precipitate shows a unique distribution which had not been observed previously and tends to form precipitate-depleted zones, called precipitate free zones (PFZ), in the vicinity of grain boundaries. These PFZs lower yield strength in spite of the small size of the grains. In comparison with conventional deep-drawing sheets, SFGHITEN has a high r-value, combined with excellent resistance to secondary embrittlement imparted by B addition. Try-out pressing for an automotive front-fender model was successfully conducted and demonstrated that the new material has excellent formability, displaying a wider formable range than the conventional steel.

1. Introduction

Introduction of high-strength steel sheets which meet weight reduction needs in auto body panels is progressing rapidly. Among these, strengthening by adding solid solution strengthening elements such as Si, Mn, and others, based on an IF (interstitial free) steel chemical composition, is used to achieve high tensile strength in exposed panels in view of the high formability and surface property requirements of this application.[1] However, it is difficult to secure formability equal to that of mild steel because addition of solid solution strengthening elements deteriorates deep-drawability (r-value), and the work hardening index (n-value) decreases as yield strength increases. Moreover, when the strength of the parent phase is increased, grain boundary strength shows a relative decrease, raising the problems of grain boundary embrittlement, which can be observed after forming, and deterioration of coated surface quality caused by addition of the strengthening element Si, which is particularly a problem in galvannealed (GA) products.[2,3]

To solve these problems, JFE Steel developed a new high-strength sheet with excellent formability and surface quality, in which grain boundary strength is increased and secondary work embrittlement is suppressed by refining the grain size in comparison with conventional steels, and excellent deep drawability and a low yield ratio are also successfully realized by applying a unique fine precipitate dispersion hardening technique which does not use solid solution elements that deteriorate surface properties. The newly developed steel has been commercialized by JFE Steel as SFGHITEN® (super fine grain, high strength steel sheet) in the TS340, TS390, and TS440 MPa grades, and is available in the form of cold-rolled steel sheets and GA steel sheets.[4–7]

This paper describes the metallurgical background which produced the unique mechanical properties of

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SFGHITEN and the advantages of this product when applied in automotive exposed panels.

2. Phenomena Observed in SFGHITEN and Their Basic Mechanism

In achieving high strength based on IF steel, use of the solid solution hardening mechanism, in which strengthening elements such as Si, Mn, P, and others are added in large quantity, is a concept with a long history. However, because these strengthening elements, and particularly Si, deteriorate deep drawability and the quality of the coated surface, products manufactured by this technique cannot be used in exposed panels in which complex forming is required. In contrast, as the concept of the present development, high strength is achieved by precipitate dispersion hardening using fine Nb(C, N), without adding Si as a strengthening element, in combination with ferrite grain refinement, while the deep drawing property is secured by improving the cold-rolling recrystallization texture by grain refinement in the hot-rolled sheet and encouraging achievement of the {111} texture, which is advantageous for deep drawability. As a result, high strength and improved deep drawability are realized simultaneously. An IF steel composition design was adopted, with Nb added for grain refinement. The C content was set at more than double that in ordinary ultra-low carbon steel (30 ppm or under) in order to strengthen grain refinement, and Nb was added in a quantity sufficient to fix the C. In the following, these basic phenomena and their mechanism are clarified based on experimental findings.

Table 1 shows the chemical compositions of the laboratory melts used in the experiments. Steel A is a steel in which Nb was added to ordinary ultra-low carbon in a quantity exceeding an equivalent ratio of 1, and Mn and P were added as solid solution strengthening elements. Steel B is a steel in which the absolute amounts of Nb and C were increased while maintaining basically the same Nb/C ratio as in Steel A.

After vacuum melting 50 kg ingots of these steels, the samples were hot-rolled to slabs with a thickness 30 mm. After solution treatment at 1 200°C for 3.6 ks, finishing hot rolling was performed to a sheet thickness of 2.8 mm at a finishing temperature of 900°C, followed by treatment equivalent to coiling at 640°C × 3.6 ks. The hot bands obtained in this manner were pickled and cold-rolled at a reduction ratio of 80% to produce cold-rolled strips with a thickness of 0.56 mm. In annealing, heat treatment was performed for 50 s at 600–870°C using a salt bath. These annealed strips were then temper-rolled at a reduction ratio of 0.7% and used in various tests.

Table 1 Chemical composition of steels A and B

(mass%)

Steel	C	Si	Mn	P	N	Nb	Nb/C
A	0.002 0	0.02	0.66	0.043	0.002 9	0.022	1.42
B	0.005 2	0.01	0.62	0.040	0.003 2	0.068	1.69

Recrystallization behavior in the hot rolling stage was evaluated based on the softening ratio obtained from changes in the flow stress on the stress-strain curve when 2-step compression was performed at an inter-pass time of 0.1 s to 500 s at 900°C using samples with a diameter of 8 mm and height of 12 mm by a forming Formaster. Recrystallization behavior during annealing after cold rolling was evaluated by measuring the Vickers hardness at the sheet center-of-thickness position at each annealing temperature.

A tensile test was performed with JIS No. 5 test pieces taken in the normal direction (90° relative to the rolling direction). The Lankford value (mean r-value) was calculated by the following equation from the respective r-values obtained by 15% tensile deformation of test pieces taken from the 0°, 45°, and 90° directions relative to the rolling direction.

$$\bar{r}\text{-value} = \frac{r_{0°} + 2r_{45°} + r_{90°}}{4}$$

Texture was evaluated by X-ray diffraction after chemically polishing the samples to the sheet center-of-thickness. The crystallographic microstructure was observed using optical micrographs, SEM, and TEM.

Figure 1 shows the effect of C and Nb on recrystallization softening behavior at 900°C during annealing in the γ (austenite) region. The time to a 100% softening ratio with steel A and steel B, in other words, the time to complete recrystallization of γ, differs greatly, being approximately 30 s in steel A but 500 s in steel B. This indicates that the high contents of C and Nb in steel A are effective suppressing recrystallization in γ.

Photo 1 shows the ferrite microstructure of the hot bands. The structure is finer in steel B than in steel A. This can be understood as showing that the γ recrystallization suppression effect shown in Fig. 1 has occurred, and consequently, the ferrite grains are also small after transformation.

Photo 2 shows the ferrite microstructure of samples annealed at 850°C. As with the recrystallization behavior in hot rolling, the grain size after annealing is also small in steel B.

Figure 2 shows the difference in the TS (ΔTS), yield ratio, and mean r-value of samples annealed at 830, 850, and 870°C. In comparison with steel A, the tensile strength of steel B is approximately 30 MPa higher in all temperature regions. This is attributed to the small grain

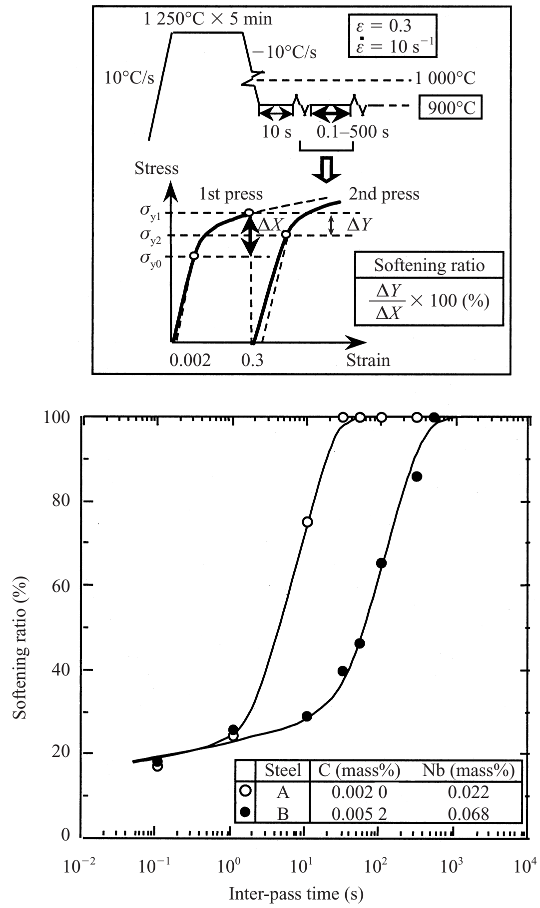

Fig. 1 Change in softening ratio with inter-pass time during hot compression at 900°C for steels A and B

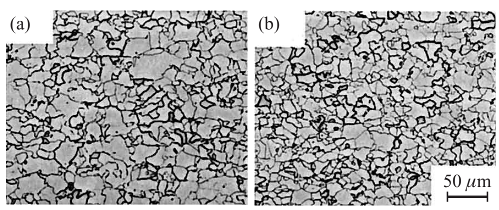

Photo 1 Optical micrographs on the cross-sections of the hot-bands of steels A (a) and B (b)

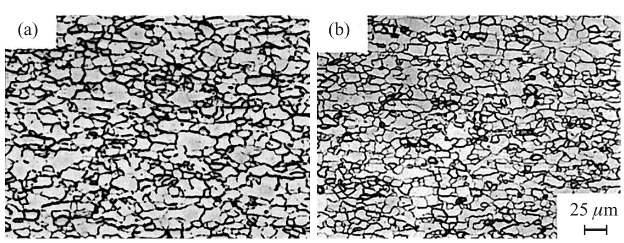

Photo 2 Optical micrographs of steels A (a) and B (b) on the cross-sections of the samples annealed at 850°C

size and presence of Nb precipitates in steel B. The yield ratio of steel B is approximately 10% lower than that of steel A. From the viewpoint of grain size and precipitates, the yield ratio should show the opposite tendency, being higher in steel B. It is therefore thought that a different mechanism causes this low YR phenomenon.

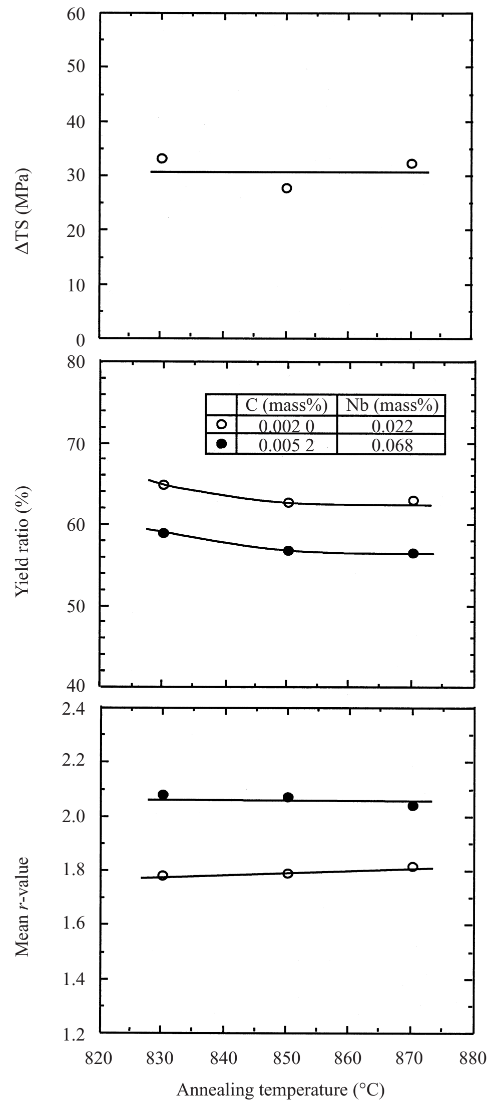

Fig. 2 Effect of annealing temperature on the mechanical properties of steels A and B

The mean r-value of steel B is more than 2.0 at all annealing temperatures, and is approximately 0.2 higher than in steel A. **Figure 3** shows the integrated X-ray diffraction strength ratio of grains with the {111} and {100} orientations in steel A and steel B with recrystallization during annealing. In the as-cold-rolled condition, there is no large difference in the intensities of grains with the {111} and {100} orientations, but with recrystallization, steel B shows remarkable achievement of the {111} plane, and as a result, this steel is thought to show a high r-value. This remarkable achievement of the {111} orientation accompanying recrystallization is caused by the increase in the surface area of the grain boundaries, which serve as sites for preferential nucleation of grains with the {111} recrystallization orientation, resulting from grain refinement in the hot-rolled sheet stage. **Figure 4** shows the relationship between the grain size and mean r-values of steels A and B in comparison with other steel grades. In general, the r-value of steel sheets increases accompanying grain growth as the annealing

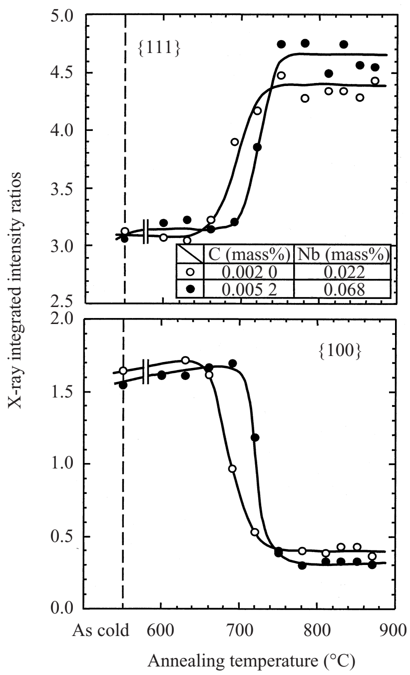

Fig. 3 Change in normalized X-ray integrated intensity ratio with annealing temperature for steels A and B

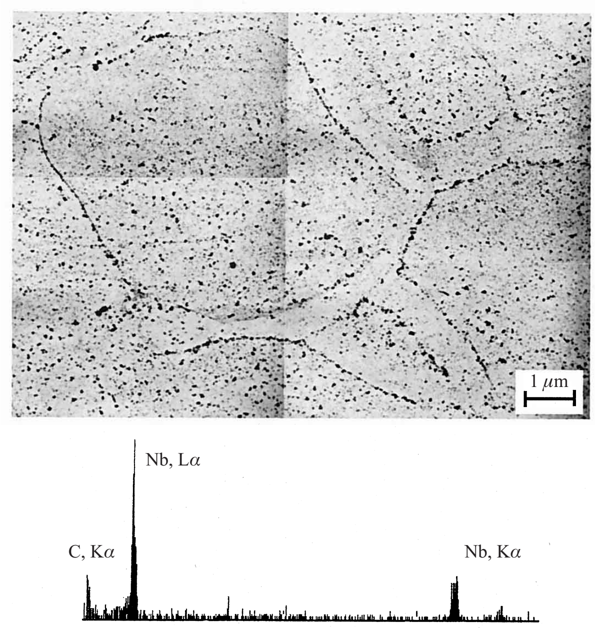

Photo 3 TEM replica image and EDS spectra of the precipitates observed in the specimen annealed at 850°C of steel B

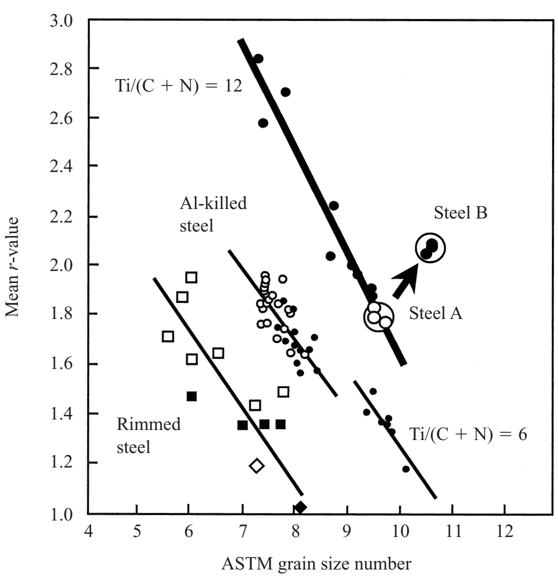

Fig. 4 Relation between mean r-values and ASTM grain size number for steels A and B with comparison to conventional steels

temperatures increases, and high r-values exceeding 2.5 can be obtained by grain coarsening, even with IF steels, as shown in this figure. However, this is of little practical value because the surface defect called "orange peel" occurs when sheets with coarse-grained structures are press-formed. From the viewpoint of suitability for practical applications, the relationship between the grain size and r-value of steel B, which consists of fine grains, is greatly improved from that of the conventional IF steels.

This fine microstructure and the existence of Nb precipitates are distinctive features of steel B. **Photo 3** shows a TEM replica image of steel B after annealing at 850°C. Fine precipitates with sizes of 10 nm to 40 nm exist in the steel, but conversely, a region where precipitates are extremely rare can also be observed in the vicinity of the grain boundaries. Relatively fine precipitates also exist inside the grains, but coarse grains show a tendency to exist in grains and at the grain boundaries. These precipitates were identified as NbC and Nb(C, N) by EDS analysis and electron diffraction.

A closer examination of the region near the grain boundaries where precipitates are rare reveals that Nb(C, N) exists only on one side of the grain boundary, and not on both sides. **Figure 5** shows the features of this microstructure. Whether these fine Nb(C, N) precipitates are present or not is considered to be the reason why steel B displays strength approximately 30 MPa higher than that of steel A. The region where precipitates are rare is called the PFZ (precipitate free zone).

Figure 6 presents a schematic illustration of the presumed mechanism by which this unique PFZ is formed. Immediately after recrystallization, fine NbC and Nb(C, N) are thought to precipitate relatively uniformly, but the NbC and Nb(C, N) which exist on grain boundaries undergo coarsening by rapid Ostward ripening due to grain boundary diffusion. As a result, the pinning force of the grain boundary is weakened, and grain boundary migration begins and the grains grow. On this assumption, comparatively coarse NbC and Nb(C, N) would remain after grain boundary migration.

Based on the above assumptions, it is thought that fine NbC and Nb(C, N) do not exist in the PFZs which form in this manner, and as a result, precipitation hardening is reduced and the PFZs are soft and have a low

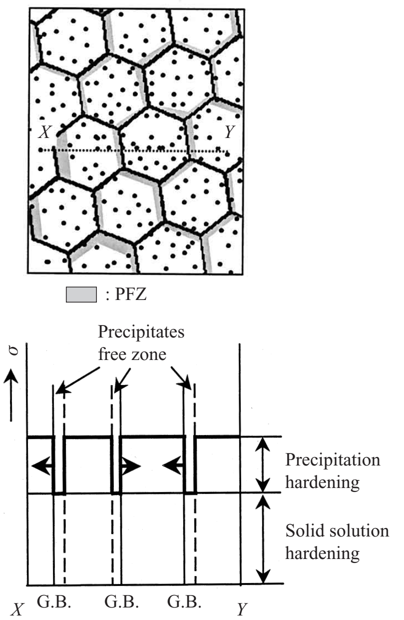

Fig. 5 Schematic illustration showing the hypothesis on the mechanism for lower yielding in steel B strengthened by the solid-solution elements and fine Nb precipitates with PFZ

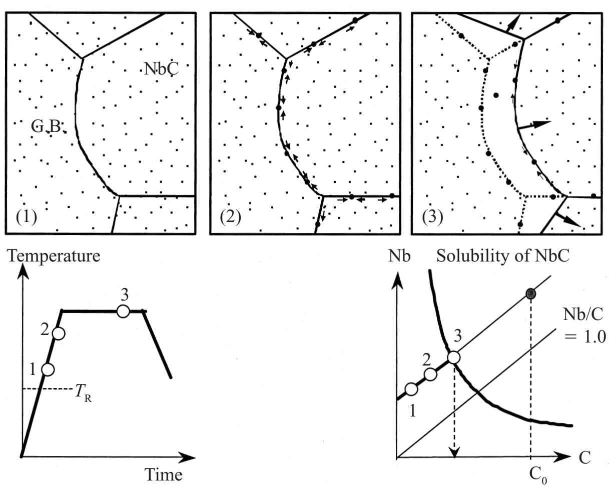

Fig. 6 Schematic illustration exhibiting the hypothesis on the mechanism of PFZ formation in steel B

yield ratio. Thus, if it is thought that material yield begins from stress concentrations in the vicinity of grain boundaries, it is reasonable to think that the distinctive mechanical properties of the PFZ determine the distinctive feature of low YR observed in steel B.

3. Effect of PFZ on Yield Strength[7)]

As described above, it is thought that the formation of PFZs results in a mechanism in which yielding begins at low stress in the initial deformation stage. To clarify this, samples with various amounts of PFZ were prepared and yield phenomena were compared.

Samples with various amounts of PFZ were prepared by hot-rolling and cold-rolling vacuum melted steels with the chemical compositions shown in **Table 2** to a thickness of 0.65 mm, and then performing recrystallization annealing at 750°C × 60 s at varying heating rates between 2 and 15°C/s.

Figure 7 shows the mechanical properties of the steels annealed at different heating rates. Although tensile strength (TS) is basically constant, yield strength

Table 2 Chemical composition of samples with various amounts of PFZ (mass%)

C	Si	Mn	S	Soluble Al	N	Nb
0.006 8	0.02	0.99	0.009	0.052	0.002 5	0.101

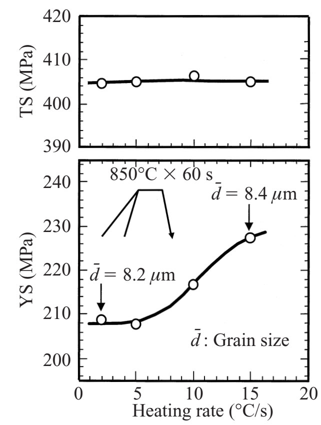

Fig. 7 Effect of heating rate on mechanical properties of the steel annealed at 850°C for 60s

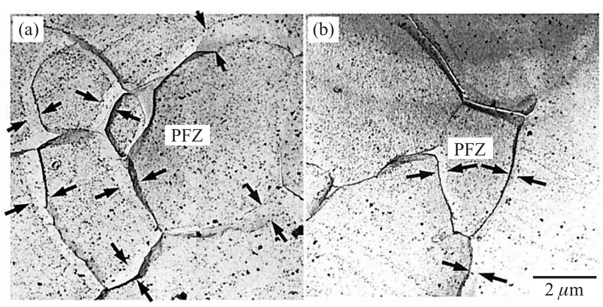

Photo 4 TEM replica micrographs of annealed specimens with the heating rates of (a) 2°C/s and (b) 15°C/s

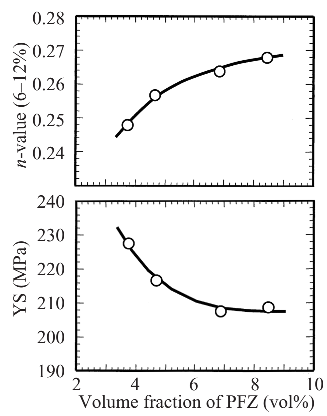

Fig. 8 Effect of volume fraction of PFZ on mechanical properties of annealed specimen

(YS) increased from 210 MPa to 230 MPa as the heating rate increased. The grain size remained virtually unchanged, showing a constant value (8.2–8.4 μm) at all heating rates.

Photo 4 shows TEM replica images of the steels annealed at heating rates of 2°C/s and 15°C/s. Although PFZs can be seen on one side of the grain boundaries, a higher PFZ formation ratio can be observed in the steel annealed at 2°C/s. **Figure 8** shows YS and the work hardening index (n-value) plotted against the volume fraction of PFZ measured from the TEM images. Yield strength decreases as the volume fraction of PFZ increases. From this, it is clear that PFZ is the reason for low YS.

4. Secondary Work Embrittlement[6)]

In general, IF steels have low grain boundary strength in comparison with intragranular strength because interstitial elements do not exist at the grain boundaries. In particular, in high-strength steels based on IF steel, it is necessary to consider measures to prevent reduced resistance to secondary embrittlement caused by the strength difference between the grains and grain boundaries. Therefore, resistance to secondary embrittlement was investigated in the new TS440 MPa grade super fine grain steel, SFGHITEN. This SFGHITEN was compared with the conventional Nb-added IF high strength steel sheet, and the effect of grain refinement and B addition, which is effective in improving resistance to secondary embrittlment in IF steels,[8)] was compared.

Using the chemical compositions shown in **Table 3**, which are based on 0.3%Si-2.0%Mn-0.075%P steel, sample steels with 4 levels of added B (2, 4, 10, and 15 ppm) were prepared. The procedure used in preparing cold-rolled and annealed steel strips was basically the same as in Chapter 2. After drawing the cold-rolled and annealed strips to a cup shape at a drawing ratio of 2.0 and mechanically grinding the edge to obtain a cup height of 35 mm, the deformation (fracture test) shown in **Photo 5** was performed at various temperatures, and the limit temperature for ductile deformation was used as the transition temperature for secondary work embrittlement.

Figure 9 shows the effect of B on the transition temperature. First, without B addition, steel A, which is a conventional TS440 MPa grade IF high strength steel sheet, displayed a transition temperature of −5°C, but in the steel B, which is an SFGHITEN, the transition temperature is reduced remarkably, to −80°C. This effect is attributed to alleviation of stress concentrations near the grain boundaries during deep drawing by refinement of the ferrite microstructure and formation of PFZs. Next, with increasing amounts of B addition, the transition temperatures of both steels improved, and with approximately 10 ppm B addition, even steel A showed resistance to secondary embrittlement equal to that of steel B without B addition. Thus, the fine ferrite structure and

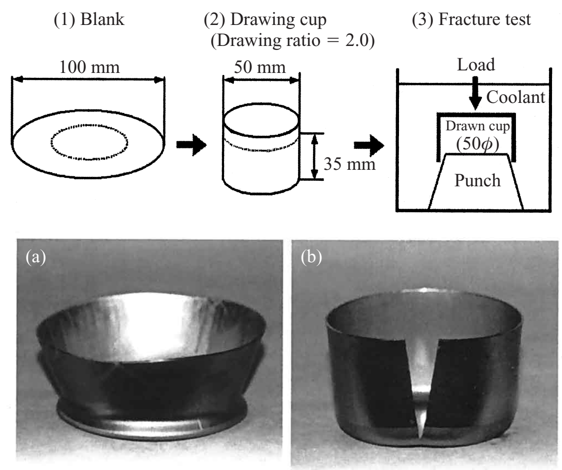

Photo 5 Schematic diagram of evaluation method of the transition temperature for secondary-work-embrittlement; (a) Non-brittle fracture, (b) Brittle fracture

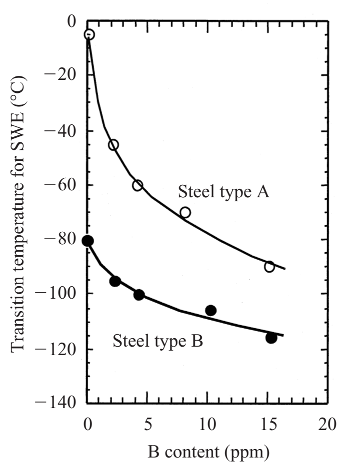

Fig. 9 Effect of B content on the transition temperature for secondary-work-embrittlement

Table 3 Chemical composition of steels investigated

Steel	C (ppm)	N (ppm)	S (mass%)	Nb (mass%)	Ti (mass%)	B (ppm)
A00	24	18	0.005	0.004	0.045	trace
A02	26	18	0.005	0.003	0.045	2
A04	26	17	0.005	0.004	0.045	4
A08	21	19	0.005	0.004	0.046	8
A15	20	19	0.005	0.003	0.045	15
B00	60	30	0.006	0.11	trace	trace
B02	66	26	0.006	0.11	trace	2
B04	59	24	0.006	0.11	0.001	4
B10	54	26	0.005	0.10	0.001	10
B15	58	25	0.006	0.11	0.001	15

Table 4 Mechanical properties of the SFGHITEN

Type	Grade	Thickness (mm)	YS (MPa)	TS (MPa)	El (%)	Mean r-value	T_c (°C)
CR	SFG340	1.0	190	345	44	1.9	−100
	SFG390	1.0	235	405	40	1.9	−85
	SFG440	1.0	290	446	37	1.9	−65
GA	SFG340	1.0	197	345	42	1.7	−90
	SFG390	1.0	227	400	38	1.7	−65
	SFG440	1.0	285	442	35	1.7	−45

CR: Cold-rolled steel sheet, GA: Galvannealed steel sheet
Tensile specimen: JIS No.5, Transverse direction
T_c: Critical temperature for anti-secondary work embrittlement in the flanging test of drawn-cup with the cup-height of 35 mm and the drawing ratio of cup diameter to blank diameter, 2.1 for 340 grade and 2.0 for 390 and 440 grade

existence of PFZ in SFGHITEN have a dramatic effect on resistance to secondary embrittlement.

5. Examples of Application

The Nb-added IF steel with the above-mentioned features was named "SFGHITEN®," and has been commercialized by JFE Steel as TS340 MPa, TS390 MPa, and TS440 MPa grade hiten steel. **Table 4** shows the typical mechanical properties of SFGHITEN.

Table 5 shows the mechanical properties of the conventional material and the developed steel when used in GA steel sheets. Even in GA, the developed steel displays a mean r-value of the soft 270E class, at r = 1.7. Yield strength is approximately 15 MPa lower than in the conventional material, and the n-value is also excellent.

Try-out pressing was performed with a die simulating the front fender of an automobile using commercially-produced samples of this steel. **Photo 6** shows the shape after pressing. Press-formability was evaluated by the formable range, which is decided by the fracture limit and wrinkle limit. **Figure 10** shows the results of this evaluation. With the developed steel, wrinkles could be prevented at a lower cushion force. This is attributed mainly to the excellent properties of the Zn-coated surface. On the other hand, no cracks occurred until the high cushion force side. It can be thought that this is mainly because the developed steel has a high r-value. As a result, it can be understood that only SFGHITEN is formable as a TS440 MPa grade GA sheet, demonstrating that the developed steel possesses outstanding formability in comparison with the conventional TS440 MPa grade hiten steel.

SFGHITEN with this excellent performance has already been adopted or is in the pre-adoption qualification stage at multiple automakers. Because weight reduction in panel parts has been achieved by adopting high-strength steel in all of these examples, this product is considered to provide performance which can fully satisfy the progressively higher weight reduction requirements of the future.

Table 5 Mechanical properties of steels used in press-forming test for a front fender model

Steel	YS (MPa)	TS (MPa)	El (%)	Mean r-value
Developed	285	442	35.0	1.70
Conventional	300	445	35.0	1.50

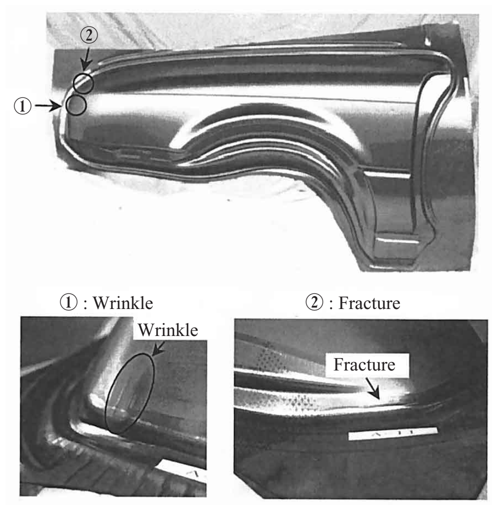

Photo 6 Front fender model for evaluating press-formability

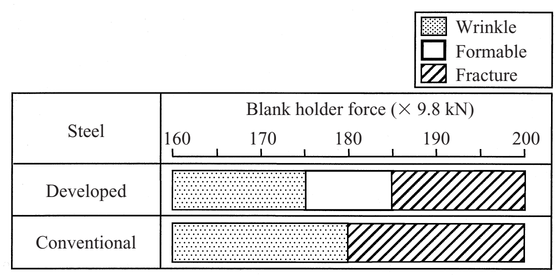

Fig. 10 Press-formability for a front fender model of the developed 440 MPa galvannealed steel sheet and the conventional galvannealed IF-HSS

6. Conclusion

A new super fine grain, high-strength steel (SFGHITEN) was developed. Responding to the present ongoing trend toward automobile weight reduction, SFGHITEN can be applied as a high tensile strength material for automotive exposed panels, which has been considered the most difficult application. Because this product possesses the properties necessary in exposed panels, including not only strength, but also deep drawability, surface precision after press-forming (shape-fixing property), coated surface quality, and resistance to secondary embrittlement, it is also capable of meeting the future needs of automobile weight reduction.

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