# METALLURGY OF TWO TYPES OF PRECIPITATION HARDENED HIGH STRENGTH FLAT-ROLLED PRODUCTS FOR AUTOMOBILE APPLICATIONS

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## Abstract

Two types of high strength flat-rolled products, which were originally developed by JFE, are presented in this paper. One is a high strength cold-rolled sheet for automobile body panels aiming to achieve a strength of 440 MPa with excellent formability, sufficient galvannealing (GA) applicability and anti-secondary work embrittlement. The other is a hot-rolled strip for under-body or chassis parts aiming at a strength of 780 MPa and having an excellent balance between elongation and stretch-flangeability, less scatter of mechanical properties and sufficient thermal stability of strength for GA application. From the metallurgical viewpoint, both products have unique features. The former contains around 50-60 ppm carbon, and Nb at the stoichiometric level in relation to carbon, which forms fine NbC precipitates in a ferrite matrix and subsequently promotes the formation of a PFZ (Precipitation Free Zone). Since the PFZ acts as a micro-yielding site, a low yield to ultimate strength ratio is attained with a fine grain structure. The latter steel contains Ti and Mo, which form MC type complex carbides by interface precipitation during  $\gamma/\alpha$  transformation after hot-rolling. Since the matrix structure is composed of a ferritic single phase, excellent stretch flangeability is achieved compared to multi-phase high strength steel types with the same strength.

### Introduction

Since the mid-1990s, the ULSAB, ULSAS, ULSAC and ULSAB-AVC projects have given the opportunity to not only reassess the conventional high strength steels (HSS) but also to work on the development of new types of HSS to achieve further weight-reduction of the car body whilst maintaining the collision safety and the stiffness of the body structure. Consequently, many new hot- and cold-rolled HSS have been developed up to the present day. For example, ultra-HSS with a tensile strength higher than 980 MPa with sufficient ductility, TRIP (Transformation Induced Plasticity) steels containing sufficient amounts of retained austenite which markedly improves the stretch formability. Regarding hot-rolled HSS, in particular, the types with a microstructure composed of a bainite or ferrite single phase, strengthened by fine precipitates, have been developed to take into account the need for enhanced stretch flangeability.

However, the body weight reduction achieved by use of HSS alone is already facing the following limitations:

- (1) Limit of formability (lack of shape retention and surface distortion),
- (2) Limit for the improvement of mechanical properties (large gap between weight saving target and expected value with improved mechanical properties),
- (3) Limit for the improvement of rigidity (required component's rigidity has, despite strength improvements, restricted the component thickness reduction attainable through the use of HSS).

Thus, the share of HSS used in the structure and safety related parts appears to be almost saturated in recent car designs.

Considering the recent research activities on HSSs for automotive applications, it seems that the TRIP and TWIP (TWinning Induced Plasticity) types, which have excellent strength and ductility balances, have attracted a lot of global research activity. However, their alloy design, with high Mn content, makes it difficult to use them widely in automobiles for economical as well as metallurgical reasons. Regarding strengthening of interstitial free (IF) steels, on the other hand, it has also been found that the addition of Mn as a solid-solution hardening element up to 2.0% markedly deteriorates the r-value. These examples indicate the definite limitation of alloying with substitutional solid-solution elements, in particular Mn, in the development of flat-rolled HSSs for automotive applications.

In this paper, two types of HSS, which were developed based on the common concept to intentionally utilize fine precipitation hardening, are introduced by extracting typical data from the technical articles presented by the authors so far. One is a high strength cold-rolled steel sheet for automobile body panels aiming up to a strength of 440 MPa which has excellent formability, sufficient GA applicability and anti-secondary work embrittlement [1-3]. The other is a high strength hot-rolled strip for under-body or chassis parts aiming at a strength of 780 MPa and having an excellent balance between elongation and stretch-flangeability, less scatter in mechanical properties and sufficient thermal stability of strength for the subsequent reheating required for GA application [4-6].

## Cold-rolled HSS with a Tensile Strength Higher than 390 MPa, Strengthened by Fine NbC Precipitates.

## Materials Design Concept

Regarding the solid-solution hardening of IF steels used for exposed panels, unavoidable problems have been experienced such as:

- (1) Addition of a high amount of Mn deteriorates the r-value,
- (2) Solid-solution hardening of the ferrite matrix by alloying with Mn, Si and P deteriorates the resistance to secondary work embrittlement,
- (3) GA qualities are detrimentally affected by high contents of Mn, Si and P.

Although grain refinement is an effective way to improve the toughness of steel, this has not been intentionally applied to flat-rolled products because the increase in yield stress deteriorated the shape retention capability of stamped panels.

Figure 1 shows the alloy design concept of grain-refined IF steels. Grain refinement and precipitation hardening are combined with solid-solution hardening to improve galvanizability and resistance to secondary work embrittlement.

The grain refinement and the precipitation hardening were achieved by an appropriate combination of a fine distribution of carbides with relatively high carbon content close to 60 ppm and a niobium (Nb) addition, enough to form NbC precipitates. By adding the precipitation strengthening on top of the base strength, the content of solid solution elements could be reduced. In particular, reduction of Si was effective in improving the GA quality.



Figure 1. Schematic diagram showing the metallurgical concept of the newly developed steel compared to the conventional IF steel.

## **Outstanding Mechanical Properties**

In general, the mean r-value of cold-rolled steel sheets is directly related to the ASTM grain size number as shown in Figure 2 [7]. The mean r-value can be improved by elevating the annealing temperature because of the further growth of the {111} grains, and can reach over 2.5 in IF steels. However, steel sheets with coarse grains cause a rugged surface, so-called "orange-peel," after stamping, which is not suitable for the surface quality required for exposed panels. The

newly developed grain-refined IF steel exhibited an outstanding balance of mean r-value and grain size compared to conventional cold-rolled sheets as shown in Figure 2. This was caused by a nucleation and growth of  $\gamma$ -fibre texture which was dominated by the grain refinement of the ferrite structure in the hot-band material.

Another feature of this steel was the unique yielding behavior, i.e. yield stress was kept low despite the tensile strength being increased by the precipitation hardening and the grain refinement as shown in Table I. For example, the 390 MPa grade steel has a yield stress which is comparable to that of the present 340 MPa BH steels which are currently applied widely for the exposure panels with anti-surface deflection.



Figure 2. Correlation between ASTM grain size number and mean r-values of cold-rolled steel sheets.

Table I. Mechanical Properties of 340 to 440 MPa Grade,
Grain-refined IF HSS, SFG-HITEN [16]

Туре	Grade	Thickness (mm)	YS (MPa)	TS (MPa)	El (%)	Mean r-value	Tc (°C)
CR	SFG390	1.0	235	405	40	1.9	-85
	SFG440	1.0	290	446	37	1.9	-65
GA	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

Tc : Transition temperature for anti-secondary work-embrittlement with a cup-height of 35 mm and a drawing cup ratio of cup-diameter to blank diameter, 2.1 for 340 grade and 2.0 for 390 and 440 grades.

## Formation of Microstructure

A surprising characteristic of this steel was the formation of a PFZ (Precipitation Free Zone) during annealing which dominated the unique mechanical properties as briefly discussed in the previous section. Figure 3 shows the distribution of fine precipitates observed in the 390 MPa grade steel. Fine NbC precipitates with diameter of 10 to 40 nm were observed. The arrays of relatively coarser precipitates seem to be distributed along the grain boundaries. Some of the arrays of coarse precipitates accompany the parallel precipitate arrays along the grain boundaries. In the areas between these pairs of arrays, very few fine precipitates are observed, which is close to the precipitation free zone (PFZ) besides the small numbers of coarser precipitates. While the so-called PFZ is formed on both sides of a grain boundary, in general, these areas with few precipitates were observed to be located along one side of the grain boundaries.



Figure 3. TEM image from replica and EDS spectra of precipitates, observed in the annealed sheet of 390 MPa grade of grain-refined IF HSS [10].

The increment in strength due to precipitation hardening and grain refinement is approximately 30 MPa. Therefore, it can be considered that the strength of the PFZ is lower than that of the grain matrix due to the absence of precipitation hardening. This is the dominant feature, which leads to the lower yield strength. However, with progress of deformation after yielding, intergranular deformation mainly takes place, and the tensile strength nearly equalizes the level of the grain matrix, strengthened by the fine NbC precipitates. Although it is well known that both grain-refinement and precipitation hardening increase the yield strength of steel, the grain-refined IF-HSS exhibits a yield/TS ratio lower than that of conventional solid-solution hardened IF-HSS, which is not consistent with conventional understanding.

Figure 4 shows a TEM micrograph highlighting the sub-structure, which was developed near the grain boundaries in the early stage of deformation after 0.5% tensile straining, in which the yielding has just occurred. It was observed that the dislocations were pinned by the NbC precipitates at the boundary between the PFZ and the precipitation dispersed matrix, and they bowed from the grain boundary side to the matrix side as indicated in the figure by the white

arrows. It was also found that the dislocation density was very low in the PFZ. Since the grain boundary is considered to act as the effective Frank-Reed source, it is suggested that the dislocations were generated at the grain boundaries and moved towards the matrix through the PFZ.



Figure 4. TEM micrograph of specimen deformed with 0.5% tensile strain [18].



Figure 5. Mechanism of providing low yield strength.

Furthermore, the PFZ region should be softer than the matrix where the NbC particles are dispersed homogeneously. Therefore, it was inferred that the dislocations could easily be generated at the grain boundaries accompanied by a PFZ. This is the probable reason why this steel exhibits a low yield strength with a fine grain structure as schematically depicted in Figure 5. Other results also clearly demonstrated that the yield strength of this steel depended on the volume fraction of the PFZ, which was controlled by the heating rate during annealing.

Regarding the anti-secondary work embrittlement, it was also improved by the grain refinement. The micro alloying of boron (B) was effective in further improving the anti secondary-work embrittlement.

## Hot-rolled HSS with Tensile Strength Higher than 780 MPa Strengthened by the Interphase Precipitation of (Ti,Mo)C Fine Complex Carbides.

## Concepts of the Material and Process Design

Several problems for utilizing TRIP steel have been experienced, such as:

- (1) High amount of Mn addition to obtain a sufficient volume fraction of meta-stable retained austenite is costly,
- (2) Variation of mechanical properties over the entire coil was large, caused by the instability of austempering condition,
- (3) Deterioration of the stretch-flangeability by secondary working, such as shearing and blanking, was large caused by the high susceptibility for deformation,
- (4) Softening of the hard phase was unavoidable during reheating such as in hot-dip galvanizing and welding.

As a solution to these problems, precipitation hardening by TiC with suppressed ferrite and pearlite transformation promoted by the addition of Mn and Mo was used, as schematically depicted in the CCT diagram in Figure 6.



Figure 6. Schematic CCT diagram of alloy design and on-line microstructure control.

## **Outstanding Mechanical Properties**

The most outstanding mechanical property characteristic of the 780 MPa HSS was the excellent balance between elongation and stretch flangeability as shown in Figure 7. (Stretch flangeability as measured by the hole expansion test). This balance was achieved by the steel microstructure which was composed of single phase ferrite in comparison to other steels whose microstructure is composed of mixed phases containing bainitic and/or martensitic hard phases.



Figure 7. Outstanding El-  $\lambda$  balance of hot-band strengthened by interphase precipitation (IPP).

## Formation of Microstructure and Origin of Mechanical Properties

The remarkable feature of this steel was the achievement of a tensile strength of up to 780 MPa with a ferrite single phase. This resulted from the precipitation of MC type (Ti,Mo)C, nano-scale fine complex carbides which were formed by interphase precipitation during the  $\gamma/\alpha$  transformation after the finish of hot-rolling, as shown in Figure 8. Against initial expectation, the results proved that Mo formed complex carbides, substituting half of the Ti atoms in TiC, formed by interphase precipitation.



Figure 8. Typical microstructure of 780 MPa class hot-strip, hardened by nano-scale interphase precipitates.

XRD analysis of the precipitates revealed that the (Ti,Mo)C formed a super lattice and satisfied the Baker-Nutting relationship with the ferrite matrix as shown in Figure 9. This precipitation was the dominant cause of both the marked increase in hardenability and the thermal stability of the ferrite phase in this steel.



Figure 9. Lattice structure of precipitates and crystallographic relationship with ferrite matrix.

The strengthening mechanisms in this steel were estimated as follows. Since the yield stress caused by the sum of solid-solution hardening of the given chemistry and hardening by grain refinement is around 400 MPa, the remaining 300 MPa must be made up by precipitation hardening ( $\Delta\sigma$ ).

The  $\Delta\sigma$  was calculated by applying the Orowan model to the nano-scale precipitates. From the results shown in Figure 10, the gap in strengthening from 400 MPa to 700 MPa, which was the yield stress of this steel, could be made up by precipitation hardening based on the Orowan mechanism.

Regarding the outstanding thermal stability of the strength of this steel, the hardness change of three steels, i.e. Steel A(Ti-Mo), Steel B(Ti-Nb), Steel C(Ti), isothermally held for  $8\times10^5$  seconds at 650 °C, was evaluated. In this experiment, the average size of the precipitates in each steel was 3 nm, 6 nm, and 13 nm in steels A, B, C, respectively. The hardness of Steel A was very stable compared to steels B and C as shown in Figure 11. Subsequent studies revealed that the thermal stability of the precipitates was improved by reducing their interfacial energy which was dominated by the excess amount of carbide forming elements in solution.



Figure 10. Strengthening of ferrite matrix by nano size fine carbides.



Figure 11. Softening behavior of three types of steels after isothermal holding at 650 °C.

## Summary

The typical features of the two types of steel developed by the authors were introduced. Most of the unique features of the steels were hard to predict at the beginning of the investigation.

When starting the R&D program, adding C up to around 60 ppm and coiling at a temperature higher than 600 °C appeared to be an unlikely way for the development of cold-rolled and hot-rolled HSSs, respectively. Yet, it led to the discovery of metallurgically innovative flat-rolled products.

There might be more undeveloped concepts that are worthy of assessment. It also seems interesting to further explore the feasibility of precipitation hardening with Nb, Mo and Ti, compared to conventional strengthening by solid-solution hardening and phase transformation hardening in flat-rolled products.

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