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DEVELOPMENT OF DEEP DRAWABLE GALVANNEALED TI-IF HSS CONTAINING NIOBIUM IN SOLUTION FOR AUTOMOTIVE BODY WEIGHT REDUCTION

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Abstract

It is important to improve (1) the Lankford value and (2) the wetting characteristics by molten Zn for the development of deep drawable galvannealed Ti-IF HSS containing Nb in solution having a tensile strength of more than 390 MPa corresponding to automobile industry's demand for weight reduction and high corrosion resistance. Concerning item (1), the effect of the coiling temperature under hot-rolling conditions on the Lankford value and the recrystallization texture of cold-rolled and annealed steel sheets were investigated together with basic research on hot-rolled microstructures and textures. In addition the metallurgical role of Nb in solution related to the improvement of the recrystallization texture and the Lankford value was discussed. Concerning item (2), the effect of reduction heating conditions before hot dipping on the Zn wetability was examined. Here the relationship between the formation of a Mn-related oxide layer and the Zn wetability was considered. Furthermore, Ti-IF mild steels containing Nb in solution are described as a base metal of deep drawable galvannealed Ti-IF HSS and various properties including the mechanical ones of industrial products developed by Nisshin Steel Co., LTD are introduced together with their automotive application.

Introduction

It is well known that extra low carbon (ELC) interstitial free (IF) steels, in which almost all C and N is stabilized by super-stoichiometric addition of Ti and/or Nb are suitable for automotive steel sheets because of not only their excellent deep drawability and stretch formability but also their non-aging property. In particular, due to the latter characteristics IF steels make it possible without difficulty to be treated in Continuous Galvanizing Lines (CGL) having no overaging zone. Therefore, the industrial mass-production of IF steels has been rapidly increased not only for hot and cold rolled steel sheets but also for hot-dip galvanized or galvannealed steel sheets. Furthermore, IF steels are now being applied to high strength steel sheets (HSS), especially in the field of automotive parts such as exposed panels, inner panels, members and so further for the purpose of reducing automotive body weight.

Based on the above mentioned background, the development of deep drawable galvannealed Ti-IF HSS containing Nb in solution with a tensile strength of more than 390 MPa was carried out by taking into account the Lankford value (r-value) determining the deep drawability and the wetting characteristics by molten Zn.

Ti-IF Steel Containing Nb in Solution as Base Metal of Galvannealed Ti-IF HSS

Superformable steels are required for producing automotive exposed panels and inner panels requiring high stretch formability and good deep drawability. For this purpose, several types of IF steels were developed in the past, as shown in Table I [1-4]. Ti-IF steels (No. 1) containing TiC, $TiS, Ti_4C_2S_2$ and TiN precipitates with small amounts of Ti in solution seem to be more or less preferred for mass-production from the standpoint of easy manufacturing as well as Nb-IF steels [5] (No.2) whose precipitation behavior is not the same as that of Ti-IF steels. On the other hand, Nb and Ti co-added IF steels [6, 7] (No.3), which seem to have some difficulties in industrial mass-production owing to some solute carbon retained in the end product, were reported to have desirable bake hardenability (BH) properties, little powdering of galvannealed steels and deep drawing-induced brittleness together with an excellent r-value.

Table I. Outlines of changes in precipitates and Ti or Nb in solution of IF steels.

No.	IF steels	Precipitates			X in solution
1	Ti-IF	TiC	$TiS, Ti_4C_2S_2$	TiN	small amounts of Ti or Nb
2	Nb-IF	NbC	MnS	AlN	
3	Nb,Ti-IF	NbC	MnS	TiN	-
4	Ti-IF+Nb	TiC	$TiS, Ti_4C_2S_2$	TiN	Large amounts of Nb

Here an outline of experimental results obtained with Ti-IF mild steel containing large amounts of Nb in solution (No.4) as a base metal for galvannealed Ti-IF HSS are described by taking into account the grain refinement with respect to the resistance against secondary cold work embrittlement, the r-value and the planar anisotropy of the r-value [8].

Experimental Procedure

Vacuum-molten ELC steels were used with the chemical compositions listed in Table II. The ingots were hot-forged, hot-rolled (slab reheating temperature SRT: 1230 ºC, finishing temperature FT: 920 ºC), heat treated for coiling simulation (coiling temperature CT: 700 ºC), cold rolled (cold-rolling reduction CR: 75%) and annealed. A tensile test was performed using the JIS No. 5 type of specimen. The r-value was measured after straining of 15% in the longitudinal (L), diagonal (D) and transverse (T) directions and the mean r-value was calculated from the equation of

$$r_{mean}=(r_L+2r_D+r_T)/4. \quad (1) \qquad (1)$$

Table II. Chemical compositions (mass %) of steels used.

C	Si	Mn	P	Ti	Nb	N
0.003	0.01	0.16	0.005	0.07	0~0.06	0.002

Experimental Results and Discussion

Figure 1 shows the influence of the Nb content in solution on changes in the hardness for cold rolled Ti-IF steels as a function of the annealing temperature. The soaking time at each temperature for continuous annealing treatment was 60 seconds. The hardness of each steel falls gradually with increasing annealing temperature until it reaches the level of Ti-IF steel annealed at 750 ºC. However, it is clearly seen that the tendency of its decrease depends on the Nb content in solution, i.e., it is suggested that Nb in solution influences the recrystallization kinetics in Ti-IF steels and retards the recrystallization finishing temperature accompanied by an increase of the Nb content in solution. Moreover it is found by means of optical microscopy that the recrystallization temperature of 0.04 to 0.06% Nb microalloyed steels is about 50 ºC higher than that of Ti-IF steel and 0.02% Nb steel in the present work, which appears to be caused by a solute drag effect of Nb in solution, although it is still necessary to clarify the mechanism in terms of variation in precipitates and microstructure of hot rolled steels.

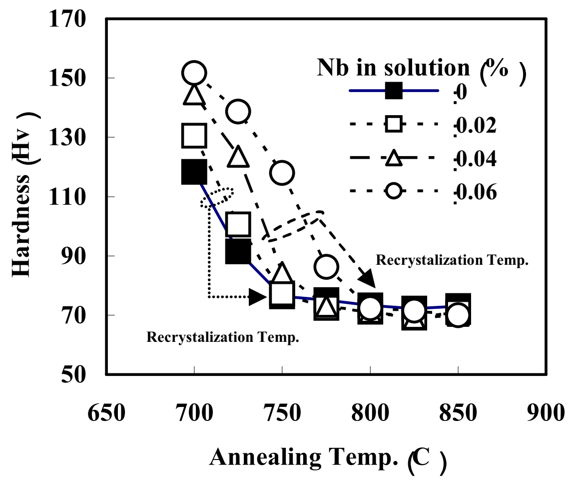

Figure 1. Effect of Nb content in solution on hardness change in function of the recrystallization temperature of Ti-IF steels.

Figure 2 shows the influence of the Nb content in solution on the change of ferrite grain size for Ti-IF steels as a function of the annealing temperature. This figure demonstrates that there is little change in the ferrite grain size of 0.04%Nb and 0.06%Nb steels while the ferrite grain size of Ti-IF steel gradually raises with an increasing annealing temperature. Therefore Nb in solution is considered useful to obtain fine grains in Ti-IF steels without deteriorating the r-value by the grain refinement.

Figure 3 shows the influence of the Nb content in solution on the r-value for Ti-IF steels annealed at 850 ºC. The r-value of Ti-IF steel increases with increasing Nb in solution and reaches a value of more than 2 at 0.05% Nb in solution. Moreover planar anisotropy of the r-value improves by Nb in solution and reaches a Δr ($=<r_L+ r_T-2r_D>/2$) level below about 0.5 at 0.05% Nb in solution.

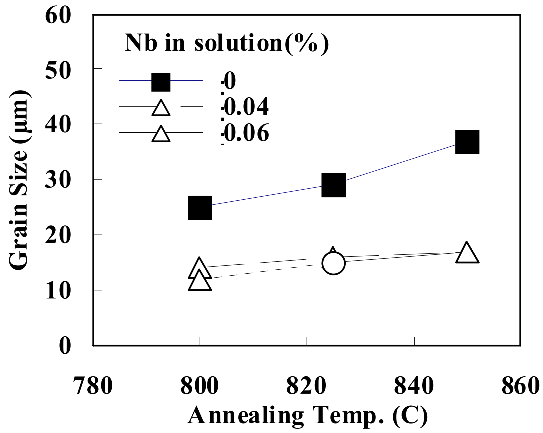

Figure 2 Effect of Nb in solution content and annealing temperature on changes in ferrite grain size of Ti-IF steels.

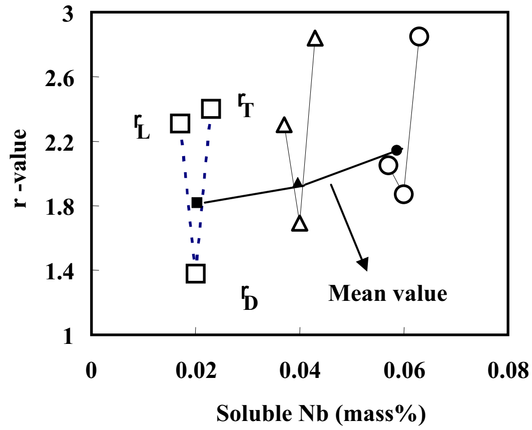

Figure 3 Effect of Nb in solution content on changes in r-value of Ti-IF steels annealed at 850C.

Development of Deep Drawable Galvannealed Ti-IF HSS Containing Nb in Solution

Deep drawable galvannealed IF HSS with a tensile strength higher than 390 MPa is required for the weight reduction of automobile bodies by down gauging in order to save energy thus contributing to ameliorate the global ecological problems. To meet this strong demand, several types of IF HSS strengthened by solid-solution hardening with Si, Mn, P and Cr have been reported during the last decade [9-11]. Among them, P alloyed IF steels with a strength level of 340-370 MPa are widely employed in current industrial production because a P addition can increase the strength most effectively and cheaply at minimal deterioration of the drawability [13]. However, when higher strength was pursued, further addition of P was experienced to cause a deterioration of the resistance against cold work embrittlement associated with its segregation to the grain boundaries [14]. On the contrary, the addition of Si, which exhibits the strongest solution hardening effect besides P, is barely suitable for galvannealed steels [14]. Therefore, the co-addition of Mn and P to IF steels with minute amounts of B added for the grain boundary strengthening was selected. As a result, deep drawable galvannealed Ti-IF HSS containing Nb in solution with a strength level of 390 to 440 MPa were newly developed for automotive unexposed panels [15]. At the same time, the effect of the coiling temperature after hot-rolling on the r-value and the effect of reduction heating conditions before hot dipping on the Zn wetability were investigated for the Ti-IF high strength steels developed.

In the following, experimental results and the role of Nb in solution related to deep drawability are described [16-18].

Improvement of r-value for galvannealed Ti-IF HSS containing Nb in solution

Experimental procedure

The chemical composition of the steel used in this experiment is shown in Table III, which is the same as that of the developed galvannealed Ti-IF HSS with a strength level of 440 MPa containing Nb in solution. Here Ti-IF mild steel is used for comparison. After reheating at 1250 ºC, the slabs were hot-rolled to 3.2 mm thick sheets above 920ºC. Subsequently the hot rolled steels were cooled to 550ºC or 700ºC as coiling simulation. After cold rolling to 0.8mm thickness, annealing at 850ºC for 60 seconds was performed. Tensile testing, observations of microstructures and precipitates by TEM (Transmission Electron Microscopy) and investigation of texture by EBSP (Electron Back Scattering Pattern) were carried out with hot rolled steels and annealed steels. Moreover the softening ratio was used to evaluate recrystallization behavior in γ region of Ti-IF HSS containing Nb in solution compared with Ti-IF HSS.

Table III. Chemical composition (mass %) of steel used.

C	Si	Mn	P	Ti	Nb	N
0.003	0.01	1.5	0.10	0.07	0.05	0.002

Experimental Results and Discussion

Table 4 shows the mechanical properties of cold rolled and annealed steels. In the case of annealed steels, low temperature coiling improved considerably mean r-value and total elongation (T.El) without lowering tensile strength grades, although it is well known that high temperature coiling is effective for improving the r-value in mild IF steels.

Table 4 Mechanical properties of annealed steels.

CT / ºC	TS /MPa	T.El /%	Mean r-value
550	467	33.8	1.54
700	459	31.0	1.31

Figure 4 shows hot rolled microstructures observed by optical microscope and with electropolished thin foils by using TEM, together with the observation of texture distributions by EBSP. In hot-rolled steels coiled at different temperatures, two types of microstructures are observed. For Ti-IF HSS containing Nb in solution, the microstructure coiled at low temperature showed a bainitic-ferritic structure with high dislocation density accompanied by a reasonable degree of recovery, which was recently reported to be a tempered martensite-like structure, although the microstructure coiled at higher temperature of 700ºC showed a polygonal ferritic structure with smooth grain boundaries. On the other hand, the microstructure coiled at each temperature for Ti-IF mild steel showed a polygonal ferritic structure with smooth grain boundaries. For Ti-IF HSS containing Nb in solution, a hot-rolled steel coiled at a low temperature, with a bainitic-ferritic microstructure, showed appreciably strong fiber textures from near {111}<112> to {211}<011> and from {100}<011> to {211}<011>, which were well known as typical cold-rolled textures, whereas textures of other steels including Ti-IF HSS containing Nb in solution coiled at 700ºC, with a polygonal ferritic structure, were randomized.

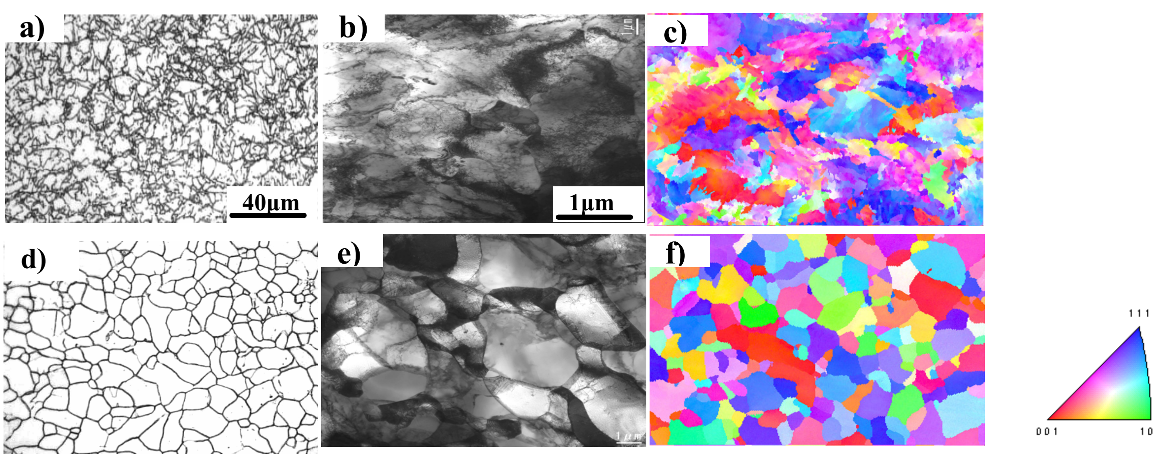

Figure 4. Optical micrographs a), b) and observations of microstructures by TEM b), e) and distributions of texture by EBSP c), f) for hot-rolled steels coiling at 550 ºC. a), b), c) : Ti-IF HSS containing Nb in solution, d), e), f) : Ti-IF mild steel.

Figure 5 shows the continuous cooling transformation diagram of Ti-IF HSS containing Nb in solution obtained with optical microscopy for the relative evaluation of microstructures. It was clear that changes in microstructures of hot-rolled steels depended on the cooling rate. It was found that hot rolled microstructures changed from a polygonal ferritic structure to a bainitic-ferritic structure with increase of cooling rate. Therefore it was concluded that a bainitic-ferritic structure obtained by a low temperature coiling was presumably due to rapid cooling after hot rolling.

Figure 6 shows the change in the softening ratio with the holding time after deformation at 900 and 950ºC for Ti-IF HSS containing Nb in solution and Ti-IF HSS. It was evident that the onset of recrystallization was retarded by addition of Nb in solution. Therefore it was suggested that recrystallization behavior after hot rolling in γ region affected by Nb in solution, which mainly segregated in grain boundaries, as shown in Figure 4, reflected a strong fiber texture in hot rolled steel coiled at low temperature.

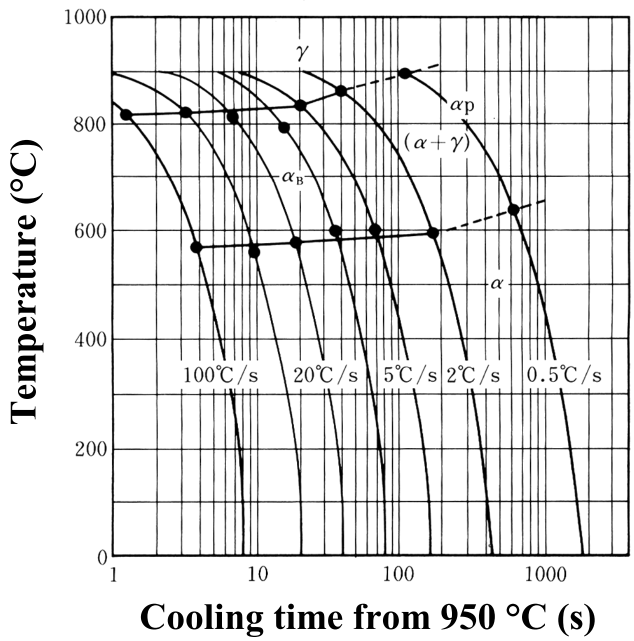

Figure 5. CCT diagram of ELC-1.2%Mn-0.05%P-Ti-Nb steel.

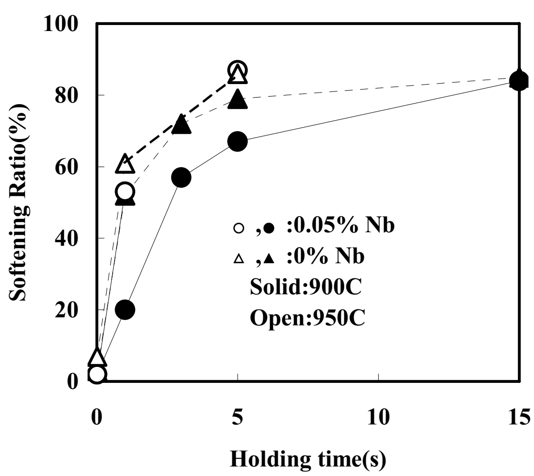

Figure 6. Softening curves of Ti-IF HSSs containing Nb in solution compared with Ti-IF HSSs.

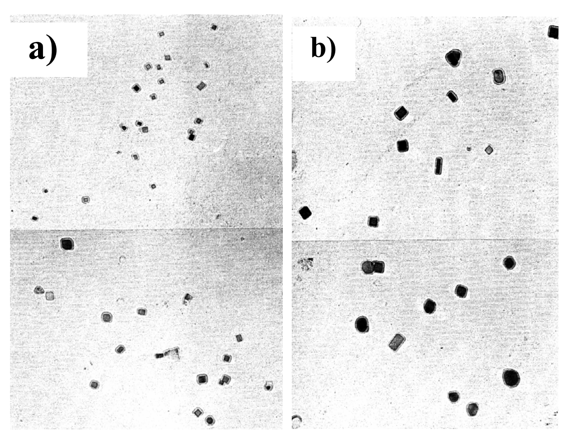

Figure 7. TEM images of precipitates extracted from hot-rolled steels for Ti-IF HSS containing Nb in solution. a) coiled at 550 ºC, b) coiled at 700 ºC

Figure 7 shows one of the typical TEM images of morphological change of precipitates extracted on carbon-replicas from hot rolled Ti-IF HSS containing Nb coiled at different temperatures. In both steels, Ti sulfides including TiS and $Ti_4C_2S_2$ were mainly observed. Furthermore FeTiP precipitates were frequently observed. On the other hand, it was clear that hot-rolled steel coiled at a low temperature exhibited a dense dispersion of fine precipitates, whereas that coiled at a high temperature showed a sparse dispersion of coarse precipitates. The mean diameter of precipitates in the low temperature coiled steel was about from 50nm to 100 nm, whereas that in the high temperature coiled one was approximately more than 100 nm. From these observations, it was concluded that the effect of size and dispersion of precipitates on development of recrystallization texture could be disregarded.

Therefore, it was considered that the improvement of the r-value and the recrystallization texture of Ti-IF HSS containing Nb in solution resulting from a low temperature coiling was attributed to hot-rolled textures which was obtained by a change in the recrystallization behavior after hot rolling in the austenite region affected by Nb in solution and a bainitic-ferritic structure which was generated by a rapid cooling just after hot rolling.

Effect of reduction heating conditions before hot dipping on Zn wetability

Experimental procedure

Ti-IF HSS containing Nb of 0.8 mm thickness was used as specimens, with the composition shown in Table III. They were preheated for 40 seconds at given temperatures from 650 to 850 ºC. At the same time the hydrogen content in H_2-N_2 atmosphere gas was changed 5 to 50 vol.% in the reduction heating zone of a lab-simulator, after which they were immersed at a temperature between 430 and 580ºC for 2 seconds in a solution of zinc and aluminum (0.14 mass%), that was heated in the same atmosphere to 460 ºC, thus fabricating hot-dip galvanized steel sheets with a coating weight of 60 g/m^2 per side. The surface oxide layer before dipping was investigated by use of Auger Electron Spectroscopy (AES) and X-ray diffraction (XRD), and hot-dip galvanized steel sheets were assessed according to counting number of bare spots on surface (50×150mm area) with respect to the wetting characteristics with molten zinc.

Experimental results and discussion

Figure 8 shows effect of the heating temperature and the H_2 content on the number of bare spots compared with conventional Ti-IF steel. As is shown, for conventional Ti-IF steel, good zinc wetability was obtained under each condition. On the other hand, a lot of bare spots were recognized on the surface of Ti-IF HSS containing Nb in solution. Thus, it is suggested that zinc wetability of Ti-IF HSS containing high Mn and P was not good in comparison with conventional Ti-IF steel. However it can be seen that the number of bare spots in Ti-IF HSS containing Nb depended on the heating temperature and the H_2 content in the reduction gas, i.e., it remarkably decreased with raising heating temperature and H_2 content, although in the case of heating temperatures of less than 750ºC numerous bare spots were observed under each H_2 content. Moreover it is evident that bare spots disappeared when the heating temperature was raised above 800ºC in an atmosphere of 40~50vol.% H_2-N_2 reduction gas.

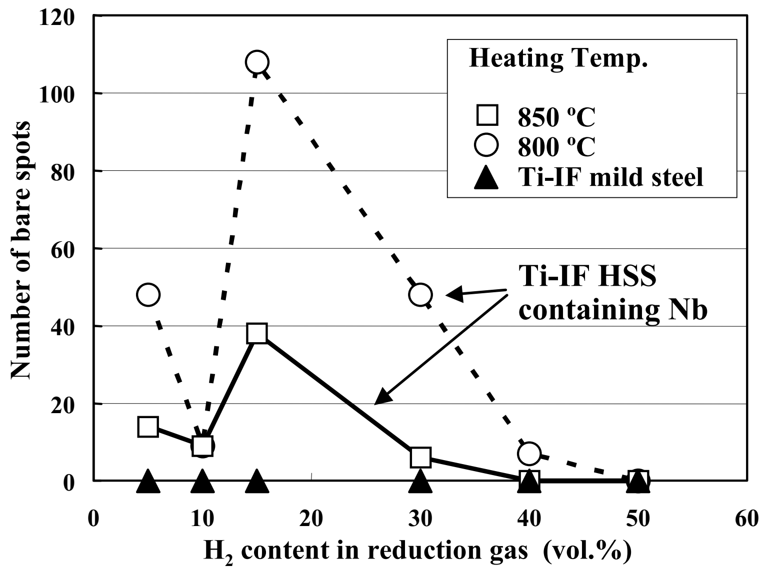

Figure 8. Effect of heating temperatures and H2 content on bare spots compared with Ti-IF mild steel. (Ti-IF mild steel:heating at 650~850 ºC).

Figure 9 shows the effect of the steel temperature before dipping on the number of bare spots compared with conventional Ti-IF steel. As shown, for Ti-IF mild steel, good zinc wetability was obtained under each condition, whereas for Ti-IF HSS containing Nb poor wetability appears. However, it can be clearly seen that the number of bare spots significantly decreased with a raising steel temperature before dipping and disappeared by means of increasing steel temperature above 490°C when Ti-IF HSS containing Nb sheets were heated to 850ºC in an atmosphere of 50 vol.% H_2-N_2 reduction gas.

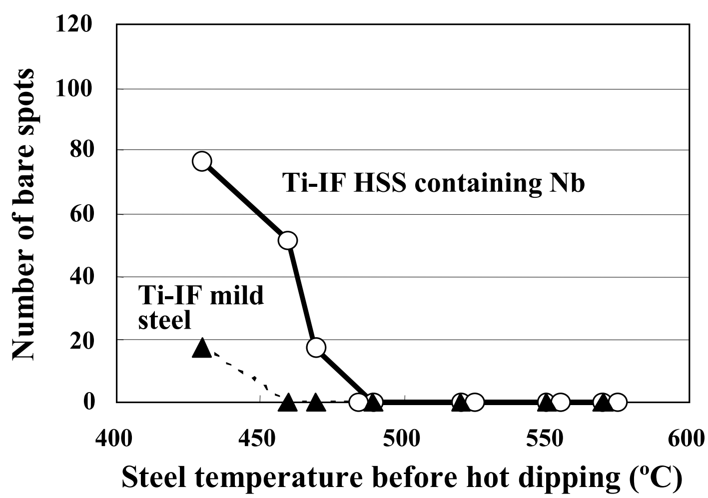

Figure 9. Effect of steel temperature before hot dipping on bare spots. (Heating temperature:850 ºC).

Figure 10 shows an AES depth profile in Ti-IF HSS containing Nb for the elements Fe, O and Mn after heating at 700ºC in an atmosphere of 50vol.% H_2-N_2 reduction gas before dipping. As appears, Mn enrichment in Ti-IF HSS containing a Nb surface layer was observed, while neither P nor Si enrichment can be identified in the present work. However Mn enrichment in the surface layer decreased with a raising temperature and disappeared when heating to above 800 ºC. Figure 11 shows a XRD profile for Ti-IF HSS containing Nb after heating at 700ºC in an atmosphere of 50 vol.% H_2-N_2 reduction gas before dipping. It is evident that Mn-related oxide films including MnO, Mn_2O_3, and Mn_3O_4 were formed on the surface. Nevertheless, as the heating temperature was raised above 800ºC and the H_2 content increased to more than 40vol.%, Mn-related oxide films disappeared.

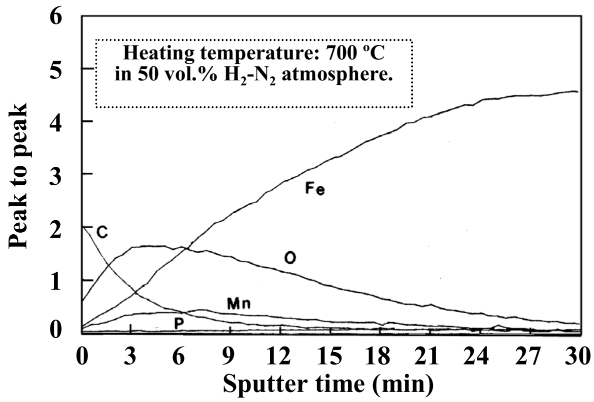

Figure 10. AES depth profile in Ti-IF HSS containing Nb for elements of Fe, O, Mn and P.

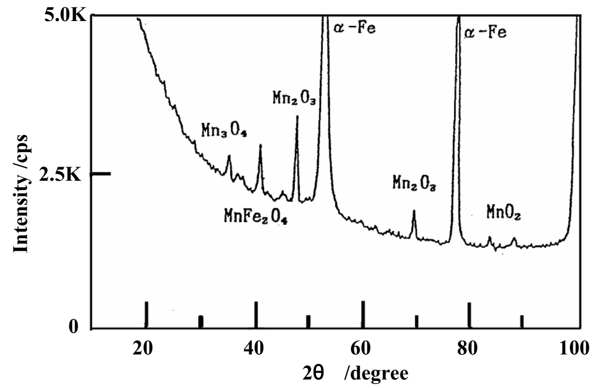

Figure 11. XRD profile of Ti-IF HSS containing Nb after heating at 700ºC in 50vol.%H2-N2 atmosphere before hot dipping.

From these experimental results and observations, it is concluded that wetting characteristics with molten zinc in galvannealed Ti-IF HSS including high Mn improved as the heating temperature was raised above 800 ºC and the H_2 content in the reduction gas increased to more than 50 vol.% resulting in the disappearance of Mn-related oxide films at the surface. Furthermore it is found that sheet temperature before dipping should be higher than that used for conventional Ti-IF steel.

Properties of Developed Products and Application to Automotive Usage

Table V shows typical mechanical properties, anti-powdering properties and resistance to deep-drawing induced brittleness of the developed products. Each has excellent properties for automotive parts due to controlling chemical compositions and processing conditions described above. Deep drawable galvannealed Ti-IF HSS containing 0.05% Nb in solution with a strength level of 390~440 MPa developed and r-value improved by a low temperature coiling were applied to not only automotive inner panels but also members such as front side member, center cross member and others for lightening of the automotive body weight by gauge down.

Table V. Typical properties of Ti-IF HSS containing Nb in solution developed.

	Mechanical Properties				Anti-powdering properties (g□m^{-2})	Deep drawing-induced brittleness (ºC)
	YS (MPa)	TS (MPa)	T.El (%)	r-value		
390 MPa	260	410	35.9	1.6	Less than 0.1	-80
440 MPa	310	470	32.5	1.5		-60

Conclusions

The effect of the coiling temperature after hot rolling on the Lankford value and the recrystallization texture of annealed steels, and the effect of reduction heating conditions before dipping on zinc wetability has been investigated in order to achieve further improvement of formability and wetting characteristics with molten zinc for galvannealed Ti-IF HSS, which contains a large amount of Nb in solution, Mn and P. The results obtained are as follows. In this paper, the application of modern Ti-IF steels and galvannealed Ti-IF HSSs containing Nb in solution to automotive usage were mainly reviewed paying some attention to their physical metallurgy. As shown here, improvement of planar anisotropy of r-value of Ti-IF steels was carried out with grain-refined microstructure and the mean r-value of galvannealed Ti-IF HSS increased with a low temperature coiling using Nb in solution, which contributed to expanding the application to automotive inner panels, members and other parts.

Effect of coiling temperature after hot rolling on Lankford value and recrystallization texture

(1) The Lankford value, closely associated with deep drawability of galvannealed Ti-IF HSS, depends on the coiling temperature. Lower temperature coiling increases the r-value.
(2) Hot-rolled sheets with bainite ferrites after low temperature coiling show a strong fiber texture from near {111}<112> to {211}<011> and from {100}<011> to {211}<011>, which result in the formation of a texture with developed {554}<225> orientation and an improved r-value in galvannealed Ti-IF HSS.
(3) Hot-rolled microstructures and texture play an important role in the development of {554}<225> orientation in galvannealed Ti-IF HSS.

Effect of reduction heating conditions before dipping on zinc wetability

(1) Zinc wetability depended on the heating temperature and H_2 content in the reduction gas. Bare spots related to zinc wetability remarkably decreased with raising heating reduction H_2 content and steel temperature before dipping.

(2) Bare spots disappeared when the heating temperature was raised to more than 800 ºC in an atmosphere of 40~50vol.% H_2-N_2 reduction gas. It was found that disappearance of Mn-related oxide films resulted in the improvement of wet ability.

Based on the experimental results described above, deep drawable galvannealed Ti-IF HSS containing Nb in solution having a tensile strength of 390~440 MPa have been actually produced using a CGL.

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