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Synopsis :
A thin Zn-Ni alloy electroplated steel sheet RIVER HI-ZINC was developed to reduce the perforation corrosion of automotive panels. The Zn-Ni alloy, consisting of a γ single phase, contains about 12% Ni. Coating adhesion and weldability of the alloy are excellent. Its corrosion resistance is four times greater than that of pure Zn-coated steel with the same coating weight. However, phosphatability and paintability of Zn-Ni alloy are somewhat poor. In their improvements, Fe-P (≦0.5wt.%) was electroplated on the Zn-Ni alloy, and double-layered Fe-P/Zn-Ni electroplated steel RIVER HI-ZINC SUPER has been developed for use on the exposed side of automotive panels. Uniformly distributed small amount of P in the Fe-P layer enhances the phosphating reaction and contributes to forming closely-packed phosphophyllite, which improves wet adhesion and reduces creepages (blisters) after the 3-coat painting in corrosion tests. Moreover, the Fe-P layer reduces crater-form paint defects during the cationic electrodeposition.

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Development of Zn-Ni Electroplated Steel Sheets "River Hi-Zinc" and "River Hi-Zinc Super"*1

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A thin Zn-Ni alloy electroplated steel sheet **RIVER HI-ZINC** *was developed to reduce the perforation corrosion of automotive panels. The Zn-Ni alloy, consisting of a γ single phase, contains about 12% Ni. Coating adhesion and weldability of the alloy are excellent. Its corrosion resistance is four times greater than that of pure Zn-coated steel with the same coating weight. However, phosphatability and paintability of the Zn-Ni alloy are somewhat poor. In their improvements, Fe-P ($\leqq$ 0.5 wt.%) was electroplated on the Zn-Ni alloy, and double-layered Fe-P/Zn-Ni electroplated steel* **RIVER HI-ZINC SUPER** *has been developed for use on the exposed side of automotive panels.*

Uniformly distributed small amount of P in the Fe-P layer enhances the phosphating reaction and contributes to forming closely-packed phosphophyllite, which improves wet adhesion and reduces creepages (blisters) after the 3-coat painting in corrosion tests. Moreover, the Fe-P layer reduces crater-form paint defects during the cationic electrodeposition.

1 Introduction

In recent years, corrosion of automotive bodies caused by deicing salt applied to roads in North America and Northern Europe has led to serious customer complaints and a corresponding effort of the part of automobile manufacturers and steelmakers to find practical solutions. Wide-ranging countermeasures have been adopted, including both the rethinking of auto body design and the use of precoated steel sheets and improvements in phosphate treatment and electrodeposited primers.[1-3]

In order to ensure adequate corrosion resistance, heavily zinc-coated steel sheets and prepainted steel sheets have been widely used as precoated materials. These types of sheet, however, cause such certain production problems as susceptibility to peeling of coated and prepainted film during press forming and early deterioration of welding electrodes during body manufacture. To solve these application problems, thinly coated, highly corrosion-resistant precoated steel sheets are considered necessary. As a result, various products, such as zinc-alloy electroplated steels (single or double layer), composite coated steels,[4-8] and zinc-alloy electroplated steels coated with conductive zinc-rich paint have been developed.[9,10]

In these circumstances, Kawasaki Steel has developed **"RIVER HI-ZINC"**, a Zn-Ni alloy electroplated steel, and **"RIVER HI-ZINC SUPER"**, a double-layer electroplated steel, to meet the needs of automobile manufacturers for corrosion resistant body materials. These steels are produced by the electroplating process and are suited to use in any of the automotive steel materials, such as the high tensile strength steel or bake-hardened steel, frequently used in recent years. In addition, the process permits easy coating on either one or both sides. The authors have given attention to Zn-Ni alloy electroplating, which produces excellent corrosion resistance.

RIVER HI-ZINC, with its excellent corrosion resistance, allows thin coating, and it possesses superb formability and weldability, unobtainable with previous precoated steels. RIVER HI-ZINC SUPER, a double-layer electroplated steel,[11] has been developed to improve on the phosphatability of RIVER HI-ZINC and to increase its wet adhesion after 3-coat painting. Its upper-layer Fe-P coating permits excellent phosphatability and wet adhesion of paint film with either spray- or dip-type phosphating, making it suitable for exterior body use in

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automobiles.

This report concerns the properties of these two newly developed steels.

2 Composition of RIVER HI-ZINC Coating Layer

To determine Ni content in the Zn-Ni alloy electroplated layer, an examination was made of the effect on corrosion resistance of the Ni content in the electroplated layer. Corrosion resistance of Zn-Ni alloy electroplating depends on the Ni content of the plated layer. In the salt spray test, the entire surface of a sample sheet with a pure Zn coating containing no Ni developed red rust in 24 h, while, on the other hand, a Zn-Ni alloy plating with a Ni content of 10 to 18% developed no red rust even after 120 h, indicating that Zn-Ni plating has at least four times the corrosion resistance of pure Zn plating, coating weight being equal.

The crystalline structure of the 10 to 18% Ni plated layer, which showed an excellent corrosion resistance, was found, upon X-ray diffraction examination, to have formed a γ single phase. This is considered a complex cubic lattice of a solid solution of intermetallic compound of $NiZn_3$, Ni_5Zn_{21}, etc.[12] When the Ni content drops to below 10%, an η phase appears, while, when Ni content exceeds 18%, an α phase is present in addition to the γ phase, thereby forming a two-phase structure.

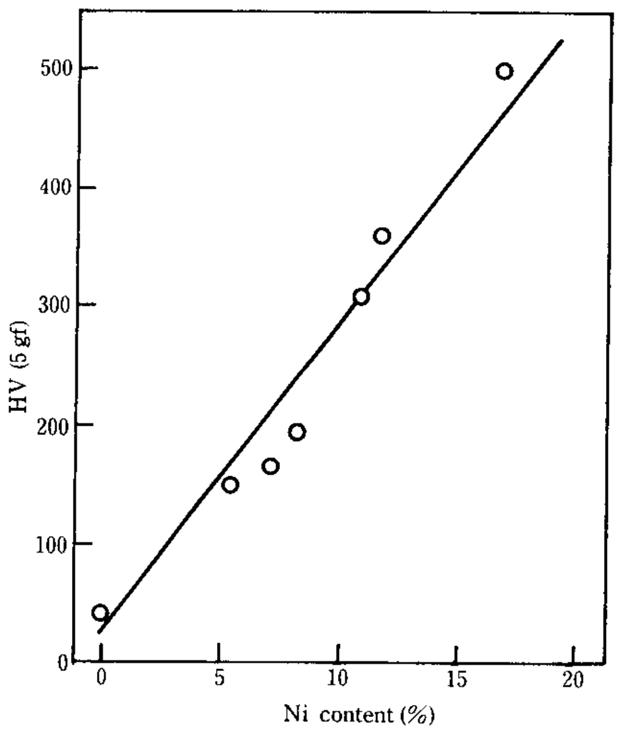


Fig. 1 Vickers hardness of Zn-Ni alloy electroplated layer

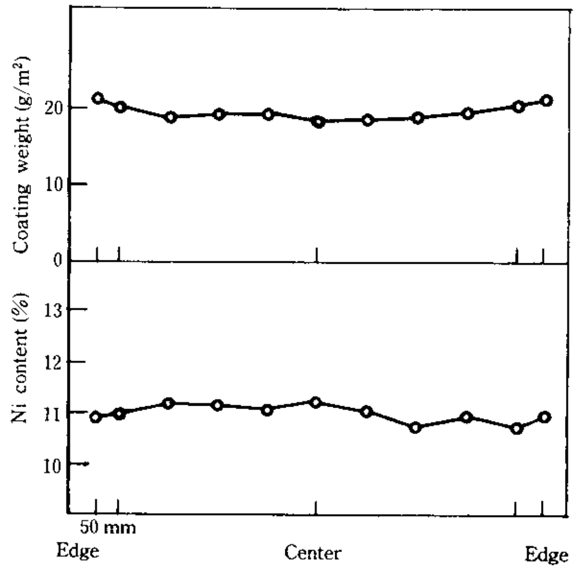


Fig. 2 Distribution of Ni content and coating weight in width direction of Zn-Ni alloy electroplated steel strip

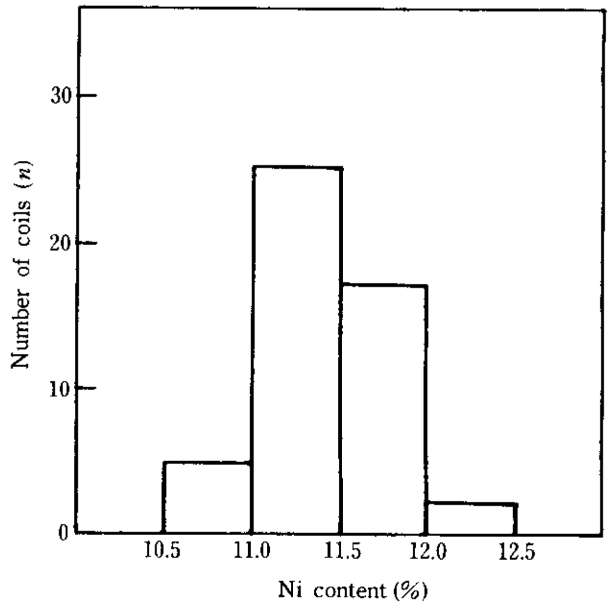
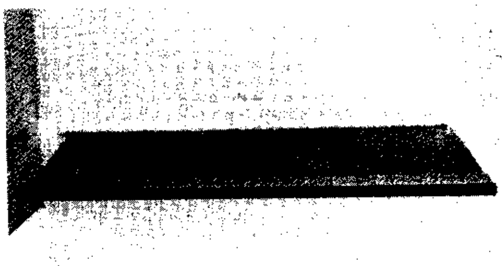


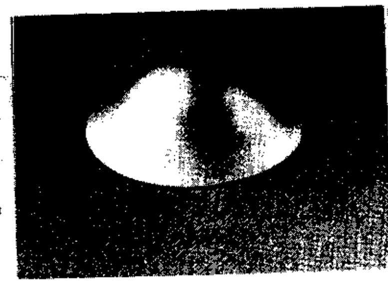
Fig. 3 Distribution of Ni content in Zn-Ni alloy electroplated steel coils ($n = 49$)

On the other hand, hardness of the coated layer rises as Ni content increases, as shown in **Fig. 1**. Consequently, as the Ni content rises, the coating becomes liable to develop microcracks. Thus it can be said that a γ single phase coating containing a lower Ni content, in the range of 10 to 13%, should have both excellent corrosion resistance and formability.

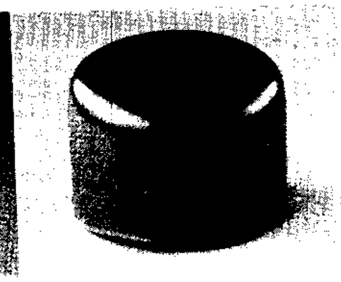
RIVER HI-ZINC meets this description, being manufactured to have a γ single phase coating with a Ni content of 10 to 13%. **Figure 2** shows that the width-



0 T bending



Erichsen dome
(height : 9 mm)



Deep-drawn cupping
(drawing ratio : 1.95)

Photo 1 Appearances after coating adhesion test on Zn-Ni alloy electroplated steel

direction coating weight distribution and Ni content of RIVER HI-ZINC are both uniform. **Figure 3** indicates that variations in Ni content among Zn-Ni alloy electroplated steel coils are as small as ±1%, and overall Ni content consistently falls within the range of 10.5 to 12.5%.

3 Performance of RIVER HI-ZINC

Basic properties of RIVER HI-ZINC as a precoated steel for automotive use were examined in comparison with those of electrogalvanized steel (EG), galvannealed steel (GA), and cold rolled steel (CR).

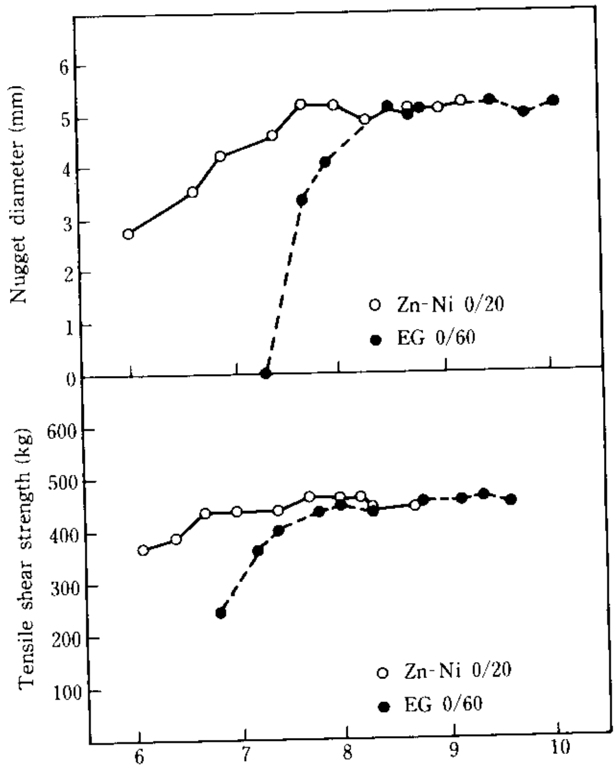
3.1 Coating Adhesion

Automotive steel sheets are subjected to various types of working, beginning with press forming. Therefore, examination was made of the coating adhesion of RIVER HI-ZINC when worked. The test was conducted by working SPCE steel sheets with a 20 g/m^2 coating weight and a 0.8 mm thickness into the shapes shown in **Photo 1** and then by attempting to peel off the coating with adhesive tape. RIVER HI-ZINC showed no peeling-off after such rigorous working as a 180° bending and cylindrical cup draw tests, thereby indicating excellent adhesion.

3.2 Spot Weldability

The spot weldability of RIVER HI-ZINC was examined by comparing it with that of EG. **Figure 4** shows results in the available welding current range. RIVER HI-ZINC can be spot-welded at a wider range of current values than EG, and gives sufficient weld strength even at comparatively lower currents.

Figure 5 shows the results of a continuous spot weldability test. Compared with EG which became unweldable after about 3 000 welding points, RIVER HI-ZINC, even after welding at 5 000 points, showed no change from the beginning of welding either in nugget diameter or tensile shear strength. The reasons for the superiority of RIVER HI-ZINC to Zn coated steel in spot-weldability are as follows:



Welding condition
Electrode : Cu-Cr
Tip diameter : 4.8mm
Electrode force: 200kg
Weld time : 12cycles/s

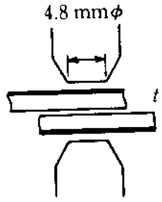
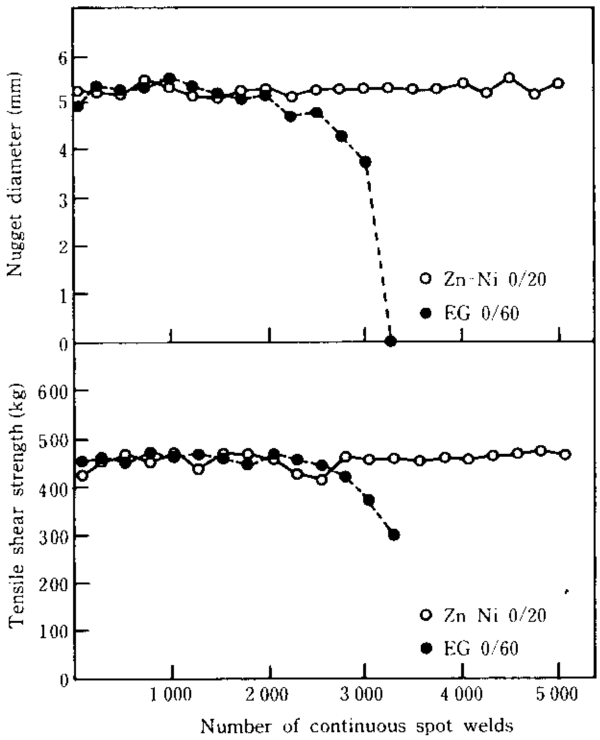


Fig. 4 Effects of welding current on nugget diameter and tensile shear strength

(1) Coating hardness is higher.
(2) Melting point of the coated layer is higher.
(3) Coating weight is lower.

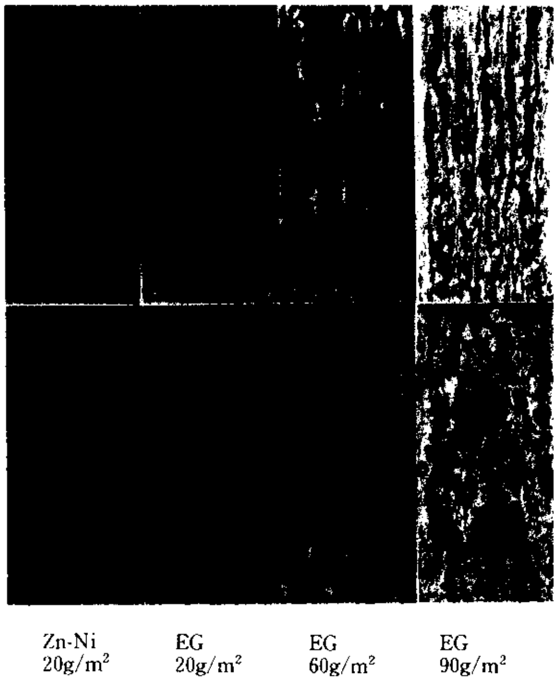
The electrode tip, therefore does not alloy easily, and loss is small.



Welding condition:

Electrode	: Cu-Cr
Tip diameter	: 4.8mm
Electrode force	: 200kg
Welding current	: 8.0kA(Zn-Ni)
	: 8.5kA(EG)
Weld time	: 12cycles/s

Fig. 5 Continuous spot weldability



Upper : Flat sample
Lower: Erchsen sample(height 9mm)

Photo 2 Surface appearances of various precoated steels after 340 h salt spray test

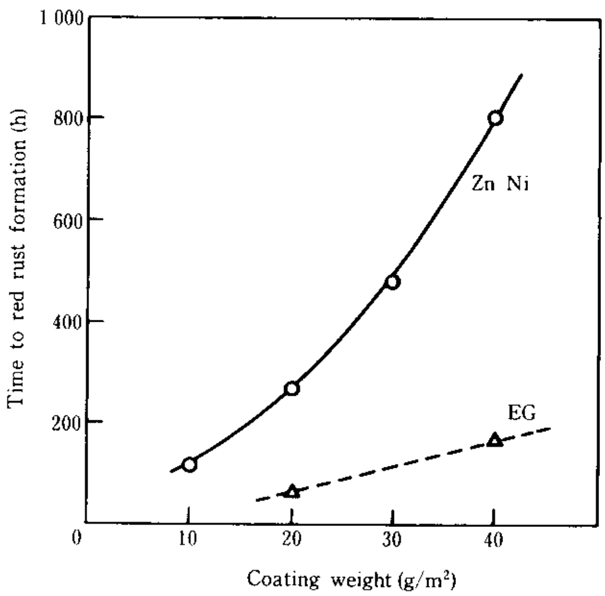


Fig. 6 Effect of coating weight on corrosion resistance in salt spray test

3.3 Corrosion Resistance in Nonpainted State

Photo 2 shows the corrosion condition of nonpainted RIVER HI-ZINC after the salt spray test (JIS Z2371), comparison with that of EG. RIVER HI-ZINC developed little red rust, although its coating weight was as small as 20 g/m^2, thereby showing corrosion resistance equal or superior to that of EG with a coating weight of 90 g/m^2. **Figure 6** shows the relation between coating weight and red rust formation time. RIVER HI-ZINC shows four times or more the corrosion resistance of EG, coating weight being equal.

Upon an X-ray diffraction analysis of corrosion products after a 24-hour salt spray test, only $ZnCl_2 \cdot 4Zn(OH)_2$ was detected in RIVER HI-ZINC but $ZnCl_2 \cdot 4Zn(OH)_2$ and a large quantity of ZnO were detected in EG. The $Zn(OH)_2$ has low electric conductivity and is said to be effective in reducing oxidation-reduction reactions on the coated surface.[13] The excellent corrosion resistance of RIVER HI-ZINC is considered attributable to the following reasons:

(1) Corrosion products exhibit excellent protective action.

(2) Since the coated layer is a γ single phase, the micro-cells in the coated layer which occur in multiphases

do not readily form.

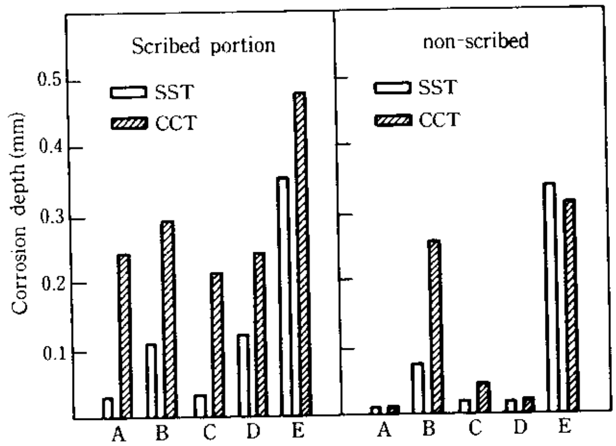
(3) The potential of the coated layer is less noble than that of Fe and has an appropriate sacrificial effect.

Further, in addition to the contribution by Ni to the formation of $Zn(OH)_2$, which itself acts to inhibit corrosion, Ni is considered to play a role as a mechanism[14] by which priority dissolution of zinc forms a concentrated Ni surface layer during corrosion. This layer acts as a protective coating.

3.4 Corrosion Resistance after Painting

When one-side coated steel sheets are used for automotive outer panels, the coated surface is used as the inner side where electrodeposited primer is difficult to apply. Furthermore, when perforation corrosion of the body is considered, a problem is posed by corrosion in enclosed structural parts, where the electrodeposited primer also cannot be adequately applied. Taking into consideration these use requirements, the corrosion resistance of RIVER HI-ZINC was examined in comparison with other steel sheets under conditions of 5 μm cathodic electrodeposition.

Table 1 shows the test conditions, and **Photo 3** shows appearances after the corrosion test. RIVER HI-ZINC developed comparatively smaller blisters and less red rust, indicating corrosion resistance equal or superior to that of GA 60 g/m^2. **Figure 7** shows thickness reductions after the corrosion test. In the salt spray test, RIVER HI-ZINC showed the least thickness reduction, both at scribed and nonscribed portions, indicating excellent corrosion resistance, equal to that of EG 90 g/m^2. In the cyclic corrosion test, RIVER HI-ZINC also showed excellent corrosion resistance, and all other precoated steel sheets show greater thickness reductions at scribed portions. These facts indicate that RIVER HI-ZINC is inactive in comparison with Zn, so that the development of corrosion under the painted film is retarded. Its corrosion resistance is comparable to that of EG 90 g/m^2, and similar to the test results for nonpainted sheets.



A : Zn-Ni 20 g/m^2　D : GA 60 g/m^2
B : EG 20 g/m^2　E : CR g/m^2
C : EG 90 g/m^2

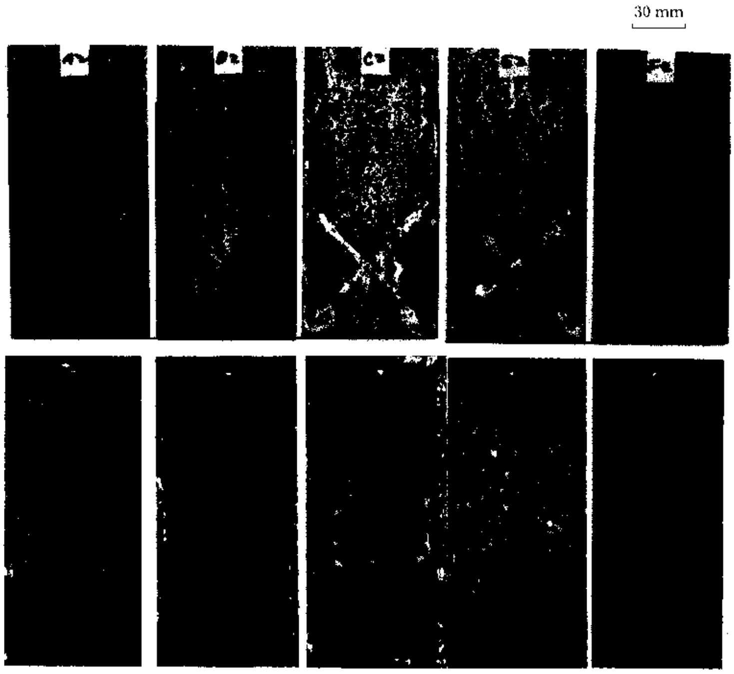
Fig. 7 Corrosion depth of various precoated specimens with cathodic electrodeposition painting after 2500 h salt spray test and 200 cycles in cyclic corrosion test

4 Development of RIVER HI-ZINC SUPER

Before an automotive body is painted by electrodeposition method, phosphate treatment is performed. The quality of the phosphate film formed by this pretreatment is one of the important factors in corrosion resistance and coating adhesion after painting. Since the alloy coating on RIVER HI-ZINC contains mainly Zn, this phosphate treatment forms a hopeite film [$Zn_3(PO_4)_2 \cdot 4H_2O$] a needle-like phosphate coating. It is generally known that hopeite has certain disadvantages, for example, it is more soluble in alkaline solutions than the phosphophyllite [$Zn_2Fe \cdot (PO_4)_2 \cdot 4H_2O$] which develops on cold rolled sheets. Further, hopeite is lower in after-painting corrosion resistance, and also shows poorer wet adhesion with 3-coat films.[15] The phosphate coating formed on RIVER HI-ZINC consists of hopeite and as a result, this material has poor "wet adhesion" which

Table 1 Corrosion test methods

		Conditions
Specimen preparation	Specimens	Zn-Ni 20 g/m^2, GA 60 g/m^2 EG 20, 90 g/m^2, CR
	Pretreatment	Dip type phosphate treatment (Gr#SD2000)
	Electrodeposition	Cathodic electrodeposition: 5 μm (Power Top U-30) Baking: 180°C, 20 min
	Specimen shape	Plain sheet with cross scribe
Corrosion test	Salt spray test	5% NaCl solution, 35°C
	Cyclic corrosion	Salt spray (35°C, 4 h)—dry with hot air (50°C, 2 h)—humidify 50°C (95% RH, 4 h)—freeze (−5°C, 2 h); One cycle 12 h



Zn-Ni 20g/m^2 EG 20g/m^2 EG 90g/m^2 GA 60g/m^2 CR

Upper : After SST for 1000 h
Lower : After CCT for 200 cycles

Photo 3 Appearances of cathodic electrodeposited (5 μm) steels after salt spray and cyclic corrosion tests

refers to the adhesion of paint film after immersion in hot water. In seeking to improve the phosphate coating of RIVER HI-ZINC, therefore, attention was focused on the fact that the phosphate coatings on cold rolled sheets have excellent wet adhesion. Consequently, a further Fe-based coating was given to RIVER HI-ZINC. It was found, however, that a pure Fe coating gives insufficient improvement, but a Fe-P coating of about 2 g/m^2 which contains P of 0.5% or below improves phosphatability significantly. The Fe-P coating is obtained by electroplating from a bath consisting of a well-known Fe plating solution to which a micro-quantity of a phosphatic compound has been added.

The following describes the performance of an Fe-P/Zn-Ni double-layer alloy coated steel sheet, the RIVER HI-ZINC with improved phosphatability, which is termed RIVER HI-ZINC SUPER, in comparison with the performance of Zn-Ni alloy coated steel sheets and steel sheets with pure Fe upper-layer coating.

4.1 Phosphatability

It is well known that the phosphatability of steel sheets varies with phosphating methods and solutions. Recent improvements in the phosphating solution are noticeable, and a phosphating solution which can give a compactly-packed phosphate coating to Zn-Ni alloy coated steel sheets is being developed. At automotive makers, however, dip- and spray-type processing methods are both used, and a precoated steel is desired which shows excellent phosphatability regardless of the processing method or solution.

In view of this, the authors examined phosphatability, using commercially available spray-type (Gr16NC) and dip-type (Gr SD2000) phosphating solutions. **Photo 4** shows scanning electron micrographs (SEM) of phosphate coatings; **Fig. 8** shows coating characteristics. The Zn-Ni coated steel sheet develops needle-like hopeite and does not show phosphophyllite growth with any phosphating solution. The Fe/Zn-Ni coated steel sheet develops a phosphophyllite coating consisting of fine crystals in dip-type processing, but develops a hopeite coating with a larger crystal grain size of 13 μm in spray-type processing. On the other hand, the RIVER HI-ZINC SUPER Fe-P/Zn-Ni steel sheet develops a very fine phosphophyllite coating having a crystal grain size of 3–5 μm with both the dip- and spray-type processing solutions. The reason that it is possible to obtain compactly-packed phosphate coatings with Fe-P coated steel sheets is considered to be that the micro-quantity

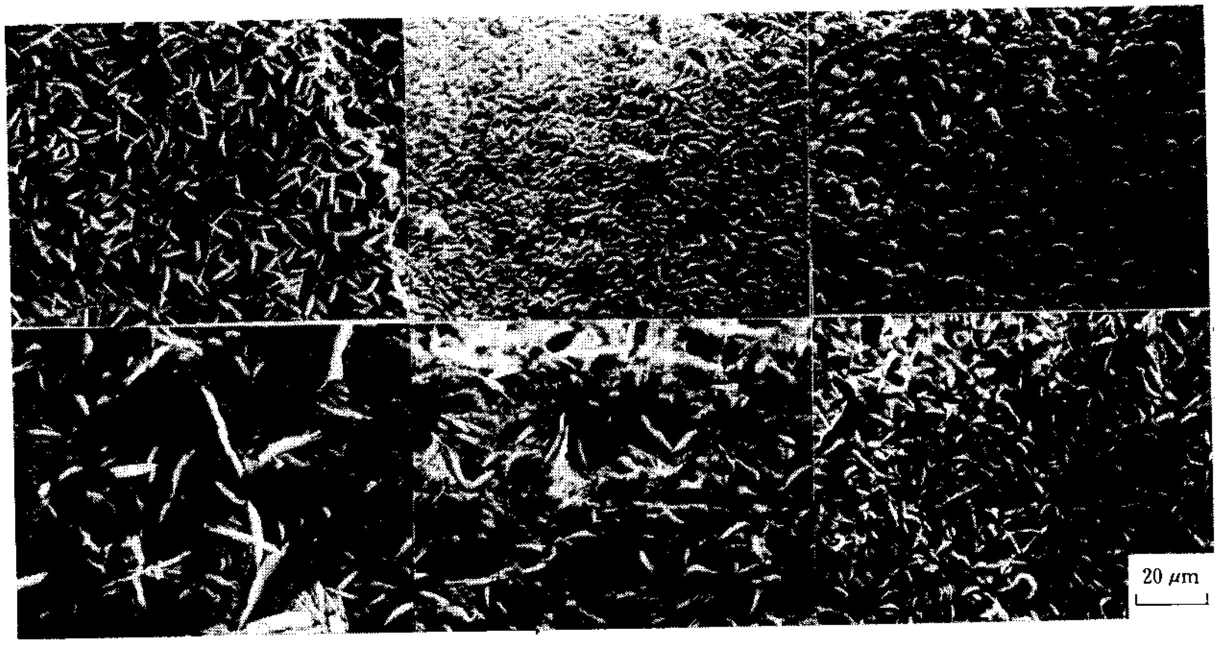


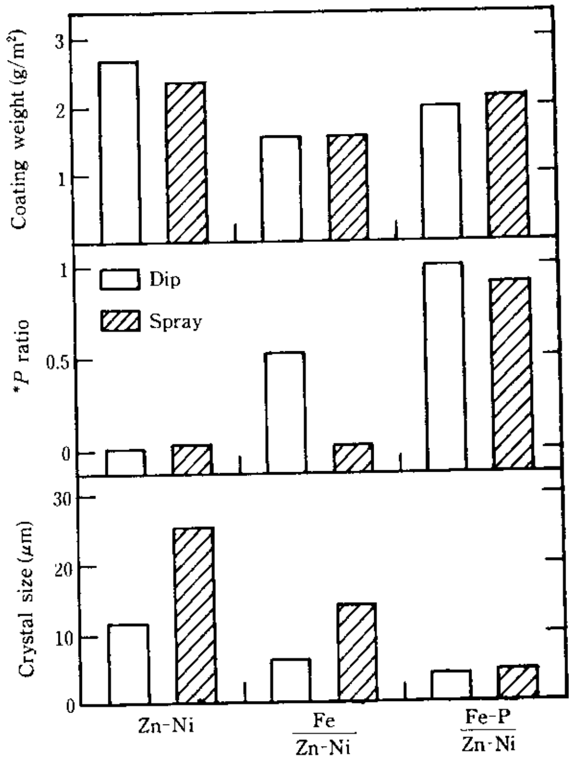
Photo 4 Scanning electron micrographs of phosphate coatings

of P which is uniformly dispersed in the Fe coatings activates the surface and enhances the reaction with the phosphating solution.

Figure 9 shows the relation between Fe-P upper coating weight and the *P* ratio [phosphophyllite/(phosphophyllite + hopeite)]. When the dip-type phosphating solution is used, the *P* ratio becomes 1; that is, phosphophyllite coatings will be obtained if the Fe-P coating weight is 0.7 g/m^2 or above. However, when the spray-type phosphating solution is used, the *P* ratio will approximate to 1 only if the coating weight is 2.1 g/m^2. This indicates that to obtain satisfactory phosphate coatings regardless of the phosphating solution used, the Fe-P upper coating weight must be 2 g/m^2 or above.

4.2 Wet Adhesion

Wet adhesion is evaluated by the following procedure: after being given 3-coat films consisting of cathodic electrodeposition, a sealer coat, and a top coat, the steel sheet is immersed in deionized water at a temperature of 50°C for 240 h and, after being removed from the water, immediately subjected to a 2 mm square grid-patterned adhesive tape peeling-off test. Wet adhesion is governed by the phosphating treatment and coating thickness, and the result of the peeling-off test show considerable variation. When cathodic electrodeposition painting is applied to Zn-coated steel sheets, results generally tend to be unsatisfactory. This tendency has been attributed to the fact that the phosphate coating



**P* ratio = phosphophyllite/(phosphophyllite + hopeite)

Fig. 8 Phosphatability of single and double layered Zn-Ni alloy electroplated steel

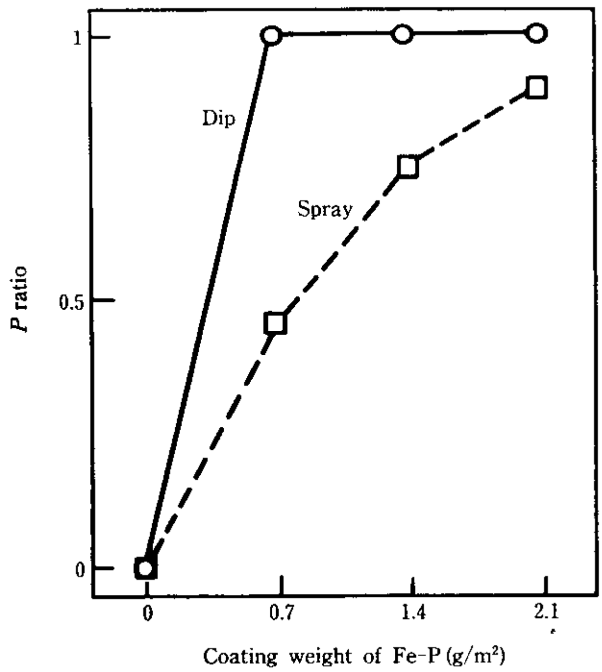


Fig. 9 Effect of Fe-P upper coating weight on P ratio of double layered Zn-Ni alloy electroplated steel

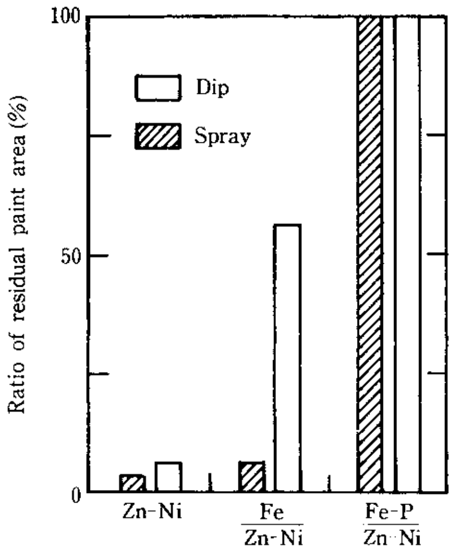


Fig. 10 Wet adhesion of 3-coat painted panels

consists of hopeite, which, as mentioned above, is relatively soluble in alkaline solutions,[16] but the actual reason is not yet known.

Figure 10 shows the results of the wet adhesion test. The Zn-Ni steel sheet developed peeling-off of paint films and showed poor adhesion with both dip and spray treatments. With the Fe/Zn-Ni steel, no improvement was observed after spray treatment, and even after dip treatment, only about 60% of coating remained intact. However, with the Fe-P/Zn-Ni steel sheet, satisfactory,

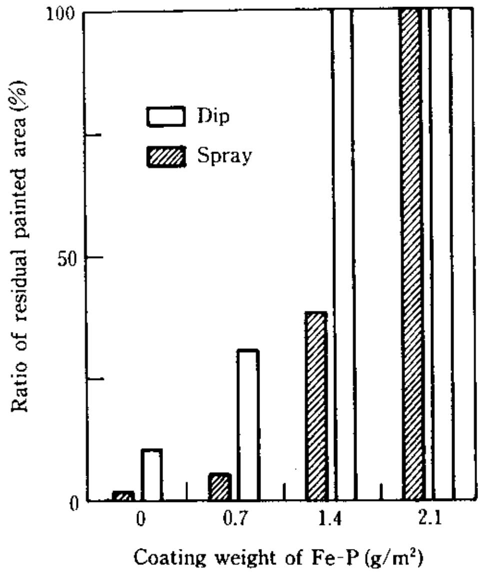


Fig. 11 Effect of Fe-P upper coating weight on wet adhesion of double layered Zn-Ni alloy electroplated steel

stabilized adhesion was obtained regardless of whether the dip or spray treatment method was used.

Figure 11 shows the relation between wet adhesion and Fe-P upper coating weight. As can be clearly seen when compared with Fig. 9, wet adhesion and the P ratio show comparatively good correlation. When the dip-type phosphate solution is used, peeling-off of paint films will rarely occur if the coating weight is 1.4 g/m^2 or above. If the spray type is used, a coating weight of 2.1 g/m^2 is necessary. The reason for this satisfactory wet adhesion with Fe-P coated steel sheet is considered to be the closely-packed fine phosphophyllite coating obtained.

4.3 Corrosion Resistance after Phosphating Treatment

It has been found that an upper-layer Fe-P coating will improve phosphatability and wet adhesion, but attention must also be given to the effect of upper-layer Fe-P coating on the excellent corrosion resistance of Zn-Ni alloy coatings mentioned in Secs. 3.3 and 3.4. Therefore, the authors investigated the corrosion resistance of unpainted bare steel sheets before and after phosphating. The results are shown in **Fig. 12**. Before phosphating, the Fe-P/Zn-Ni steel sheet tends to show slightly less corrosion resistance than the Zn-Ni steel sheet, but conversely, after phosphating, the Fe-P/Zn-Ni steel sheet shows greater corrosion resistance, indicating that phosphating improves its corrosion resistance.

4.4 Adaptability to Automotive Exposed Panels

When steel sheets are used for exposed side of outer panels of automotive bodies as a countermeasure

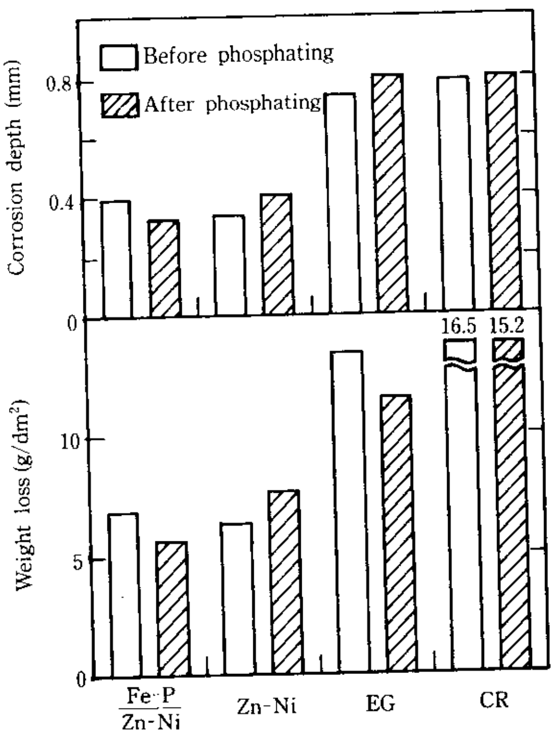


Fig. 12 Corrosion resistance of various precoated steels after 1500 h salt spray test

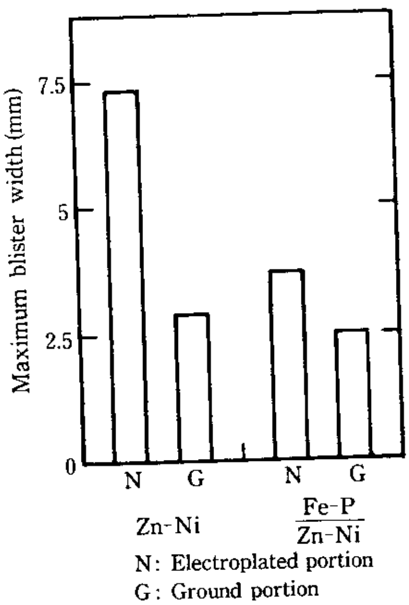


Fig. 13 Effect of grinding on maximum blister width from cross scribe after 840 h salt spray test (CED 20 μm)

against cosmetic corrosion, they must satisfy such requirements as the followings:

(1) Resistance against crater-form paint defects

(2) Post-painting corrosion resistance at the location of minor repairs

(3) Corrosion resistance after 3-coat painting.

When Zn-Ni alloy coated steel sheets are painted by

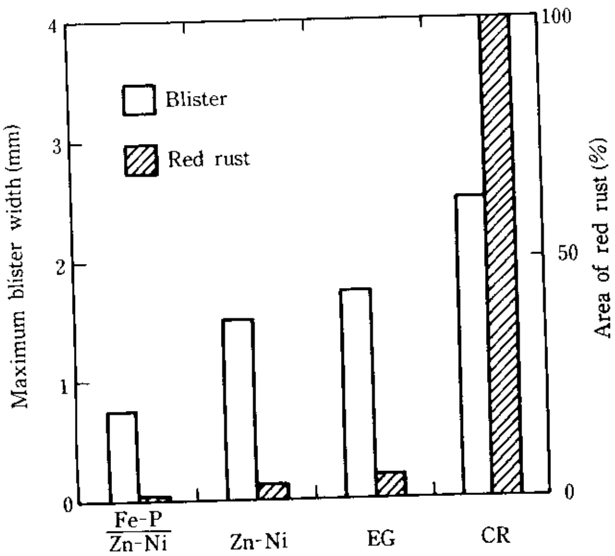


Fig. 14 Maximum blister width and area of red rust of various precoated specimens with 3-coat painting in cyclic corrosion test (10 cycles)

cathodic electrodeposition, cratering will occur as electrodeposition voltage rises. Therefore, conditions for the occurrence of cratering were investigated using a cathodic electrodeposition paint "Power Top" (produced by Nippon Paint Co., Ltd.) while varying voltage. The Zn-Ni sheet developed slight cratering at 250 V, and the number of craters increased as the voltage was raised. The Fe-P/Zn-Ni sheet, however, as with cold rolled sheet, did not develop cratering even at 350 V, thereby showing satisfactory resistance against cratering defects.

When minor defects are found on the exposed auto body panels after press-forming, they are frequently corrected by grinding. Therefore, after grinding only the center part of a test specimen, phosphating and 20 μm cathodic electrodeposition were performed, in order to investigate blistering in cross-scribed portions by the salt spray test. As shown in **Fig. 13**, blister width in the ground portion (G, in the figure) of the Zn-Ni sheet is small, whereas that on the coated surface (N, in the figure) is relatively large. The ground surface developed red rust, probably due to exposure of the cold rolled surface. On the other hand, both the coated and ground surfaces of the Fe-P/Zn-Ni sheet developed only small blisters, showing satisfactory results.

To investigate cosmetic corrosion resistance, after 3-coat painting the samples were scribed with an arrowhead and subjected to a cyclic corrosion test consisting of a salt spray test (one day), wetting (5 days), followed by standing (one day). **Figure 14** shows the results of examination of corrosion at scribed portions. The blister width on the Fe-P/Zn-Ni sheet is smaller than that on Zn-Ni and EG sheets, with no occurrence of red rust. On the other hand, CR developed red rust and relatively wide blisters. The excellent corrosion resistance of Fe-P/Zn-Ni is considered attributable to the facts that closely-packed phosphate coating formed, ensuring paint adhesion and the Zn-Ni coating exists in the under-layer.

As can be seen clearly from these results, Fe-P/Zn-Ni RIVER HI-ZINC SUPER has excellent cosmetic corrosion resistance properties, and can be considered suitable for application in exposed auto body panels.

5 Conclusions

Kawasaki Steel developed Zn-Ni alloy electroplated steel sheets (RIVER HI-ZINC) and double-layer-type Fe-P/Zn-Ni alloy coated steel sheets (RIVER HI-ZINC SUPER) as thin-coated precoated steel sheets with excellent corrosion resistance for automotive applications.

Main points concerning these two steels can be summarized as follows:

(1) The Zn-Ni alloy electroplated layer consisting of a γ single phase coating with an Ni content of about 12% shows corrosion resistance four times or above that of a pure Zn coated layer, coating weight being equal.

(2) The Zn-Ni alloy electroplated steel sheets have excellent coating adhesion, and can be welded at wider current ranges than heavily Zn-electroplated steel sheets, and have improved continuous spot weldability.

(3) With application of an upper layer Fe-P coating of 2 g/m^2, a close-packed fine phosphophyllite coating can be obtained with either spray- or dip-type phosphate processing, thereby yielding excellent wet adhesion after 3 coat painting.

(4) Fe-P coating prevents crater-form defects during cathodic electrodeposition painting, and inhibits the occurrence of red rust and blisters at scratched portions of 3-coat painted sheets.

These two types of Zn-Ni alloy electroplated steel sheets are expected to find wide use as precoated steel sheets for the inner and outer automotive body surfaces, in view of the properties required for various applications.

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