KAWASAKI STEEL TECHNICAL REPORT
No.12 (July 1985)
Special Issue on Hot-and Cold-rolled Steel Sheets

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Synopsis :
"Zincrometal-KII", a new organic composite-coated steel sheet with high corrosion resistance has been developed, in which improvements are made in coating adhesion during forming and weldability of conventional zinc rich paint coated steels for automobiles. It consists of a specially improved thinner zinc rich paint layer on a thin Zn-Ni alloy electroplated layer, and has been improved according to the following ideas: (1) Corrosion resistance is improved by the complex effects of a Zn-Ni alloy electroplated layer and zinc rich paint layer which contains zinc potassium chromate as an anti-corrosive pigment. (2) Better coating adhesion during forming is obtained by the addition of MoS2 as a lubricant agent. (3) Weldability is improved by the thinner paint layer. Therefore, coating adhesion during forming and corrosion resistance are superior than conventional paint coated steels, and the number of welds in continuous spot welding is more than 5000.

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Development of Organic Composite-Coated Steel Sheet "Zincrometal-KII" with High Corrosion Resistance[*1]

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"Zincrometal-KII," *a new organic composite-coated steel sheet with high corrosion resistance has been developed, in which improvements are made in coating adhesion during forming and weldability of conventional zinc rich paint coated steels for automobiles.*

It consists of a specially improved thinner zinc rich paint layer on a thin Zn–Ni alloy electroplated layer, and has been improved according to the following ideas:

(1) Corrosion resistance is improved by the complex effects of a Zn–Ni alloy electroplated layer and zinc rich paint layer which contains zinc potassium chromate as an anti-corrosive pigment.

(2) Better coating adhesion during forming is obtained by the addition of MoS_2 *as a lubricant agent.*

(3) Weldability is improved by the thinner paint layer.

Therefore, coating adhesion during forming and corrosion resistance are superior than conventional paint coated steels, and the number of welds in continuous spot welding is more than 5 000.

1 Introduction

In snow-belts in North America, Canada, and elsewhere, large amounts of salt are spread on streets and roads to prevent icing in winter. The resulting corrosion of automobile bodies, termed "salt damage", has become a major problem.[1,2] Coated steel sheets are used to prevent this car-body corrosion, and the demand for such steel sheets is increasing every year.

Besides zinc- and zinc-alloy-coated steel sheets, organic coated steel sheets[3-6], as exemplified by Zincrometal, a coated steel sheet, are used in automobiles. Organic coated steel sheets have better corrosion resistance in an unpainted condition than such galvanized steel sheets.[5,7] Steel sheets with excellent corrosion resistance in an unpainted state are required for hems and enclosed portions,[8-10] where treatment by zinc phosphating or electrodeposition painting process after car-body assembling is inadequate, and where salt and muddy water tend to collect. As can be seen in **Fig. 1**, organic coated steel sheets are widely used for this application.[11] With conventional organic coated steel sheets, however, the coating is apt to exfoliate during press forming, generating pimples, and the wear of the electrode tip during continuous spot welding is excessive. Thus, problems arise in workability and ease of operation during the assembly of car bodies. Furthermore, the corrosion resistance of steel under damaged coating is insufficient. Therefore, the development of surface-treated steel sheets with high corrosion resistance in which these drawbacks are eliminated is highly desirable.

To meet this requirement, Kawasaki Steel developed a new organic composite-coated steel sheet with high corrosion resistance **"Zincrometal-KII"** (hereafter called "KII").

[*1] Originally published in *Kawasaki Steel Giho*, **16**(1984)4, pp. 320–327

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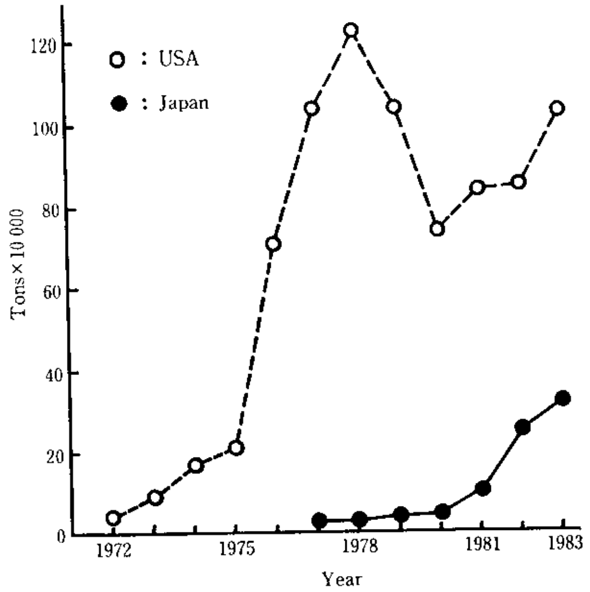


Fig. 1 Production tonnage of Zincrometal[11] .

2 Development of Organic Composite-Coated Steel Sheet with High Corrosion Resistance (KII)

In developing this highly corrosion-resistant organic composite-coated steel sheet, an investigation was made into the improvement of zinc-rich coating and the applications of coated steel as base metal.

2.1 Experimental Methods

2.1.1 Test material

The production method for the organic composite-coated steel sheet tested is shown in **Fig. 2**.

2.1.2 Test methods

The test methods used for evaluating corrosion resistance, coating adhesion, and weldability are shown schematically in **Table 1**. Corrosion resistance was evaluated by two cyclic corrosion tests, the CCT-A method, applicable to automobile parts which dry after exposure to salt water and muddy water, and the CCT-B method, applicable to parts which remain wet after salt water and muddy water collect. To conduct a simple comparative investigation of coating adhesion during press forming, the specimen was worked by cylindrical cup drawing, cellophane adhesive tape was then applied to the drawn part, and the exfoliated coating weight was measured after the tape was removed. Weldability was evaluated by a type of accelerated welding test method which employs a radius-type electrode that is rapidly consumed during continuous spot welding.

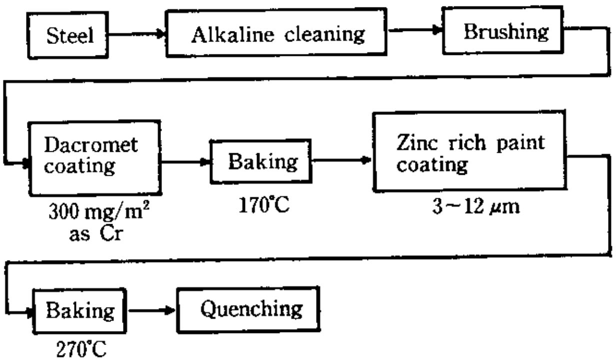


Fig. 2 Producing process of organic composite-coated steels

2.2 Results and Discussion

2.2.1 Effects of thickness of zinc-rich film

With organic coated steel sheets, the elongation of the organic coating is small because the coating contains a large amount of metallic powders such as zinc powder. In addition, the shrinkage strain after coating formation is large because of the coating thickness. Therefore, the coating is apt to exfoliate during press forming. Moreover, weldability is inadequate because of high coating resistance.[5,7] Therefore, the effect of zinc-rich coating thickness in the range of 3 to 12 μm on improved formability and weldability was examined.

The effects of the zinc-rich coating thickness on each kind of performance are shown in **Fig. 3**. Formability and weldability improve as coating thickness decreases to 4.5 μm. Further improvement cannot be observed below this value. Corrosion resistance also decreases with decreasing coating thickness. However, the coating thickness dependence is a little larger in the CCT-B cor-

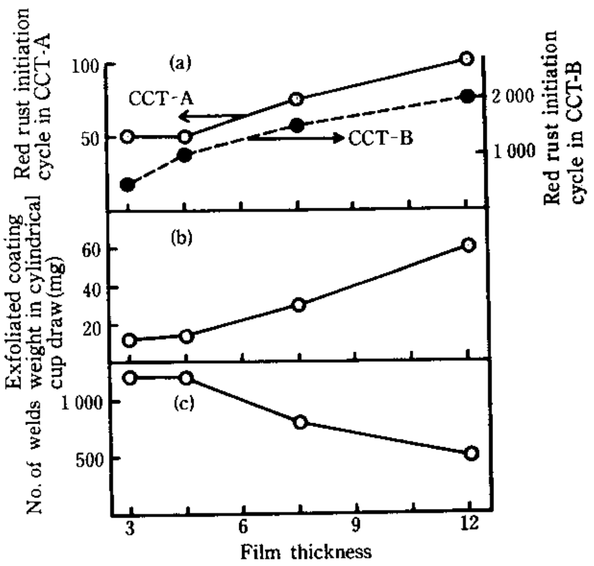
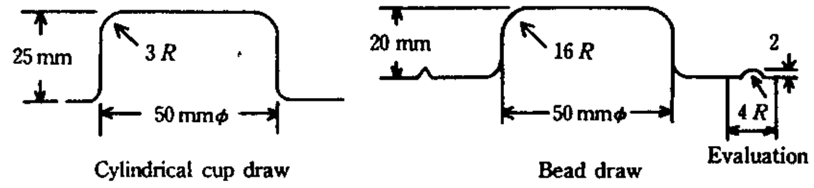
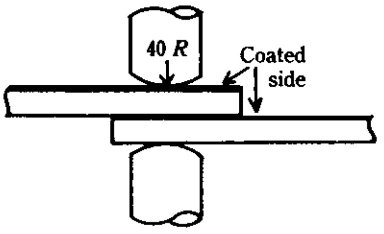


Fig. 3 Effect of thickness of zinc-rich coating on (a) corrosion resistance (b) coating adhesion and (c) weldability

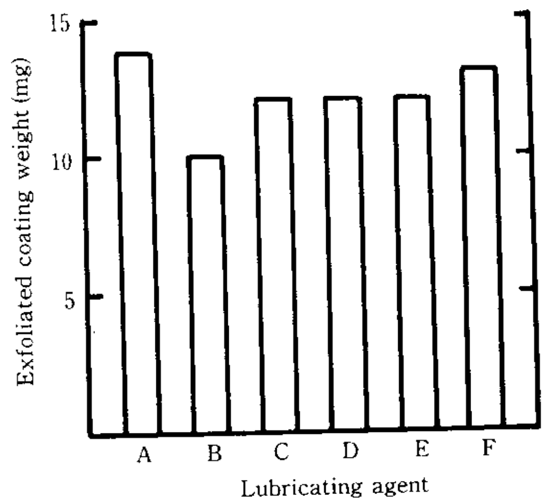
Table 1 Testing method

Properties	Testing methods
Corrosion resistance	Specimens → Cross Cutting (in lower half side) → Cyclic corrosion tests (CCT-A* and CCT-B**) → Evaluation by red rust initiation cycle at which the red rust is spread over 10% of the total area * CCT-A: → Salt spray 5% NaCl, 40°C 4h → Drying 60°C 2h → Humidity 50°C, 95%RH 2h → ** CCT-B: → Immersion 5% NaCl, 40°C 7.5 min → Humidity 40°C, 95%RH 15 min → Drying 60°C 7.5 min →
Coating adhesion in drawing	 Evaluation: Exfoliated coating weight peeled off with adhesive tape
Weldability	Welding condition: Shape of electrode: R type (40 R) Electrode force : 200 kg Welding time : 10 cycle Holding time : 15 cycle Welding current : 8.5 kA Sheet thickness : 0.8 mm Evaluation: Number of welds by continuous welding where the joint strength is more than 400 kg 

rosion resistance test than in CCT-A. Therefore, 4.5 μm is the optimum zinc-rich coating thickness to maximize coating adhesion and weldability while minimizing the decrease in corrosion resistance. That is to say, the amount of exfoliated coating after forming decreases to about 25% and the number of continuous spot welds approximately doubles if the coating thickness of KII is reduced to about one-third that of conventional organic coated steel sheets. Red rust initiation time in the corrosion resistance test decreased to about half. The apparent reason is that the barrier effect of the coating decreases due to the decrease in coating thickness, indicating that it is necessary to improve the zinc-rich coating in order to compensate for the decrease in barrier effect.

2.2.2 Effect of addition of lubricant agent to zinc-rich coating

It was expected that the coating exfoliation during forming would decrease further if the lubricating property of the zinc-rich coating was improved, and an investigation was made into the addition of a lubricant agent to Zincromet. The lubricant agents examined were solid lubricatant agents in the form of powders of molybdenum disulfide, boron nitride, polyethylene tetrafluoride, carbon fluoride, graphite, etc. which are known to give an excellent lubricating performance and to be stable both thermally and chemically.[12] The effect of a 1% addition of lubricant agent to the zinc-rich coating is shown in **Fig. 4**. Although all these lubricant agents yielded improvements in the coating adhesion, molybdenum disulfide was most effective. An investigation was therefore conducted into the effect of various amounts of molybdenum disulfide, with the results shown in **Fig. 5**. A remarkable improvement in coating adhesion was observed at 0.4 wt% molybdenum disulfide. However, further improvements were not observed above this value. On the other hand, corrosion



A :	No addition	D :	Fluorinated carbon
B :	MoS_2	E :	Poly tetrafluoroethylene
C :	Boron nitride	F :	Graphite

Fig. 4 Effect on coating adhesion of the addition of a lubricating agent to zinc-rich coating

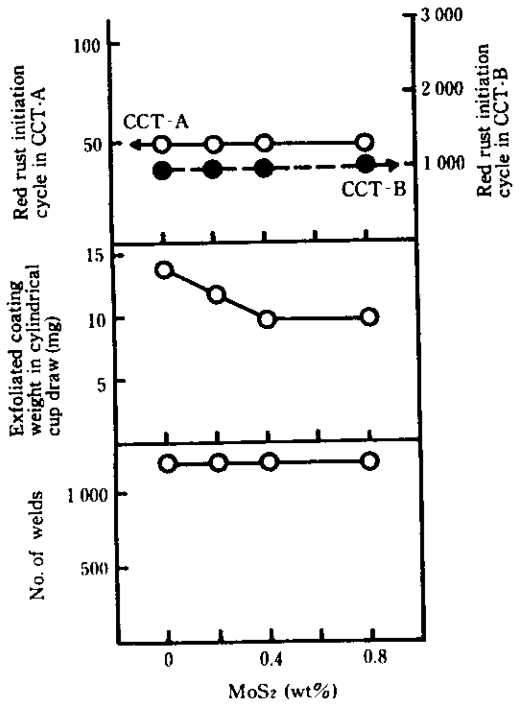


Fig. 5 Effect of the addition of MoS_2 to zinc-rich coating on (a) corrosion resistance, (b) coating adhesion, and (c) weldability

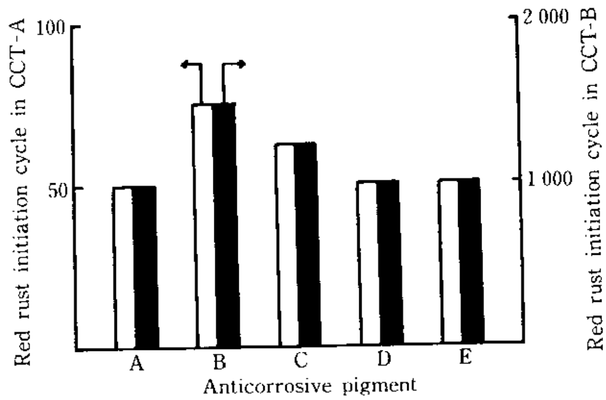
resistance and weldability showed no change at up to 0.8 wt%. It may be understood from the foregoing that 0.4 wt% is the optimum value.

2.2.3 Effect of addition of anticorrosive pigment to zinc-rich coating

The addition of anticorrosive pigment to the zinc-rich coating was examined as a measure to prevent decrease in corrsion resistance resulting from a decrease in coating thickness.

The anticorrosive pigments examined consisted of four kinds of soluble anticorrosive pigments used as paint additives and known to have excellent anticorrosive effects[13-15]. They were zinc potassium chromate ($K_2O \cdot 4CrO_3 \cdot 4ZnO \cdot 3H_2O$), zinc tetraoxy chromate ($ZnCrO_4 \cdot 3H_2O$), strontium chromate ($SrCrO_4$), and aluminum dihydrogen tripolyphosphate ($H_2AlP_3O_{10} \cdot 2H_2O$).[16] The effect of addition of 5 wt% of anticorrosive pigment to the zinc-rich coating is shown in **Fig. 6**. It can be seen from this figure that zinc potassium chromate and strontium chromate are effective in improving corrosion resistance and that the addition of zinc potassium chromate, especially, has a great effect. The remaining two anticorrosive pigments; i.e. zinc tetraoxy chromate and aluminum dihydrogen triployphosphate, had virtually no effect. Zinc potassium chromate and strontium chromate contain 6 to 8% of water-soluble components, and zinc tetraoxy chromate and aluminum dihydrogen tripolyphosphate of less than 1%. It was considered that water-soluble chromic acid ions render the steel sheet surface inactive and thereby increase the anticorrosion effect.

An investigation was made into the amounts of zinc potassium chromate with the best anticorrosion effect. The results of this investigation are shown in **Fig. 7**. Corrosion resistance tended to improve as the amount of zinc potassium chromate was 4 wt% or more. However, weldability decreased markedly when the content reached 6 wt%. Film adhesion did not change with amounts up to 8 wt%. It was found, therefore, that the optimum amount of zinc potassium chromate to improve corrosion resistance without deteriorating for-



A : No addition
B : ZPC ($K_2O \cdot 4CrO_3 \cdot 4ZnO \cdot 3H_2O$)
C : $SrCrO_4$
D : ZTO ($ZnCrO_4 \cdot 4Zn(OH)_2$)
E : $H_2AlP_3O_{10} \cdot 2H_2O$

Fig. 6 Effect on corrosion resistance of anticorrosive pigment addition (5 wt%) to zinc-rich coating (left-hand bars, CCT-A; right-hand bars, CCT-B)

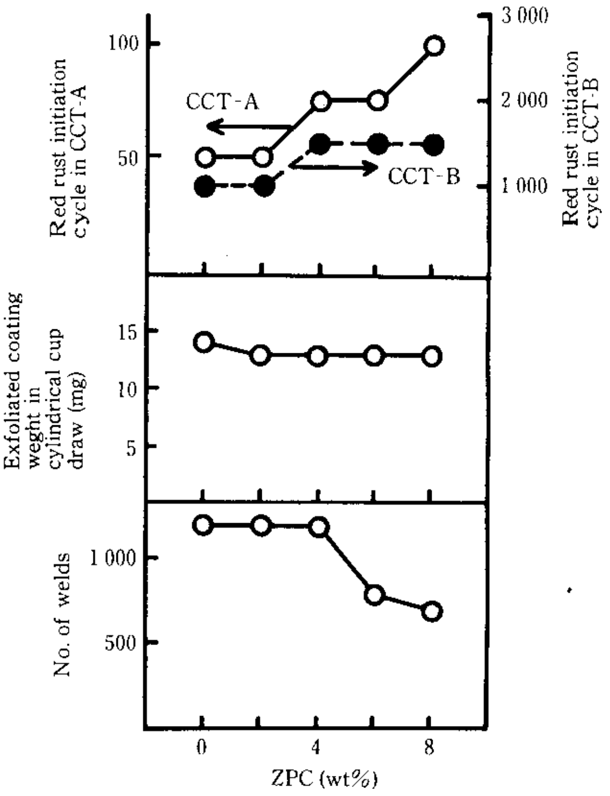


Fig. 7 Effects of the addition of ZPC to zinc-rich coating on (a) corrosion resistance (b) coating adhesion, and (c) weldability

mability was 4 wt%. Nevertheless, the corrosion resistance of KII is inferior to that of conventional organic coated steel sheets. It was suggested that it was not possible to compensate for the deterioration in corrosion resistance due to a decrease in the coating thickness simply by adding anticorrosive pigment to the zinc-rich primer, and that the use of a zinc-coated base metal to provide sacrificial protection would be necessary.

2.2.4 Effect of base metal coatings

After Decromet coating, the above-mentioned improved zinc-rich coating was applied to zinc-coated steel sheets to a thickness of 4.5 μm and the effects of base metal coatings were investigated. Electrogalvanized steel sheets (EG20, coating weight 20 g/m^2), galvanealed steel sheets (GA45, coating weight 45 g/m^2), Zn-Fe alloy electroplated steel sheets (EGA20, coating weight 20 g/m^2) and Zn-Ni alloy electroplated steel sheets (EZN20, coating weight 20 g/m^2) were used as base metals.

Effects of base metal coatings on corrosion resistance are shown in **Fig. 8** and **9**. These results reveal that the precoated steel sheet using EZN20 is best in both CCT-A and CCT-B corrosion resistance tests. A base metal effect that compensates for the deterioration in corrosion resistance due to a decrease in the zinc-rich coating thickness was not observed with other zinc-coated steel sheets (EG20, EGA20 and GA45). Although it provides excellent sacrificial protection, EG20 is susceptible to blistering, and red rust is apt to form from blisters. In the case of EGA20, it is thought that red rust is apt to occur because the coating layer contains iron. In the test CCT-B, red rust formed more slowly on GA45 than on Zincrometal. In the CCT-A test, however, the formation of red rust in GA45 occurs as early as in EGA20.

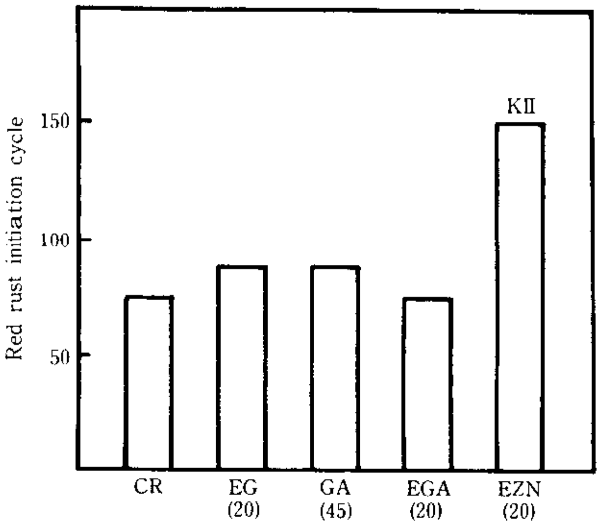


Fig. 8 Effect of zinc-rich coating base metals on corrosion resistance in CCT-A

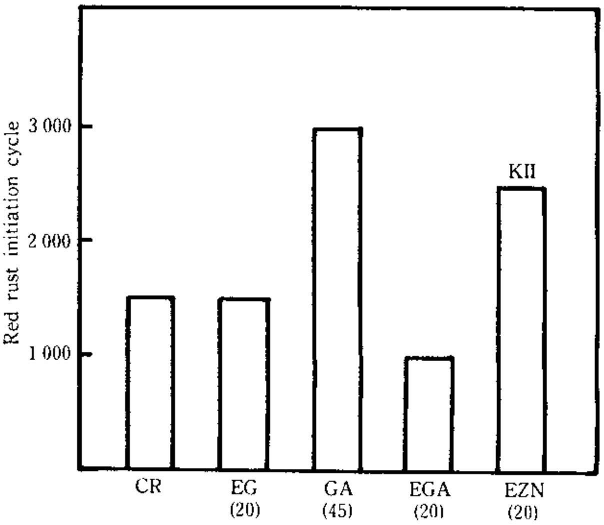


Fig. 9 Effect of zinc-rich coating base metals on corrosion resistance in CCT-B

Thus, an organic composite-coated steel sheet (KII) with better corrosion resistance, weldability, and coating adhesion than conventional organic coated steel sheets was obtained by applying improved zinc-rich paint, to which zinc potassium chromate as anticorrosive pigment and molybdenum disulfide as a lubricant agent

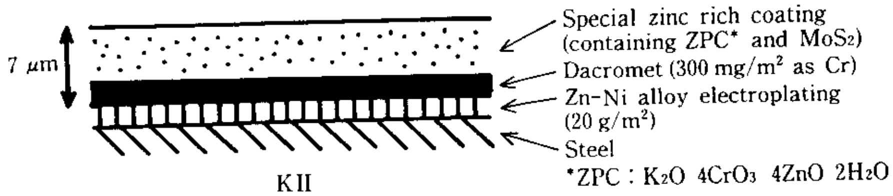


Fig. 10 Schematic cross-section of KII

was added, to a Zn-Ni alloy electroplated steel sheet (20 g/m^2) at a coating thickness of 4.5 μm after Dacromet coating (300 mg/m^2 as Cr).

3 Properties of Organic Composite-Coated Steel Sheet with High Corrosion Resistance (KII)

KII was produced on the coil coating lines at the Hanshin Works of Kawasaki Steel and the Chiba Works of Kawatetsu Galvanizing Co., Ltd. using RIVER HI-ZINC (Zn-Ni alloy electroplated steel sheet, coating weight 20 g/m^2, Ni content 12%) as material. The coating system of KII is schematically shown in **Fig. 10**. Properties of KII are described below.

3.1 Corrosion Resistance in Unpainted Condition

Steel sheets for automobiles are required to provide high corrosion resistance in an unpainted condition in enclosed areas where zinc phosphating and electrodeposition painting are difficult. Therefore, the red rust initiation cycle and perforation corrosion resistance of KII were investigated by conducting the cyclic corrosion tests (CCT-A and CCT-B) and the salt spray test (SST) on unpainted steel sheets.

3.1.1 Red rust initiation cycle

The red rust initiation cycles in the cyclic corrosion tests are shown in **Table 2** and the red rust condition over a period of time is shown in **Photo 1**. The red rust initiation cycles of KII are longer than those of conventional organic coated steel sheets in both CCT-A and CCT-B. Thus, KII shows good corrosion resistance. As is apparent from Photo 1, red rust tends to occur over the whole surface in CCT-B due to the high corrosiveness of salt water immersion, while red rust has a tendency to propagate only from cross-cut portions in CCT-A and SST. In KII, the initiation of red rust is slight even in cross-cut portions in CCT-A and SST. Thus, KII displays better characteristics than conventional products.

Table 2 Cyclic corrosion test results of KII

Cyclic corrosion test	Red rust initiation cycle
CCT-A	150
CCT-B	2 500

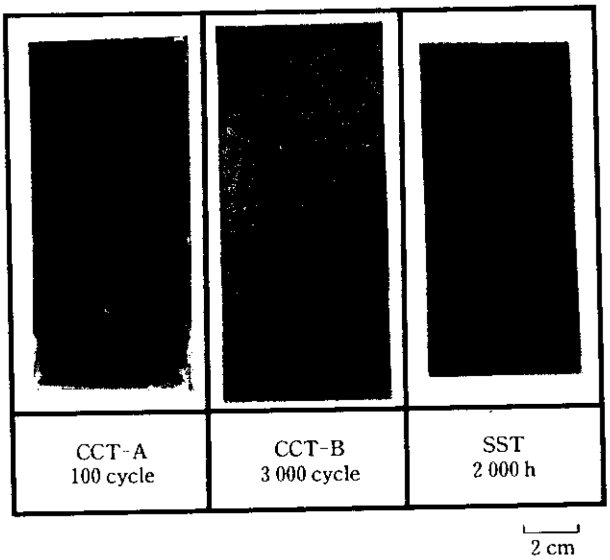
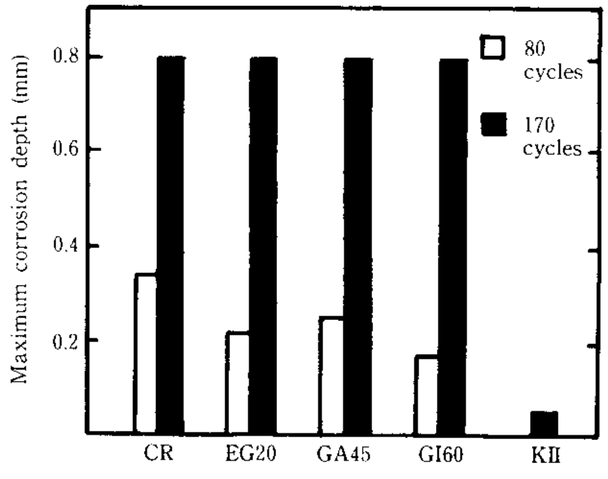


Photo 1 Appearance of KII (unpainted condition) after the corrosion tests

3.1.2 Perforation corrosion resistance

The perforation corrosion resistance of KII was compared with that of conventional zinc-coated steel sheets in terms of the maximum thickness reduction in CCT-A and CCT-B. These resuslts are shown in **Figs. 11** and **12**.

In both CCT-A and CCT-B, KII, which is a precoated steel sheet, has much better perforation corrosion resistance than ordinary zinc-coated steel sheets, such as electrogalvanized steel sheet (EG20, coating weight 20 g/m^2), hot-dip galvanized steel sheet (GI 60, coating weight 60 g/m^2) and galvanealed steel sheet (GA45, coating weight 45 g/m^2). This shows that the sacrificial protection of zinc-coated steel sheets alone is insufficient while the use of corrosion-resistant precoated steel sheets with the barrier effect gained from organic coating is effective in ensuring long-term corrosion protection in automobile parts where uncoated steel sheets are used due to the difficulty of coating treatments such as zinc phosphating and electrodeposition painting and which are subjected to severe corrosive environments.



CR : Cold-rolled
EG : Electrogalvanized (20 g/m²)
GA 45 : Galvannealed (45 g/m²)
GI 60 : Galvanized (60 g/m²)

Fig. 11 Maximum corrosion depths of various precoated steel sheets in cyclic corrosion test A (80 and 170 cycles)

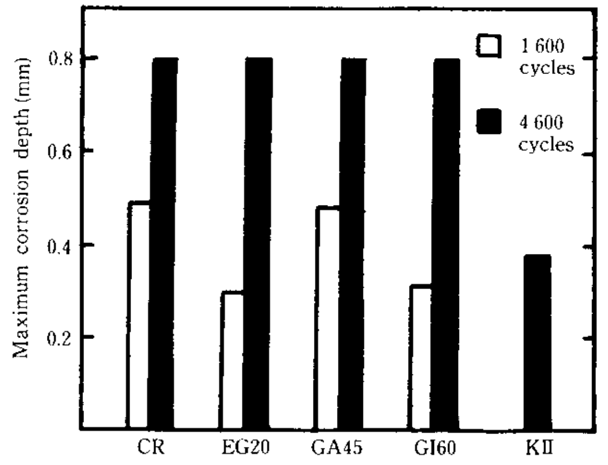


Fig. 12 Maximum corrosion depths of various precoated steel sheets (indicated in figure 12) in cyclic corrosion test B (1600 and 4600 cycles)

KII provides better perforation corrosion resistance than conventional organic coated steel sheets due to a combination of the sacrificial protective effect of the base metal coating and the barrier effect of the organic coating.

3.1.3 Corrosion resistance after forming

With conventional organic coated steel sheets for automobiles, the coating is apt to exfoliate during press forming, leading to reduced coating thicknesses and exposed steel surfaces. As a result, corrosion resistance decreases. The corrosion resistance of KII after forming was investigated by conducting the CCT-A corrosion resistance test on test specimens after cylindrical cup drawing (**Photo 2**).

In general, the drawn part show more damage than flat parts, and corrosion in the drawn part tends to proceed rapidly. In KII, the deterioration in corrosion resistance in the drawn part is very slight because the coating film exfoliation is limited. The low degree of paint film exfoliation is due to the good lubricating property of the film and the sacrificial protective effect of the base metal coating. KII shows substantially improved corrosion resistance after forming compared to conventional organic coated steel sheets.

3.1.4 Corrosion resistance after electrodeposition coating

Corrosion resistance after electrodeposition coating was investigated, in addition to that of uncoated materials. A cross cut was made in an electrodeposition-coated steel sheet and the CCT-A corrosion resistance test was conducted. **Photo 3** shows a comparison of red rust initiation between KII and a cold-rolled steel sheet (CR).

It can be seen from this photo that the development of red rust from the cross-cut portion of KII is suppressed by the anticorrosive pigment in the zinc-rich coating

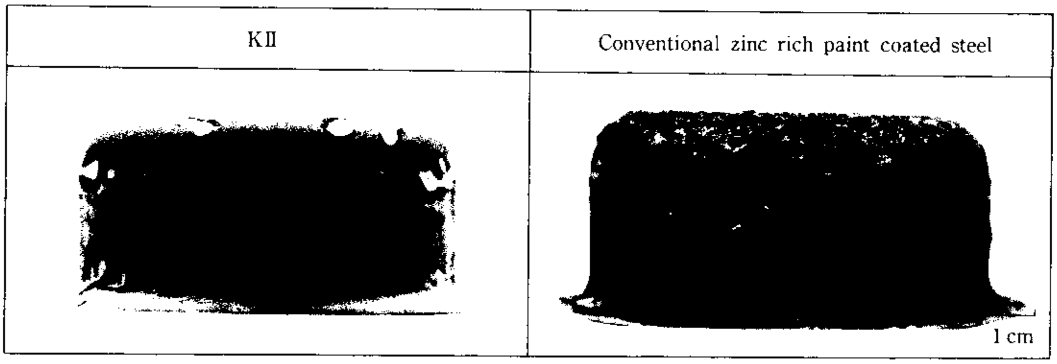


Photo 2 Appearances of KII and conventional zinc-rich paint coated steel in cyclic corrosion test A (95 cycles) after cylindrical cup drawing

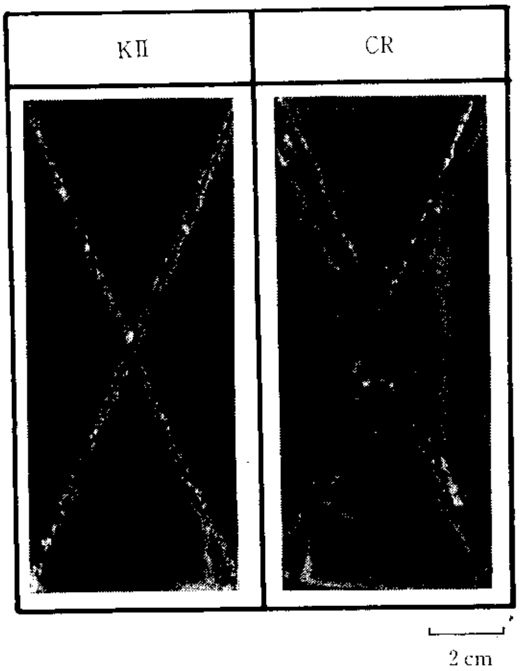
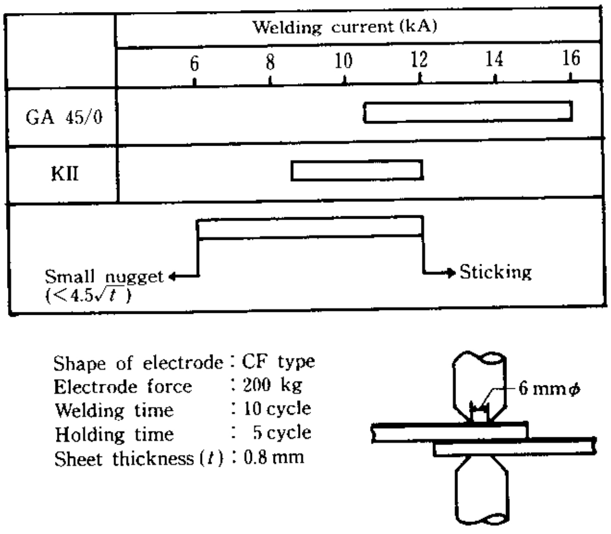


Photo 3 Corrosion behaviors of KII and CR in cyclic corrosion test A (150 cycles) after electrodeposition painting



Shape of electrode : CF type
Electrode force : 200 kg
Welding time : 10 cycle
Holding time : 5 cycle
Sheet thickness (t) : 0.8 mm

Fig. 13 Acceptable spot welding current ranges for GA 45 and KII

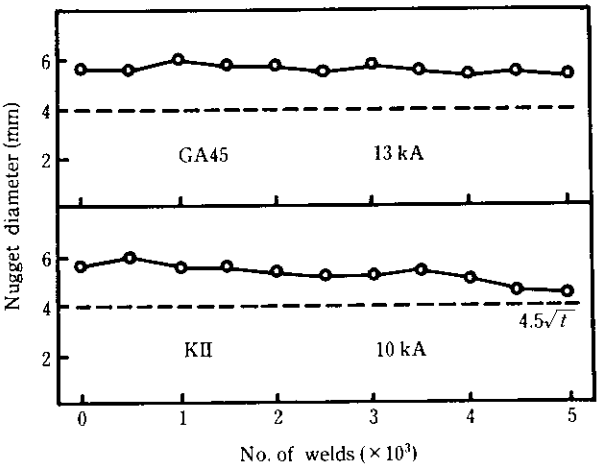


Fig. 14 Continuous spot-weldability of GA 45 and KII

and the sacrificial protective effect of the Zn-Ni coated layer.

3.2 Coating Adhesion during Forming

The cylindrical cup drawing and the bead drawing tests were conducted to evaluate coating adhesion of KII during forming. Cellophane adhesive tape was applied to the drawn part and coating adhesion was evaluated by measuring the exfoliated film weight after removing the tape.

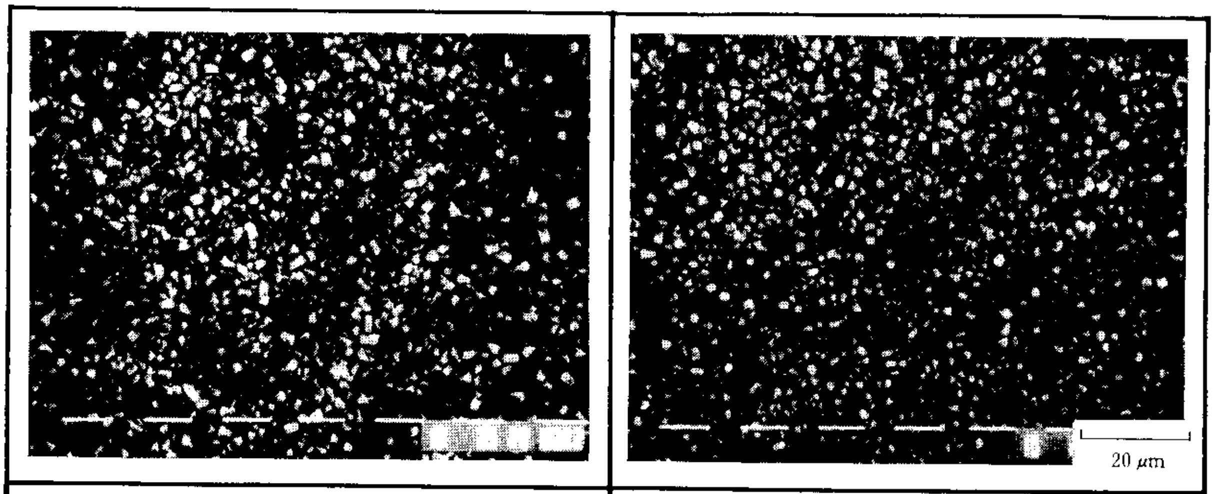
The exfoliated coating weight was about 8 mg in the cylindrical cup drawing test and about 18 mg in the bead drawing test. These values are about one-seventh those of conventional organic coated steel sheets. Thus, the coating adhesion of KII is substantially better than that of conventional organic coated steel sheets. This improvement is due to a decrease in the zinc-rich coating and an improvement in the lubricating property caused by the addition of molybdenum disulfide to the coating.

3.3 Weldability

First, the available spot welding current range with a minimum current value at which a sufficient nugget diameter is obtained and with a maximum current value at which sticking does not occur was sought, and then the continuous spot welding test was conducted at a median current value. The available spot welding current range and continuous spot weldability of KII as compared to those of GA45, one of the zinc-coated steel sheets, are shown in **Figs. 13** and **14**, respectively.

As with conventional organic coated steel sheets, the available welding current range of KII is shifted to small current side and slightly narrower than that of GA45.

The number of continuous spot welds possible with KII was over 5 000, as with GA45. In ordinary zinc-coated steel sheets, alloying of the electrode with zinc develops progressively with the number of continuous spot welds. As a result, the electrode tip softens and deforms, and the contact area between the electrode and the steel sheet increases, resulting in decreased current density. As a consequence, the nugget diameter decreases gradually. In ordinary organic coated steel sheets, tar-like organic matter generated by the thermal decomposition of the coating accumulates on the elec-



Coating weight : 2.4 g/m^2	Coating weight : 2.5 g/m^2
P ratio : 0.93	P ratio : 0.95
Crystal size : 1.7-2.6 μm	Crystal size : 1.3-2.6 μm
KII	CR

Photo 4 SEM photomicrographs of uncoated side of KII and CR after phosphate treatment (Nihon Parkerizing Co., Ltd. Bt-3004)

trode surface as the number of continuous spot welds increases. Electric power is applied causing the dielectric breakdown of the coating, and the resulting substance contaminates the electrode. As a result, the resistance of the electrode tip increases sharply and electric conductivity is lowered, leading to a marked decrease in the nugget diameter.[17] In KII, however, the coating resistance is low because of the thinner organic coating thickness. The electric conductivity is improved because the contamination of the electrode by the accumulation of tar-like organic matter is negligible. It seems that this is the reason excellent continuous spot weldability was obtained with KII.

3.4 Phosphatability of Uncoated Side

An organic coated steel sheet is a one-side coated steel sheet. This steel sheet is used as an outer panel of an automobile body with the coated side as the inner side. Thus, the coated side is subjected to a severe corrosive environment, while the uncoated side as the outer side allows easy finish coating and has a good appearance which is an important marketing considerations. The outer, visible side, i.e., the uncoated side of an organic coated steel sheet, is treated in order of zinc phosphating, electrodeposition painting, middle coating, and top finish coating, and must provide phosphatability which is as good as that of ordinary cold-rolled steel sheets in terms of corrosion resistance after painting and appearance. Therefore, the phosphatability of the uncoated side of KII was investigated and evaluated by using the phosphate coating weight, *P* ratio, size of phosphate crystal by SEM, and the presence of uncoated portions. Results are shown in **Photo 4**. KII showed the same results as for cold-rolled steel sheet in all of these evaluation. Thus, it was ascertained that KII possesses good phosphatability.

4 Conclusions

Kawasaki Steel developed an organic composite-coated steel sheet with high corrosion resistance "Zincrometal-KII" which has better coating adhesion during forming, weldability, and corrosion resistance than conventional organic coated steel sheets.

Corrosion resistance was improved by applying a zinc-rich coating containing 4 wt% zinc potassium chromate as an anticorrosive pigment to a Zn–Ni alloy electroplated layer (20 g/m^2). Coating adhesion was improved by adding 0.4 wt% molybdenum disulfide as a lubricating agent to the zinc-rich coating and reducing the coating thickness to 4.5 μm. This decrease in the organic coating thickness proved very effective in improving weldability.

KII, which resulted from these improvements, is suitable for application to those parts of automobile bodies where zinc phosphating and electrodeposition coating are difficult. The mass production of KII as an organic composite-coated steel sheet with high corrosion resistance may soon result in the replacement of conventional organic coated steel sheets.

The authors would like to extend their sincere thanks to the people at Nissan Motor Co., Ltd. for their kind cooperation in conducting this study.

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