Inorganic Dry Film Lubricant Coated Galvannealed Steel Sheet with Excellent Press Formability and Adhesive Compatibility*



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Synopsis:

An inorganic dry film lubricant coated galvannealed steel sheet has been developed in order to improve the press formability of automotive body panels. The frictional property and the press formability of this steel sheet are equivalent to those of galvannealed steel sheet coated with an Fe-P electroplated upper layer. The developed inorganic dry film lubricant coated galvannealed steel sheet shows good compatibility with various kinds of adhesives, such as structural adhesives and mastic adhesives. The spot weldability of this steel sheet is equal to that of a galvannealed steel sheet. The properties followed by phosphating are the same as those of galvannealed steel sheet, due to the film dissolvable property into alkaline decreasing solution. The inorganic dry film lubricant layer can be easily coated with a conventional coater roll and a drier on galvannealed steel sheets.

1 Introduction

In order to improve the corrosion resistance of automotive body panels, various zinc coated steel sheets have been developed. Among these steels, the use of galvannealed steel sheet has been increased with its good corrosion resistance and reasonable production cost. In 1980's the corrosion resistance target called "10-5", meaning no perforation corrosion for 10 years and no cosmetic corrosion for 5 years, has been set. To meet the target, a coating weight of 50 g/m^2 was considered to be necessary. However, the increase in coating weight of galvannealed steel sheets has induced press troubles such as fracture in some cases, because of on increase in the friction coefficient by the presence of soft ζ phase on the surface.[1)] Further the galvannealed steel sheets some times show the poor paintability for the exposed panel quality. To improve these properties, double layered galvannealed steel sheets coated with an Fe base electroplating upper layer such as Fe-P or Fe-Zn has been developed.[2,3)] These Fe base electroplating layers provides feasible frictional properties with proper coating hardness.[4)] The double layered galvannealed steel sheets have been applied to improve press formability as well as paintability.

In recent years, a demand arises to eliminate the Fe base upper layer due to the material cost reduction and worldwide purchasing policy. At the same time, a strong demand arises to develop a dry film lubricant coating on galvannealed steel sheets, which can be easily produced by using existing coating facilities in the world. In addition, further improvement of press formability has been requested for the coating for complex forming panels of complicated shape including body side outer and so on.

To meet these requirements, various kinds of inorganic dry film lubricant coatings with good frictional properties have been developed.[5–12)] However, these coatings do not satisfy all the desired properties for automotive use, such as frictional properties, compatibility with adhesives and weldability.[13,14)]

Kawasaki Steel has recently developed an inorganic dry film lubricant coated galvannealed steel sheet which satisfied all the properties for automotive use. This newly developed steel sheet shows superior press formability equivalent to double layered galvannealed steel sheet, and also shows all the desired properties such as

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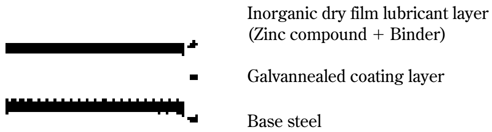

Fig. 1 Cross-sectional view of inorganic dry film lubricant coated galvannealed steel sheet

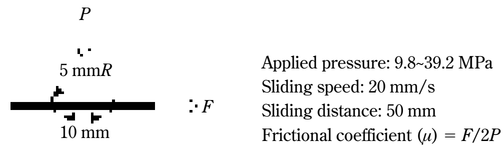

Fig. 2 Schematic illustration of friction test

compatibility with adhesives, weldability and phosphatability. This article describes the design conception of the inorganic dry film lubricant layer and characteristics of the developed steel sheet.

2 Structure and Design Conception of the Developed Steel Sheet

Figure 1 shows a cross-sectional view of the developed inorganic dry film lubricant coated galvannealed steel sheet. The inorganic dry film lubricant layer is coated on a galvannealed coating layer. This inorganic dry film is composed of a zinc compound and binder. The zinc compound works as a lubricant to prevent metallic contact between a galvannealed coating layer and a press die during press forming and improves the material flow and thus the formability.

Another superior characteristic of this developed steel sheet is excellent compatibility with various adhesives. It is well known that the compatibility with adhesives depends on the surface condition of the substrates and the wetting ability of the adhesives on the substrates.[15] The binder in this developed film not only hardens the film, but also improves compatibility with adhesives by dissolving into an adhesive component. Therefore, the developed inorganic dry film lubricant coated galvannealed steel sheet shows good compatibility with various adhesives, and in some cases shows even better compatibility than that of galvannealed steel sheets.

The developed inorganic dry film lubricant layer can be easily applied with a conventional coating facilities for any galvannealed steel sheets.

3 Experimental Procedure

3.1 Test Specimens

Table 1 summarizes the test specimens used in this study. Conventional galvannealed steel sheets (hereafter called GA) with a thickness of 0.8 mm and a coating weight of 45 g/m^2 were used as base substrates. The developed steel sheets (hereafter called GA-K) were prepared by generating the inorganic dry film layer on galvannealed steel sheets by roll coating and drying, with a coating weight of 0.8 g/m^2. To compare frictional properties and press formability, double layered galvannealed steel sheets coated with an Fe-P electroplated upper layer with a coating weight of 4 g/m^2 (hereafter called GAX) were used.

Table 1 Test pieces used in this study

Symbol	Coating
GA	Galvannealed steel sheet
GA-K	Galvannealed steel sheet with inorganic dry film lubricant layer (0.8 g/m^2)
GAX	Galvannealed steel sheet with electroplated Fe-P coating layer (4 g/m^2)

3.2 Frictional Properties

The frictional properties were examined by using the friction tester shown in **Fig. 2**. The frictional coefficients in case of using no oil were measured with an applied pressure of 9.8 MPa, and that, in case of using 1.5 g/m^2 of rust preventive oil, were measured with an applied pressure of 39.2 MPa. Frictional tests were performed in such conditions as a sliding speed of 20 mm/s, and a sliding distance of 50 mm. The frictional coefficient μ was evaluated in terms of the ratio of the applied force, P and the drawing force, F. In addition, the surface appearance of the specimens after frictional tests without oil was observed by using a scanning electron microscope (SEM).

3.3 Press Formability

The press formability was examined by the cup forming test shown in **Fig. 3**. The tests were performed using specimens with 1.5 g/m^2 of rust preventive oil, under conditions of a blank diameter of ϕ74 mm, a blank holding force of 17.6 kN, and a drawing speed of 5 mm/s. Evaluation of press formability was done based on measured cup height when the cup fractured, so-called limiting drawing height.

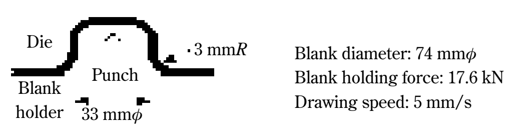

Fig. 3 Schematic illustration of cup forming test

3.4 Compatibility with Adhesives

The compatibility with adhesives was investigated using two kinds of typical adhesives, namely (a) structural adhesive and (b) mastic adhesive. Schematic illustrations of the adhesion tests are shown in **Fig. 4**. The adhesion test using structural adhesive was the T-peel test, of which procedure is as follows. First, identical

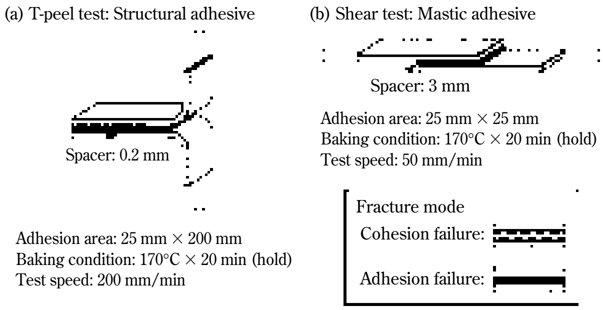

Fig. 4 Schematic illustration of adhesion test

two specimens were dipped into the rust preventive oil, taken out, and then hold upright for 24 h. Then, the adhesive was coated between two specimens spaced 0.2 mm apart with an adhesion area of 25 mm × 200 mm, after which the adhesive was cured under a baking condition of 170°C for 20 min. After 24 h, the T-peel tests were conducted at a rate of 200 mm/min. The compatibility with structural adhesive was determined by the T-peel strength and the fracture modes i.e., cohesion failure and adhesion failure denoted CF and AF respectively.

The adhesion test using mastic adhesive was a shear test. Using two specimens treated by the rust preventive oil in the same way as above, the adhesive was coated between two specimens of 3 mm apart with an adhesion area of 25 mm × 25 mm, and then cured under a baking condition of 170°C for 20 min. After 24 h, the shear tests were conducted at a rate of 50 mm/min. The compatibility with mastic adhesive was determined in terms of the shear strength and the above-described fracture modes.

3.5 Spot Weldability

Figure 5 shows the spot welding conditions in the present test. Specimens with a thickness t of 0.8 mm were welded by using DR type electrodes. The welding force was 1.96 kN, and the welding pattern (squeeze time-weld time-hold time) was 25 cycles-12 cycles-1 cycle. The weldable current range was estimated from measured nugget diameters. The lower limit of the weldable current was defined as the point satisfying $4\sqrt{t}$, and the upper limit was defined as the point at which sticking was observed. The continuous spot weldability at a suitable welding current was defined as the number of welds at which nugget diameter satisfied $4\sqrt{t}$.

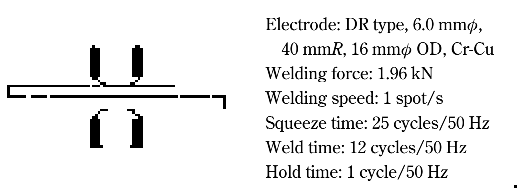

Fig. 5 Spot welding conditions

3.6 Alkaline Degreasability and Phosphatability

The alkaline degreasability was examined by using an ordinary alkaline degreasing treatment. The conditions are degreasing solution of "FC-L4460" manufactured by Nihon Parkerizing Co., Ltd., solution temperature of 43°C, and treating time of 60 s of spraying + 120 s of dipping. The film dissolvable property into alkaline degreasing solution was calculated from measured coating weight of the inorganic film before and after degreasing. The phosphatability was examined using an ordinary surface conditioning and phosphating treatment ("PB-L3020 system" manufactured by Nihon Parkerizing Co., Ltd.) after the above-described alkaline degreasing treatment. The phosphate crystals formed by this phosphating treatment were observed by using SEM.

4 Characteristics of the Developed Steel Sheet

4.1 Frictional Properties

Figure 6 shows the experimental results of frictional coefficients. The frictional properties were examined under two conditions, i.e., low applied pressure of 9.8 MPa without oil and high applied pressure of 39.2 MPa with rust preventive oil. The developed inorganic dry film lubricant coated galvannealed steel sheet showed excellent frictional properties, especially outstanding properties in case of no oil, and its frictional coefficient was lower than that of the double-layered galvannealed steel sheet. With the rust preventive oil, the frictional properties of the developed steel sheet were equivalent to those of the double-layered galvannealed steel sheet, and much better than with the galvannealed steel sheet.

To determine the reason for the frictional property improvement of this developed steel sheet, the surface appearance of the specimens was observed by SEM after

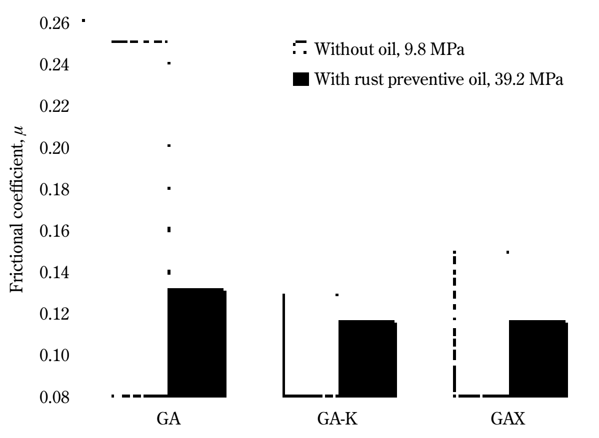

Fig. 6 Effect of inorganic dry film lubricant on frictional coefficient

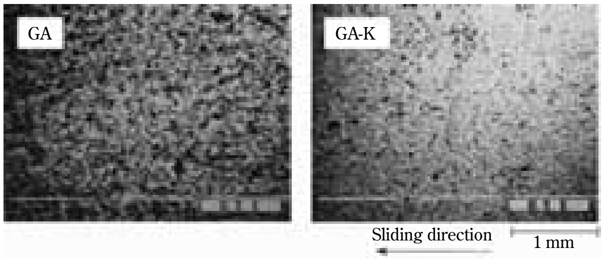

Photo 1 Surface appearances after friction test in case of using no oil

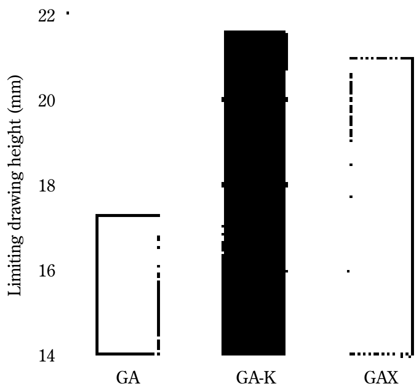

Fig. 7 Effect of inorganic dry film lubricant on press formability

the friction test in case of using no oil. **Photo 1** shows the results. The surface of the galvannealed steel sheet after the friction test had large areas of black spots where the galvannealed coating layer had rubbed off and the surface became smooth. On the other hand, the smoothed area of the developed steel sheet after the friction test was rather narrow. This fact suggests that the developed inorganic dry film lubricant layer was able to prevent metallic contact between the galvannealed coating layer and the press forming die, resulting in a remarkable decrease of the frictional coefficient.

4.2 Press Formability

Figure 7 shows the results of press formability examined in the cup forming test. The press formability of the developed steel sheet was much better than that of the galvannealed steel sheet, and was equivalent to that of the double-layered galvannealed steel sheet. This indicates that the developed steel sheet can be applied to automotive body panels which are currently made with double-layered galvannealed steel sheets.

4.3 Compatibility with Adhesives

Figure 8 shows the compatibility with adhesives. Both the T-peel strength using structural adhesive and the shear strength using mastic adhesive of the developed steel sheet were equal to or somewhat better than those of the galvannealed steel sheet. In addition, the fracture made of both the galvannealed steel sheet and

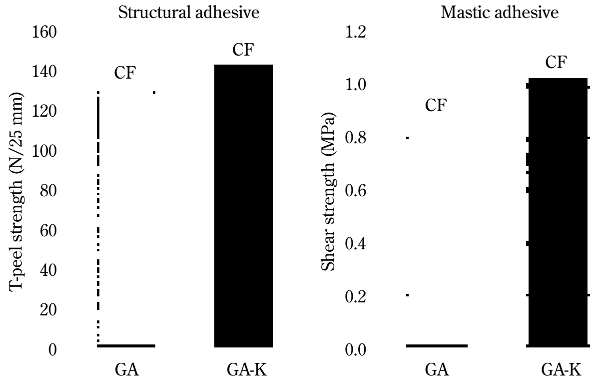

Fig. 8 Effect of inorganic dry film lubricant on adhesive compatibility

the developed steel sheet was cohesion failure (CF).

The developed steel sheet showed better compatibility than the galvannealed steel sheet with some kinds of adhesives. The reason for this, it may have been that the binder in this developed film improved compatibility with adhesives by dissolving into an adhesive component.

4.4 Spot Weldability

As **Fig. 9** shows, the weldable current range of the developed steel sheet was almost equal to that of the galvannealed steel sheet. **Figure 10** shows the results of continuous spot weldability test. The nugget diameter reduction of the developed steel sheet was similar to that of the galvannealed steel sheet. At least 3 000 welds were conducted until failing the nugget diameter of $4\sqrt{t}$ specification. Thus, these results show that the spot weldability of the developed steel sheet was equal to that of the galvannealed steel sheet.

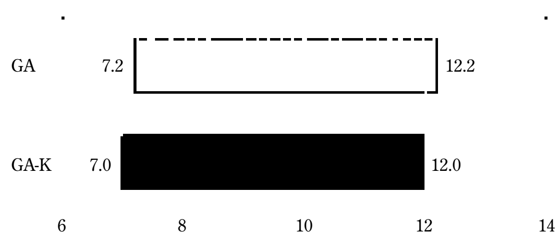

Fig. 9 Weldable current range in spot welding

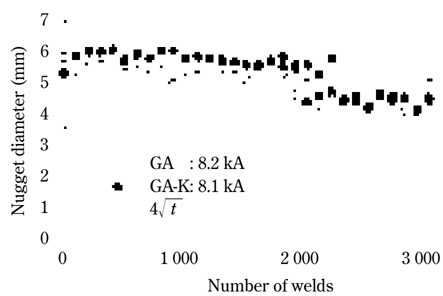

Fig. 10 Electrode life in continuous spot welding

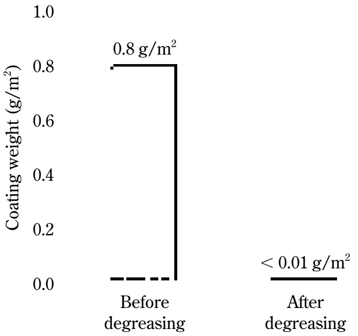

Fig. 11 Degreasability of inorganic dry film lubricant

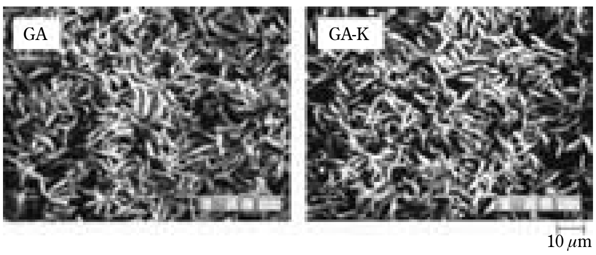

Photo 2 SEM images of phosphate crystals

4.5 Alkaline Degreasability and Phosphatability

Figure 11 shows the coating weight of the developed inorganic dry film lubricant layer before and after alkaline degreasing treatment. The coating weight of the film was degreased by alkaline degreasing treatment from 0.8 g/m^2 to less than 0.01 g/m^2. This shows that the developed inorganic dry film lubricant layer was almost completely dissolved into an alkaline degreasing solution by ordinary alkaline degreasing treatment.

Photo 2 shows the SEM images of phosphate crystals. The phosphate crystals of the developed steel sheet were equivalent to those of the galvannealed steel sheet, and the faults, such as missing or unevenly distributed crystals, were not observed. These findings indicate that the phosphatability and the properties induced by the phosphating of the developed steel sheet were the same as those of the galvannealed steel sheet, due to the dissolving of the film into an alkaline degreasing solution.

5 Conclusions

An inorganic dry film lubricant coated galvannealed steel sheet with excellent press formability and good compatibility with adhesives has been recently developed. This steel sheet can be applied to automotive body panels. The characteristics of this steel sheet are as follows:

(1) The frictional properties and the press formability of this steel sheet are equivalent to those of galvannealed steel sheet coated with an Fe-P electroplated upper layer.
(2) This steel sheet shows good compatibility with various kinds of adhesives, such as structural and mastic adhesives.
(3) The spot weldability of this steel sheet is equal to that of a galvannealed steel sheet.
(4) The properties induced by phosphating are the same as those of galvannealed steel sheet, due to the dissolving of the film into an alkaline degreasing solution.

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