

Lubricant Treated Anti-Corrosion Steel Sheets for Automotive Use —Galvannealed Steel Sheets with Excellent Press Formability—[†]

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Abstract:

This paper describes the development concept and quality properties of a high lubrication galvannealed steel sheet (GA), "GAN," in which the sliding property of GA is greatly improved, securing the high formability required in hard-to-form automotive body panels. The developed product displays press formability equivalent to that of GA with an Fe-Zn alloy electroplated layer on the GA surface (double-layered GA), and spot weldability and adhesive compatibility superior to those of GA. High lubricity is obtained in the developed product by applying an ultra-thin (50 nm) Ni-Fe-O inorganic coating. As the respective roles of these elements, Fe, Ni, and O improve the anti-sticking property during press forming, Ni improves affinity with press oil and spot weldability, and Fe improves adhesive compatibility. JFE Steel has also developed an organic lubricant coated GA with improved lubrication properties and anti-galling properties. The lubricant coating can be removed with the press oil in the alkali degreasing process after press forming.

1. Introduction

As the percentage of rust-preventive (corrosion resistant) steel sheets used in automobile bodies has increased and heavier coating weights have been adopted to improve corrosion resistance, galvannealed

steel sheets (GA) have become the mainstream in corrosion-resistant steel sheets for automotive use in Japan. In explaining this trend, for economic reasons, it is difficult to produce electrogalvanized steel sheets, with heavy coating weights and consequently, there has been a changeover to hot dip galvanized products, which are advantageous for heavier coating weights. However, GA products increase frictional resistance, inducing pressing defects such as fracture in hard-to-form parts. Double-layered galvannealed steel sheets (double-layered GA), in which an Fe-rich electroplated upper layer of Fe-Zn alloy or Fe-P is applied on the GA surface, was developed to improve the paint finishing property (prevention of crater-like defects during electrodeposition coating) when used in exposed body panels. On the other hand, as the Fe-rich layer showed the excellent sliding property, the developed material was adopted not only for improvement of paint finishing property, but also for improvement of press properties.¹⁾

In recent years, with heightened requirements for cost reduction in materials, there has been tendency to avoid the use of double-layered GA, while on the other hand, further improvement in press formability has also been required in steel sheets to meet the needs of one-piece forming of large panels and use of high strength steel sheets.

Therefore, the development of a GA sheet with a lubricant layer, which provides excellent lubrication

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properties equivalent to those of double-layered GA, together with other required properties equal or superior to those of GA, has been strongly desired.

JFE Steel carried out research and development to meet these needs over a period of many years. As a result, the company developed²⁻⁴⁾ and commercialized an inorganic high lubrication GA sheet, “GAN,” which possesses high lubricity equivalent to that of double-layered GA, together with improved spot weldability and adhesive compatibility, and can also be manufactured at low cost.

This paper discusses the concept of the coating design and the quality properties of the developed product. An organic-type solid lubricant treatment technology⁵⁾ which succeeded in imparting lubrication characteristics superior to those of double-layered GA is also described in brief.

2. Concept of Development of Lubricant Coating

The sliding property of steel sheets varies greatly depending on a various factors related to the steel sheet surface. In the case of GA, the sliding property is affected by the roughness of the sheet surface and chemical factors such as the Fe content of the coating layer and adsorbates/oxides in the coating surface layer, but with all these factors, it was difficult to improve the sliding property to the level of double-layered GA stably by controlling the factors concerned.

JFE Steel therefore carried out development to improve the sliding property of GA based on the concept of applying an ultra-thin lubricant coating.

Lubrication conditions can be classified as fluid lubrication, in which a fluid film of oil or some other substance exists between the surfaces of the steel sheet and the die, and boundary lubrication, in which the two surfaces are in contact via an adsorbed molecule film. During actual press forming, friction at low surface pressures is a mixed condition of fluid lubrication and boundary lubrication, with a high ratio of fluid lubrication. However, under high surface pressure conditions, partial direct contact occurs between the die and the material being formed, and areas in a sticking condition also exist. To reduce the friction coefficient, it is necessary to secure a wide region of fluid lubrication or boundary lubrication and a low shear force in areas of metal-to-metal contact in a sticking condition.

Thus, the design guidelines adopted to improve the sliding property were (1) improvement of the anti-sticking property and (2) achievement of improved affinity between the press oil and steel sheet surface by an ultra-thin coating. Study also targeted improved spot weldability and adhesive compatibility. As a result, an

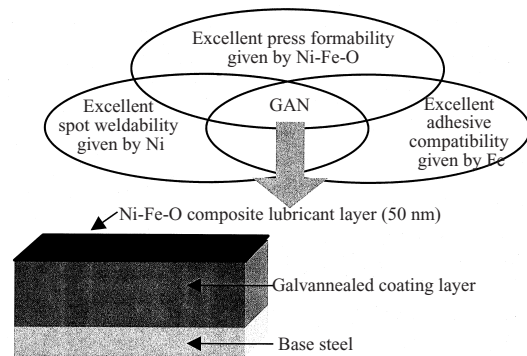


Fig. 1 Basic concept and cross sectional view of GAN

Ni-Fe-O system inorganic lubricant coating was developed, and practical application of a high lubrication GA sheet, “GAN,” was realized. **Figure 1** shows the development concept and the coating structure. The developed product satisfies a variety of property requirements at a high level by adopting a composite composition design of ingredients which are effective for the respective required properties. As its distinctive feature, excellent press formability, weldability, and adhesion were successfully imparted with an ultra-thin coating (50 nm).

3. Development of Lubricant Coating

3.1 Improvement of Anti-Sticking Property

In studying improvement of the anti-sticking property, samples were prepared by applying ultra-thin coatings (50 nm) of pure Zn, pure Fe, pure Ni, and Ni-Fe to GA surface by electroplating (sulfuric acid bath), and the friction coefficient was measured in a flat sliding test. A test piece coated with wash oil was set, and the drawing force, F was measured while pressing a tool on the test piece at a constant load, N . The friction coefficient was obtained from F/N . The tool used was 3×10 mm (surface pressure = 130 MPa), assuming the drawing bead part in actual pressing. The tool material was SKD11. Here, wash oil (extreme pressure agent-free) was used in the measurements. The results are shown in **Fig. 2**. The friction coefficient was reduced somewhat by coating with pure Zn, pure Fe, pure Ni, and Ni-Fe alloy coating, decreasing in the order of Zn, Ni, Ni-Fe, and Fe. This is attributed to differences in the anti-sticking property with the tool due to differences in the melting points of the respective metals. However, the level of double-layered GA, which was the target, was not achieved even with the Fe coating. When an Ni-Fe-O coating was used, it was found that the friction coefficient is greatly reduced in comparison with Ni-Fe coating and the other coating.

As described above, a certain degree of improvement in the anti-sticking property can be expected by using

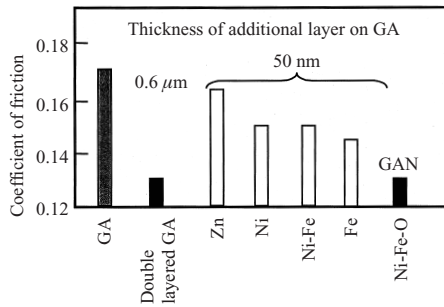


Fig.2 Improvement of friction property by additional layer on GA

high melting point metal(s) in the lubricant coating, but a satisfactory level is not reached with an ultra-thin coating of 50 nm using a simple metal system. Furthermore, the anti-sticking property can be improved greatly, which maintaining coating thickness ultra-thin, by compounding oxygen in a high melting point metal coating.

3.2 Improvement in Affinity with Press Oil

Figure 3 shows the drawing force charts for GA and the developed product when a flat sliding test was performed using (a) base oil and (b) rust-preventive oil containing additives like extreme pressure agents and anti-rust agent. In comparison with GA, GAN showed a low drawing force with either of these oils. This is attributed to improvement of the anti-sticking property achieved by coating with a high melting point Ni-Fe-O inorganic compound. Furthermore, with the base oil, the width of fluctuations in the drawing force showed virtually no difference in GA and GAN, but with the rust-preventive oil, the fluctuation width with GAN was reduced in comparison with GA. Mori et al. compared the chemical affinity of organic sulfur compounds with various transition metals and showed that Ni has high adsorption activity.⁶⁾ Based on this fact, the phenomenon in which the fluctuation width is reduced with GAN only when using the rust-preventive oil, as observed here, is considered as follows: The reactivity of the extreme pressure agents in the lubricating oil is increased by the presence of Ni in the lubricant coating (increase in affinity with press oil). It is thought that the phenomenon can be explained by

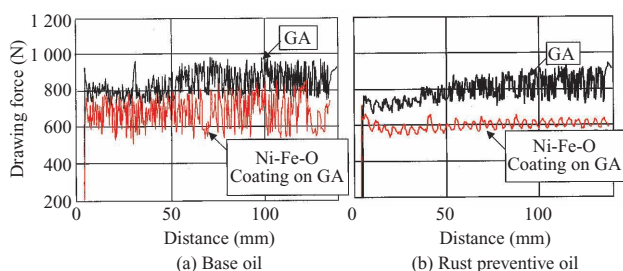


Fig.3 Change in drawing force during measurement of friction coefficient

the fact that the shear force at areas of metal-to-metal contact is reduced by adsorption of the extreme pressure agents at the new surface which occurs during pressing.

3.3 Spot Weldability and Adhesion

Figure 4 shows the results of a continuous spot welding test with GA and GAN. Since adequate welded joint strength cannot be obtained when the nugget diameter falls below a value of $4\sqrt{t}$ (t : sheet thickness), this point is considered to represent electrode life. Although the electrode life with GA is approximately 2 500 spots, with GAN, electrode life is approximately double this number. When welding Zn-coated steel sheets, electrode wear is caused by formation of a brittle Cu-Zn alloy by reaction between the Cu of the electrode and the molten Zn during heating caused by the passage of electricity, followed by separation of the Cu-Zn layer from the electrode surface. Photo 1 shows the cross-sections of electrodes after welding 2 500 consecutive spots with GA and GAN, respectively. With GA, the electrode tip displays a concave shape caused by separation of the Cu-Zn alloy, but in contrast, the electrode tip is flat with GAN. This is thought to be because Ni suppresses formation/separation of the Cu-Zn alloy, and it is assumed that electrode life is approximately twice as long with GAN in comparison with GA as a result.⁷⁾

Because many kinds of adhesive are frequently used in automobile bodies, it is also necessary to consider the adhesion property. Photo 2 shows the results of a T peel test. For this test, the samples were prepared by hardening the adhesive at a somewhat low baking temperature, using a representative adhesive. When a pure Ni coating was applied, peeling (black area) occurred

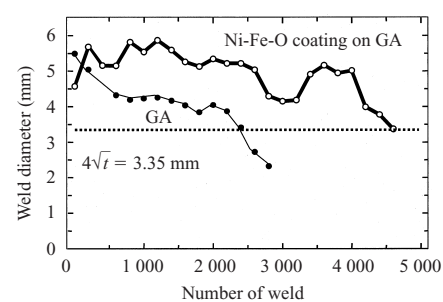


Fig.4 Change in weld diameter during consecutive spot welding

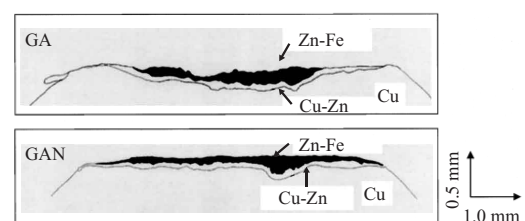


Photo 1 Cross section of electrode after consecutive spot welding test (2 500 times)

| | Appearance of adhesive layer after T peel test | Peel strength (kg/25 mm) |
|---------------------|--|--------------------------|
| GA | | 10.8 |
| Ni coating on GA | | 9.0 |
| Ni-Fe coating on GA | | 11.3 |

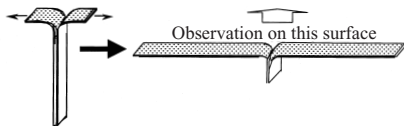


Photo 2 Change in adhesive strength made by metal layer applied on GA

at the interface between the adhesive and the GA surface, and adhesive strength was relatively low. When an Ni-Fe alloy coating was applied, the specimen exhibited satisfactory adhesion with no peeling at interface of the adhesive GA surface, and adhesive strength was superior to that of GA. Thus, adopting a composite of Fe is effective in improving adhesion.

3.4 Summary

Based on the study results described above, an Ni-Fe-O composite coating was developed as an inorganic lubricant coating. This is a composite-type lubricant coating in which the component elements each play respective roles. Specifically, Fe, Ni, and O improve the anti-sticking property during press forming, Ni improves affinity with press oil and spot weldability, and Fe improves adhesive strength.

4. Practical Properties of Developed Product

4.1 Actual Press Formability

Using GA with the same mechanical property values as a base, double-layered coated material and the developed product, an Ni-Fe-O inorganic coated material, were produced on an commercial production line and an actual press-forming test was conducted. Forming was performed at a speed of 10 strokes/min with a 1 200 t single action mechanical press using a front fender model die of actual part scale. The test was performed changing the cushion force during pressing, and the occurrence of fracture and wrinkle in the formed parts were evaluated. Press formability is evaluated from the range within which forming is possible without fracture or wrinkle, with a wider range indicating higher press-formability. **Figure 5** shows the acceptable forming range of the respective samples. It can be understood that GAN has excellent press-formability, equivalent to

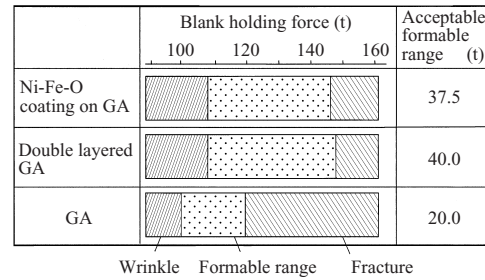


Fig.5 Improvement of press-formability by Ni-Fe-O coating on GA (fender model), 270 MPa-IF steel

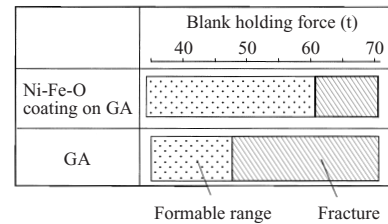


Fig.6 Improvement of press-formability by Ni-Fe-O coating on GA (center pillar), 440 MPa-high strength steel

that of double-layered GA.

Figure 6 shows the results of a press experiment with 440 MPa grade high strength GA steel sheets with a center pillar model die, using the same actual press machine. As noted in the introduction, the use ratio of high strength steel sheets has increased substantially in recent years. This experiment demonstrated that the Ni-Fe-O coating is also effective in improving the press-formability of high strength steel sheets.

4.2 Total Performance of GAN

Figure 7 shows the total performance of GAN in comparison with GA and double-layered GA. GAN achieves a high lubrication property (press-formability), equivalent to that of double-layered GA, with an ultra-thin lubricant coating. Spot weldability and adhesive compatibility are also improved, and corrosion resistance, paintability, and other properties are equal to those of GA.

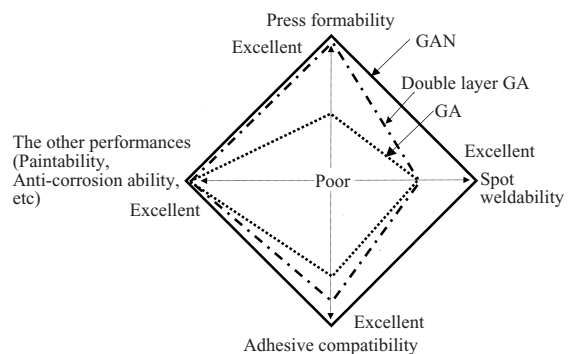


Fig.7 Total performances of GAN in comparison with GA and double layered GA

5. Organic Solid Lubricant Treatment Technology⁵⁾

GAN is a lubrication technology for expanding the formable range within which wrinkles and fracture do not occur to a range that is completely free of problems from the viewpoint of practical use by imparting a sliding property equivalent to that of double-layered GA. JFE Steel has also succeeded in developing a lubrication technology which imparts an even higher sliding property to double-layered GA. This steel sheet was developed to enable more complex, severe press forming. The lubricant coating is a technology in which the strength and toughness of the coating are improved by increasing the T_g (glass transition temperature) of the organic resin, and an improved sliding property and improved anti-galling property are achieved by adding zinc phosphate and polyethylene wax. The lubricant coating is an alkali-soluble type which can be dissolved/removed together with the press oil in the alkali degreasing process after press forming. This technology is capable of substantially improving press-formability, not only with the above-mentioned GA, but also with hot-rolled steel sheets.

6. Conclusion

To meet the needs of press-forming of large one-piece automotive panels and expanded use of high strength steel sheets, extremely high press-formability is required in the galvannealed steel sheets (GA) used as corrosion-resistant sheets in automobiles.

- (1) GAN is a high lubrication GA product which was developed to meet these needs and features an ultra-thin (50 nm) coating of Ni-Fe-O applied on the GA surface.
- (2) The developed product possesses a high sliding property and displays excellent press-formability, equivalent to that of double-layered GA.
- (3) The developed product has improved spot weldability and adhesive compatibility, which are also required for automotive panels, in addition to press-formability. Thus, in comparison with GA and double-layered GA, it is capable of satisfying diverse users at a high level.
- (4) An organic solid lubrication technology which provides an excellent sliding property superior to that of double-layered GA was developed and is successfully contributing to pressing-forming of extremely hard-to-form parts.

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