Properties of Aluminum-coated Steels for Hot-forming

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Abstract

Production of parts with high strength by means of hot-forming process has been receiving considerable attention lately. Application of aluminized steel with good hardenability for this process has been investigated*. Tensile strength over than 1,500MPa was obtained with steel containing around 0.2mass% C. Surface coated layer changed from Al-10% Si to Fe-Al(-Si) phases through the hot-forming process. The steel with Fe-Al(-Si) surface layer showed good paintability even without phosphatized treatment. After painting, they showed good corrosion resistance in JASO-CCT as galvanized steel did. The steel showed good spot weldability due to the stable surface layer at high temperature. (*license product within the frame of global strategic alliance with Arcelor (USIBOR1500))

1. Introduction

High-strength steel sheets are used more and more for automobile components in recent years to reduce the weight of an automobile to improve environmental problems and collision safety. Many car components and structural members can be made significantly lighter by using high-strength steel sheets and reducing their thicknesses. With this as a background, the strength of the steel sheets used therefor has increased from 440- or 590-MPa class to 780-MPa class. Generally speaking, as the strength of a steel sheet increases, its formability deteriorates, and to overcome this problem, various types of high-strength steel sheet products with controlled microstructures have been developed[1]. In spite of these efforts, however, press forming complicated shapes is difficult when the strength of a steel sheet is as high as approximately 1,500 MPa.

In Europe, a forming method called die quenching, hot forming, hot stamping or hot pressing is employed for fabricating automobile structural members having a strength of 1,500 MPa or so[2]. By this method, the strength of a steel sheet is increased through quenching after heating it to a temperature in the austenite temperature range, say, around 900°C, while forming dies are maintained at room temperature. Techniques similar to this are being studied in Japan; similar processes have been studied or put into actual practice using steel

sheets without surface treatment[3, 4]. In Europe, Arcelor has developed and commercially launched an aluminized steel sheet product called USIBOR 1500 for that application. It has excellent hot-pressing properties and corrosion resistance qualities[2, 5-6]. Based on a global strategic alliance with Arcelor, Nippon Steel Corporation has entered into a license agreement regarding USIBOR 1500 and studied the production technologies and properties of the product. This paper reports the results of the company's studies of the properties and characteristics that are required of aluminized steel sheet products for hot forming application.

2. Hot Forming Process

The hot forming method is already a practical reality; the process is outlined in **Fig. 1**. In this method it is necessary to heat a steel sheet to a temperature in the austenite temperature range, equal to or higher than the A_{c3} temperature: for example, steel containing about 0.2 mass % C must be heated to 850°C or higher. Heated steel sheet is extracted from a heating furnace, transferred to a pressing machine, formed into a prescribed shape using dies maintained at room temperature, and thus quenched. At the forming work, the press machine is retained at the lower dead point until the entire steel sheet is quenched sufficiently.

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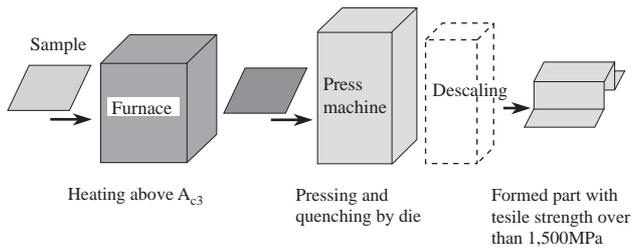
Fig. 1 Schematic illustration of hot forming process

Fig. 2 Relationship between C content and tensile strength

In these processes, when a steel sheet without surface treatment is used, the heating must be done in a non-oxidizing atmosphere for suppressing scale formation and decarburization of its surface layers. Even in the above case, the processeses from the exit from the furnace to the pressing machine are carried out inevitably in the normal atmosphere and for this reason, scale forms on the steel sheet surfaces however short the processing time may be. A formed product will be finally coated for use, but the scale causes a problem for the coating work. Therefore, when a steel sheet without surface treatment is used, an additional process step for removing the scale is required. The scale removal can be done through shot blasting, pickling, or the like, but this results in considerable increases in costs. Further, if shot blasting is employed, the shape of a work may be deteriorated. If a steel sheet with heat-resistant plating is used, the additional process step can be eliminated.

3. Material

Since a steel sheet is rapidly cooled in the hot forming process for hardening it as explained above, a material steel sheet must have good hardenability. To eliminate the furnace atmosphere control and the descaling process to simplify the work processes, the surfaces of the material steel sheet must be coated with plating layers excellent in heat resistance. An aluminized steel sheet having the chemical composition shown in **Table 1** satisfies all these requirements. The properties and characteristics of the specimen steel sheets used for authors' studies, the coating weights of which ranged from 120 to 160 g/m², are described below.

4. Properties of Base Material
4.1 Tensile properties before and after quenching

The properties of the steel sheets before and after the quenching are listed in **Table 2**. The tensile strength was measured using JIS No. 5 test pieces cut out from steel sheets of 1.4 mm thickness. Steel sheets of 490- and 590-MPa classes were quenched by the dies to a strength of 1,500 MPa or more. As seen in **Fig. 2**, the strength after

quenching can be controlled by C content. As has been known, the mechanical properties of steel after quenching change depending on its C content and consequently, the strength after quenching, or the strength of a product, can be controlled by properly adjusting the C content. Note that substitute solid solution elements such as Mn and Cr are known to have only small influence on the strength after quenching. However, since these elements have influences over hardenability, they are essential for securing hardenability, which will be discussed in the following section. It should be noted that an excessive addition of these elements increases the strength of a base steel sheet, even though the effect on the strength is small comparing to that of C.

4.2 Hardenability

Fig. 3 shows the continuous cooling transformation curves (CCT curves) of the specimen steels from 950°C, and **Fig. 4** the relationship between the cooling rate and the hardness after cooling. Mn

Table 1 Chemical composition of steel sheets used

C	Mn	Cr	B
0.22	1.2	0.15	0.002

Table 2 Tensile properties before and after quenching

	Yield strength (MPa)	Tensile strenght (MPa)	Elongation (%)
As coated	394	615	26
As formed	1,280	1,530	8
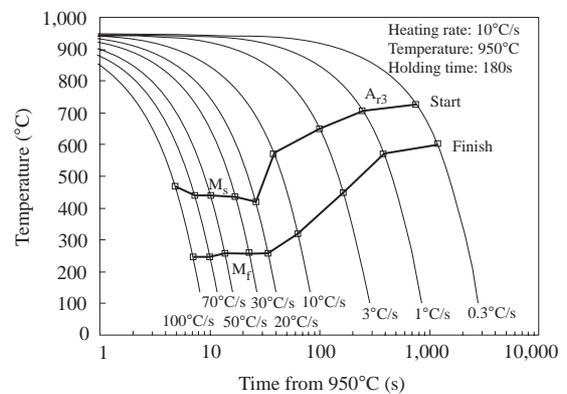
Fig. 3 CCT curves
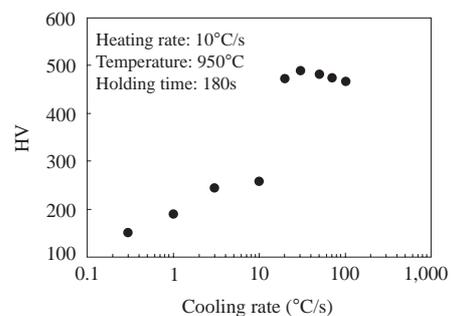
Fig. 4 Influence of cooling rate on hardness

and B had been added to the steels for increasing hardenability and as a result, when they were heated to 950°C and then cooled at a cooling rate of roughly 20°C/s or higher, a hardness of HV 450 to 500 was obtained. The cooling rate for quenching is determined by the conditions for avoiding the transformations of austenite into ferrite, pearlite and bainite during cooling. As the grain size of austenite becomes smaller, or the heating temperature becomes lower, the transformations into these phases take place quicker and as a consequence, the cooling rate for the quenching must be higher. The authors studied this aspect changing the heating temperature from 900 to 1,000°C, but no significant difference was observed in this heating temperature range. The critical cooling rate was 20 to 30°C/s or so.

From Figs. 3 and 4 and the results of the tests at other heating temperatures, it is understood that a sufficient hardness is obtained as far as the cooling rate during the hot forming is equal to or higher than 30°C/s. However, in actual practice, the cooling rate during the transfer from the heating furnace to the pressing machine and the time period before the commencement of the pressing work is close to that in the air.

When a steel sheet with thickness of about 1.4 mm is cooled in the normal atmosphere, the cooling rate is 10 to 15°C/s. It is clear from Figs. 3 and 4 that the strength of the steel sheet is decreased when it is cooled to 600°C or lower at this cooling rate. To cool a steel sheet from 950 to 600°C it takes 23 to 35 s; therefore, a material steel sheet can be quenched if its cooling with the pressing dies is commenced within 23 s after it is extracted from a heating furnace. It should be noted that this time allowance changes depending on heating conditions, the atmosphere during the cooling and surrounding environment and for this reason, it has to be determined on the basis of actual work environment.

4.3 Toughness

In the case where strength is enhanced through formation of martensite by quenching, there is a fear that toughness is lowered, especially at a low temperature. **Figs. 5** and **6** show the results of Charpy impact tests carried out for studying this aspect. Fig. 5 shows the relationship between impact toughness and test temperature, and Fig. 6 the relationship between percent brittle fracture and test temperature. 2.5 mm sub-size test pieces with a 2-mm U-shaped notch were cut out from water-quenched steel sheets of 4 mm in thickness and used for the tests. Whereas the absorbed energy does not change significantly from room temperature to –60°C, the percent brittle fracture increases at –40°C and lower, reaching 80% at –80°C. The transition temperature (Tr_{S50}) is somewhere around –60°C. Since the percent brittle fracture tends to decrease as the material thickness
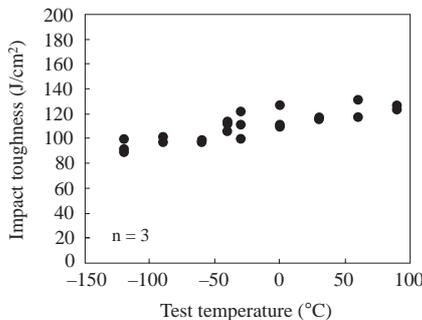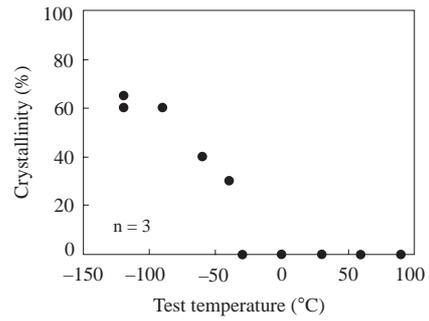
Fig. 6 Relationship between percent brittle fracture and test temperature

decreases, the transition temperature is presumably lower yet if the thickness is less than 2.5 mm. Based on these results, it is presumed that the steel sheet product, which has an as-quenched metallographic structure mainly consisting of a martensitic phase when it is finally used, does not display any abrupt change in absorbed energy or the mode of fracture even if it is exposed to a low temperature under normal conditions of use.

5. Spot Weldability

Test pieces for spot welding tests were prepared by heating aluminized steel sheets of 1.2 mm thickness having a coating weight of 120 g/m² to 950ºC, retaining at the temperature for 5 min, and then quenching by pinching them with metal dies from both the sides. The plating layers were of an Fe-Al alloy as explained in the following sections. The test pieces were spot welded to 440-MPa class cold-rolled steel sheets of 1.8 mm thickness using DR6φ type electrodes of alumina-dispersed copper at a welding time of 17 cycles (60 Hz) and an electrode force of 400 kgf. **Fig. 7** shows the relationship between the nugget diameter and the current density; the welding current range is roughly 3 kA from the current value at which the nugget diameter becomes $4\sqrt{t}$ relative to the sheet thickness t to that at which expulsion occurs. **Fig. 8** shows the relationship between tensile shear strength and welding current, and **Fig. 9** shows that between cross tension strength and the welding current. Further, **Fig. 10** shows hardness distribution near a nugget. Hardness is approximately HV 480 at the inside of a nugget and the base metal, and it falls to HV 300 or so at heat-affected zones (HAZ). Because of such a hardness distribution, failure occurs at the HAZ in most cases.

Fig. 11 shows the evaluation results of consecutive spot weldability. The test was continued up to 3,000 consecutive spot welds and no significant change in the nugget diameter was observed, and

Fig. 5 Relationship between Charpy impact toughness and test temperature
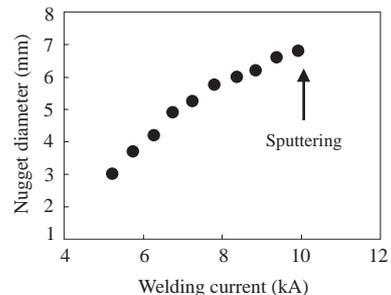
Fig. 7 Relationship between nugget diameter of spot-welded portion and welding current
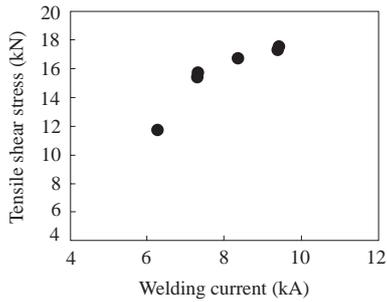
Fig. 8 Relationship between tensile shear strength of spot-welded portion and welding current
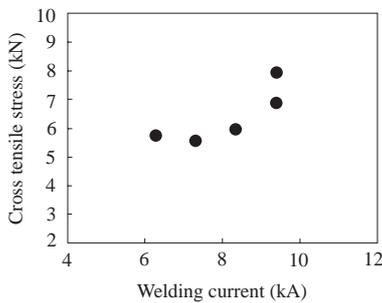
Fig. 9 Relationship between cross tension strength of spot-welded portion and welding current
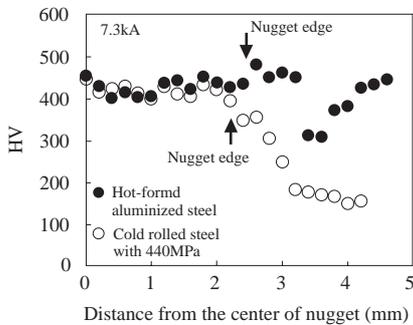
Fig. 10 Hardness distribution near spot-welded portion
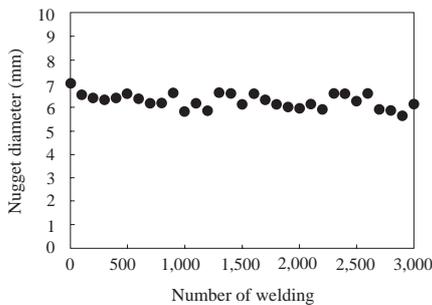
Fig. 11 Change of nugget diameter in consecutive spot welds

more consecutive spot welds were presumed to be attainable. The plating layer of the steel sheet consists of an Fe-Al alloy phase having a melting point higher than 1,000°C, and this is considered to be one of the reasons for the good electrode service life.

No data related to arc-weldability are presented herein. However, since the Fe-Al alloy phase has a boiling point higher than 2,000°C, the occurrence of blowholes, which cause a problem in the arc welding of galvanized steel sheets, does not take place.

6. Coating Layer Characteristics
6.1 Alloying of coating layer during heating

The coating layer of the steel sheet product is what is called the Type 1 coating layer, a plating layer of aluminum containing 10% Si. Its melting point is approximately 600°C, and the layer melts when heated quickly. However, during heating at a heating rate attainable with an electric furnace, an Fe-Al alloy having a high melting point forms from the interface with the base metal, and the alloying quickly reaches the surface and for this reason, no molten alloy sticks to furnace structures. The time that is required for reaching a holding temperature in an electric furnace of normal furnace atmosphere is about 2 min in the case of a 1.6-mm thick aluminized steel sheet.

Fig. 12 shows an alloy layer structure after retaining an aluminized steel sheet at 950°C for 0.5 min, and **Fig. 13** the concentrations of Fe, Al and Si in the plating layer measured in the thickness direction by SEM-EDS. As the alloying proceeds, the coating layer develops a sub-layer structure as shown in the figure. In the case of

20μm
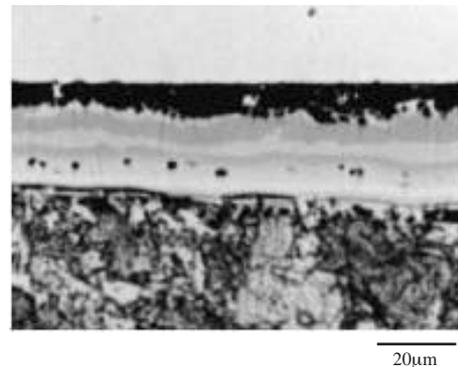
Fig. 12 Sectional optical micrograph showing plating layer structure after heated at 950°C for 30 s
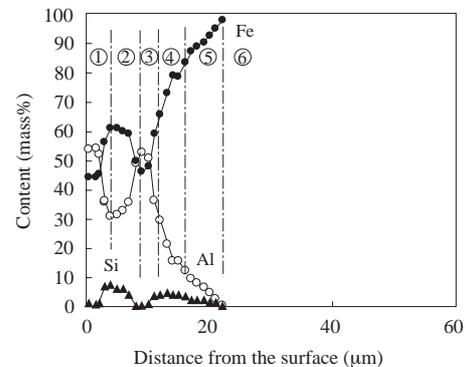
Fig. 13 Change of Fe, Al, and Si concentrations in thickness direction of sample corresponding to Fig. 12

the above heating condition, the plating layer consisted of five sub-layers, and the Al concentrations in the sub-layers were 50, 30, 50, 30 and 10%, respectively, from the surface to inside. The concentrations of the elements changed continuously from the fourth sub-layer to the base metal.

Fig. 14 is a ternary phase diagram of an Fe-Al-Si system at 950°C calculated using Thermo-Culc[7] based on the thermodynamic database of the Fe-Al-Si system presented by Z. Liu et al.[8] The authors superimposed the compositions of the plots of Fig. 13 upon the phase diagram and as a result, the sub-layers in Fig. 13 were identified, presumably, as Fe_2Al_5, an ordered BCC phase, $FeAl_2$, an ordered BCC phase, and a disordered BCC phase, respectively, from the surface to inside. As the holding time becomes longer, Al diffuses into inner layers and finally into the base steel and as a consequence, the sub-layers disappear gradually from the outermost to inside. Fig. 15 shows a plating layer after retaining at the temperature for 5 min. Although only one layer could be observed in sectional observation, an SEM-EDS analysis revealed the existence of two sub-layers; they were presumed to be an ordered and a disordered BCC phase.

6.2 Paintability and corrosion resistance after painting

Steel sheets are used an automobile components after painting by electro-deposition. Usually, they are subjected to a chemical treatment prior to the painting for ensuring good paintability. The chemical treatment generally consists of dipping a steel sheet in a solution of zinc phosphate to form coating films of phosphate on the surfaces. However, the coating films do not form on the surface of the Fe-Al alloy layer because the alloy is very stable against acid. The surface of the Fe-Al alloy phase after the heat treatment is rough as seen in Fig. 16 and thanks to an anchoring effect of the surface, its paintability is good without the chemical treatment. Table 3 shows a comparison of the adhesion of electro-deposited paint of about 20 μm thickness between samples with and without the chemical treatment; it is clear that the aluminized steel sheets hot-formed have sufficiently good paint adhesion regardless of the application or otherwise of the chemical treatment.
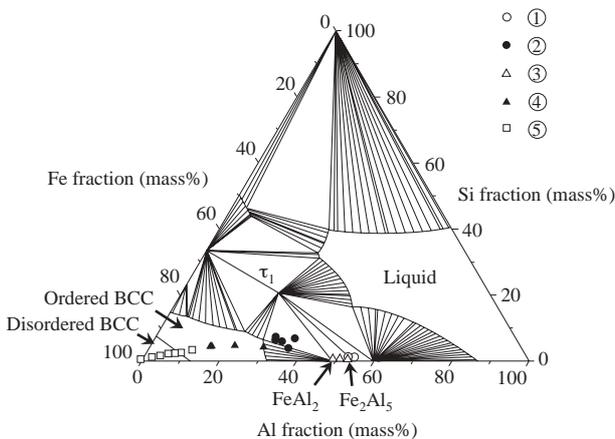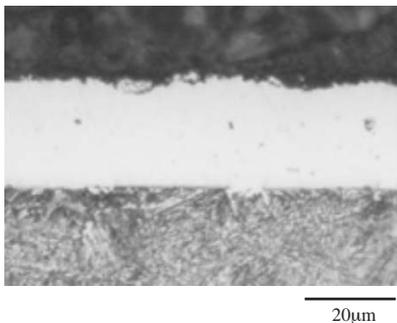
An aluminized steel sheet with a coating weight of 160 g/m² was heated to 900°C for 2 min to form an Fe-Al alloy layer at the surface, immersed in a chemical treatment solution, coated with electro-deposited paint of roughly 25 μm thickness, then, after cross-cutting the paint coating, subjected to 150 cycles (50 days) of the following cyclic corrosion test (CCT) according to JASO M 610:

Salt spray (5% NaCl, 35°C, 2 h) → drying (60°C, 4 h) → wetting (50°C, 2h)

Fig. 17 shows its surface after the CCT together with comparative samples. A cold-rolled steel sheet without surface treatment and a galvannealed steel sheet (coating weight 45 g/m²) were subjected to the chemical treatment and then, after confirming the formation of the chemical coating films, to the CCT as the comparative samples. Fig. 18 shows the change of coating blister of the above three kinds of samples during the course of the CCT, in terms of blister width on one side of a crosscut line. The corrosion depths of these samples were measured in the thickness direction after 150 cycles of the CCT, at 5 points per sample, using a laser beam. The mean values and standard deviations are shown in Fig. 19. It is clear from these results that a steel sheet having coating layers of Fe-Al alloy phases is superior to a cold-rolled steel sheet and equivalent to a galvannealed steel sheet in terms of coating blister width and corrosion depth. When especially good corrosion resistance after paint coating is required, it is desirable to increase the aluminizing coating weight to 150 g/m² or so.

Fig. 14 Isothermal section of Fe-Al-Si ternary phase diagram (at 950°C)

Fig. 15 Sectional optical micrograph showing plating layer structure after retention at 950°C for 5 min
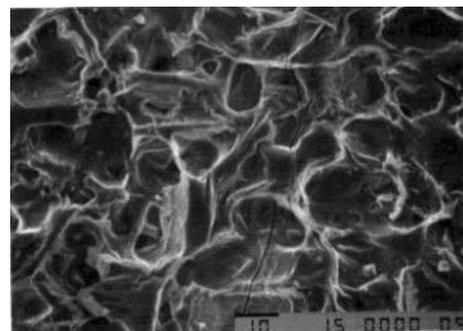
Fig. 16 SEM micrograph showing surface structure of plating layer after retention at 950°C for 5 min

Table 3 Evaluation of paint adhesion

	Surface preparation	Primary adhesion*	Secondary adhesion**
Hot-formed aluminized steel	Phosphated	100/100	100/100
Hot-formed aluminized steel	Degreased	100/100	100/100
Galvannealed steel	Phosphated	100/100	100/100
Steel	Phosphated	100/100	100/100

*Primary adhesion test: 100 grid cross-cutting by 2mm and taping after painting
**Secondary test: dipping at 40°C water for 10 days and cross-cutting and taping

Hot-formed
aluminized steel   Galvannealed steel   Steel
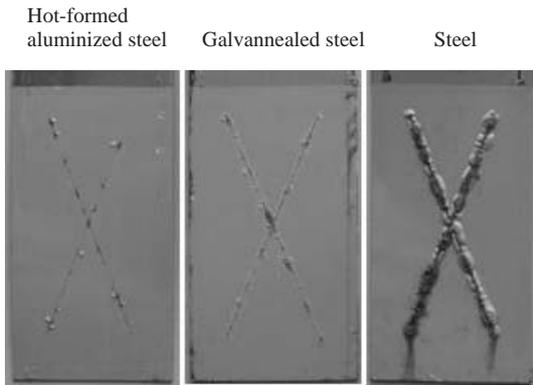
Fig. 17 Appearance of samples after 50 days of CCT according to JASO
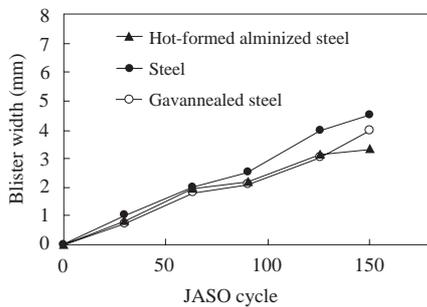
Fig. 18 Change of paint blister width during CCT according to JASO
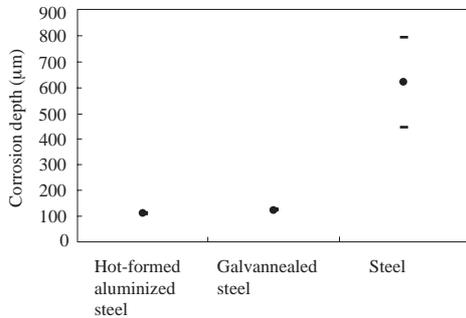
Fig. 19 Change of corrosion depth after 50 days of CCT according to JASO

6.3 Corrosion resistance of spot-welded portion

Fig. 20 shows a sectional structure of a portion just beneath a spot welding electrode and another outside a clamped portion. From these photomicrographs it is clear that the plating layer just beneath an electrode does not change in spite of the welding heat and retains its thickness throughout the welding process. For this reason, the corrosion resistance of these portions after paint coating is the same as that of the other portions. **Fig. 21** shows samples of the aluminized steel sheet with paint coating after heat-treatment of hot-forming including spot welded portions after 2,000 h of a salt spray test (SST); no corrosion is observed in any of them. In the case of other coated steel sheet products, the plating layer beneath an electrode may melt during spot welding, be squeezed out from beneath the electrode, and the plating metal can sometimes form protrusions around the portion. At such portions, paint coating applied thereafter

Alloyed layer
Base steel

Just beneath electrode   Out of spot welding
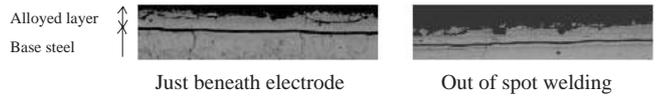
Fig. 20 Plating layers just beneath spot welding electrode and other portion (etched with bromic acid)
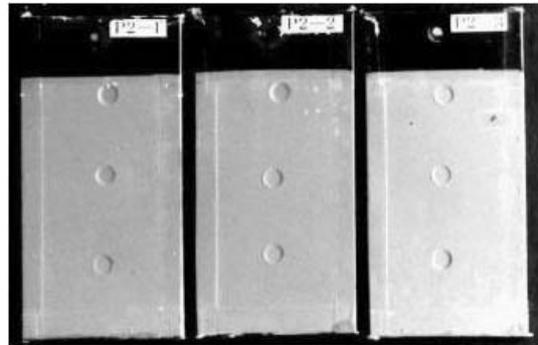
Fig. 21 Appearance of spot-welded and painted portions after SST for 2,000 h

may become thinner and there may be problems in corrosion resistance.

7. Summary

The properties of the base material and surface properties of an aluminized steel sheet for hot forming use have been explained herein. Even if the cooling rate at the die quenching is uneven during the press formation process, the base material has uniform strength after the hot forming as far as a cooling rate of 30°C/s or higher is attained. A yield strength of 1,280 MPa, a tensile strength of 1,530 MPa and an elongation of 8% can be obtained after the hot forming. The aluminum plating layer of the product transforms during the heating before the press forming into an Fe-Al alloy phase having a high melting point, and thanks to this, the spot weldability of the product is not adversely affected by the presence of coating layers. As a consequence, the consecutive spot weldability of the aluminized steel sheet product is excellent. The Fe-Al alloy phase displays a better corrosion resistance than a steel sheet without surface treatment and almost equivalent corrosion resistance to a galvannealed steel sheet.

The application of the steel sheet product having the above properties to the hot-forming process will make it possible to produce automobile components excellent in strength and corrosion resistance and having comparatively complicated shapes.

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