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Sliding characteristics of hot-dip galvanized steel sheets depending on aging time after production

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Abstract. The effect of aging time after production on the frictional characteristics of hot-dip galvanized steel sheets (HDG) was examined in this study to stabilize and improve the press formability of HDG. Conventional HDG with the Zn coating weight of 67 g/m^2 including 0.36 mass% Al were used as test specimens. The specimens were aged for a predetermined period, after which their friction coefficients were measured with a flat sliding test. It was found that an Al-based oxide grew on the HDG surface and the friction coefficient tended to decrease with increased aging time. This suggests that the Al-based oxide reduced the friction coefficient. To understand how the Al-based oxide affects the friction coefficient, surface observation and analysis of both the test specimens and the tools after sliding were carried out. Although the Al-based oxide was not detected on the HDG surface in the area of contact with the sliding tool, the oxide was detected as adhering material on the tool surface after sliding, suggesting that the adhering material on the tool reduced the adhesion force between the metallic Zn of the HDG and the tool. This could lead to a lower friction coefficient because shearing resistance was decreased.

1. Introduction

Hot-dip galvanized steel sheets (HDG) are widely used in countries around the world for automotive body parts due to those high corrosion resistance. Because HDG is used after press forming, its frictional characteristics, which affect press formability, are one important type of characteristic for users. It is generally said that the friction coefficient of HDG is higher than that of cold rolled steel sheets (CRS) due to the higher adhesion force between the metallic Zn in the HDG coating layer and metallic sliding tools [1, 2].

Galvannealed steel sheets (GA) and electrogalvanized steel sheets (EG), which are also used as automotive materials, have friction coefficients similar to that of HDG. Therefore, various lubrication treatments for GA and EG had been developed and applied practically to stabilize press forming. For example, as lubrication treatment, a surface modification technology [3-6], Mn-P-based inorganic film [7] and other organic and inorganic coatings [8-10] were developed and demonstrated on GA, and the resulting frictional behavior and press formability were reported. A pre-phosphate treatment was developed for EG and is widely used as a lubrication treatment in countries around the world [11]. Although the frictional behavior of HDG has also been investigated widely, few reports have examined lubrication treatment of HDG [12].

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Regarding the frictional behavior of HDG, it is known that the surface roughness of HDG affects the friction coefficient [13], and a technology for controlling the surface texture by temper rolling in order to control surface roughness has also been reported [14]. Hoshino et al. reported that Al-based oxides which segregate on the HDG surface decrease the friction coefficient [15, 16]. Generally, less than 1 mass% of Al is added to molten Zn in the HDG manufacturing process to decrease dross and to prevent Zn from alloying with the base steel [17, 18]. Although the amount of added Al is small, it was reported that this Al segregates on the HDG surface as oxides with increasing aging time [19]. However, the effect of aging time after production on the friction coefficient of HDG has not yet been clarified.

In this study, the effect of aging time on the friction coefficient and segregation of Al-based oxides was investigated in order to stabilize and improve the press formability of HDG and to obtain clues for the development of a new lubrication treatment for HDG.

2. Experimental procedure

2.1. Test specimens

An annealed steel sheet (IF steel) with the thickness of 0.7 mm was hot-dip galvanized, and was then temper rolled with a dull roll. This process was performed continuously. The time when temper rolling was performed was defined as the starting time of aging. The test specimens were aged for a certain time period in a desiccator without oil. The coating weight of the Zn, the Al content in the Zn coating layer and the arithmetic average roughness (Ra) of the evaluated side of the test specimens were 67 g/m², 0.36 mass% and 0.94 μ m, respectively. These values were constant, independent of aging time.

2.2. Flat sliding test

Friction coefficients were measured with a flat sliding test. A schematic diagram of the flat sliding test is shown in figure 1. The test specimen (29 mm x 220 mm) was set, and the tool (tool steel: SKD 11) was pressed with a constant normal load N. The specimen was drawn at a constant speed, and the drawing force F was measured. The friction coefficient was calculated by F/N. As test conditions, a contact area of 3 mm x 10 mm, sliding length of 130 mm, contact pressure of 130.4 MPa and sliding speed of 1.0 m/min were used in this study as this condition is susceptible to Al-based oxide on HDG [15, 16]. Before the test, the tools were polished with #2000 polishing paper orthogonally to the sliding direction. The specimens were ultrasonically degreased in alcohol, and then 2 g/m² of lubricant oil measured with a gravimetric method was applied before the sliding test. The lubricant oil was a commercial washing oil with the viscosity of 2.0 cSt at 40 °C.



Figure 1. Schematic diagram of flat sliding test.

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Three test specimens with the same history were prepared, and their friction coefficients were measured continuously in order to simulate the continuous press forming process used in industry. The friction coefficient was defined as the average value of the friction coefficients of these three specimens. The lubricant oil was applied to all the test specimens before the test, but the measurements were performed without polishing the tool between the three specimens.

2.3. Surface observation and analysis

The fluorescence X-ray intensity of Al of the test specimens before the sliding test was measured with an X-ray fluorescence spectrometer (XRF), ZSX-101E (Rigaku). The tube voltage of 45 kV, tube current of 45 mA and measured area of a 35 mm diameter circle were employed.

Al and O on the surfaces of the specimens and tools before and after the sliding test were observed and analyzed with a scanning electron microscope (SEM) and an electron probe micro-analyzer (EPMA), JXA-8100 (JEOL). The acceleration voltage of 15 kV, irradiation current of 0.1 μ A and integration time of 50 ms were employed. Regarding the specimens and tools after sliding, the center of third specimen and the tool after sliding three specimens were observed and analyzed.

3. Results

3.1. Friction coefficient and segregation behavior of Al-based oxides depending on aging time

The measured friction coefficients of HDG are shown in figure 2 as a function of aging time after production. The friction coefficient tended to decrease as the aging time increasing until 1 200 h, and then became saturated at around 0.09.



Figure 2. Relationship between friction coefficient of HDG and aging time after production.

Figure 3 shows the Al intensity of the HDG surface measured with XRF as a function of the aging time after production. The Al intensity tended to increase as the aging time increased, and it did not become saturated over 3 000 h. The intensity of Al after 24 h of aging was about 0.69 kcps and increased to approximately 15.1 kcps after 3 000 h of aging. This behavior is similar to that observed in previous reports [19] investigating the segregation behavior of the Al-based oxide layer on the HDG surface.

The surfaces of the HDG after 24 h, 360 h and 3 000 h of aging were observed with an SEM, and the Al and O were analyzed with an EPMA. The results are shown in figure 4. Although the morphology was constant independent of the aging time, as shown in the figure, a significant difference was observed in the analytical results of Al and O. Although the intensity of Al of the HDG after 24 h was low, some regions with higher Al intensities were observed on the HDG aged for 360 h

and 3 000 h. Moreover, the Al intensity of HDG after 3 000 h was higher than that of HDG after 360 h, suggesting that Al which segregated on the HDG surface increased with increasing aging time. The distributions of Al and O were substantially the same, indicating that Al segregated as an oxide.



Figure 3. Relationship between Al intensity and aging time after production.



Figure 4. SEM images of surface of HDG aged for (a) 24 h, (b) 360 h and (c) 3 000 h after production. The EPMA intensity mapping of (d-f) Al and (g-i) O of each SEM image are shown at the right.

Figure 5 shows the SEM images of HDG surface before and after temper rolling. It is well known that hollows are formed on HDG surface after temper rolling by contacting with temper roll. This suggest that the segregated Al-based oxides which is observed in figure 4 tended to segregate on areas where contact with a temper roll occurred because Al-based oxides were detected in the hollows which was formed by contacting with temper roll. It is assumed that this area was easily oxidized because the fresh Zn surface was exposed by a contact with the temper roll.

Figure 5. SEM images before and after temper rolling

3.2. Effect of Al-based oxide on friction coefficient

The similarity between the trends of the friction coefficient (figure 2) and the Al intensity of HDG (figure 3) as a function of aging time after production suggest that the Al-based oxides which segregated on the HDG surface affected the friction coefficient. The data provided in figure 6 further support this relationship. Essentially, the friction coefficient tended to decrease as the Al intensity increased. This suggests a decrease in the friction coefficient with increasing Al-based oxide layer thickness. However, above 12.3 kcps of Al intensity, the friction coefficient was saturated at around 0.09. This could be related to the mechanism of reduction of the friction coefficient by the segregated Al-based oxide on HDG.

Figure 6. Relationship between friction coefficient of HDG and Al intensity measured with XRF.

4. Discussion

4.1. Surface observation and analysis of HDG after sliding

The surface of the HDG after sliding was observed with the SEM and analyzed by EPMA. The results are shown in figure 7. A flattened area containing micro-scratches can be observed in the SEM images. As this flattened area was not observed in the SEM images before sliding (figure 4), it can be said that it was formed by actual contact with the sliding tool, and the other areas are areas of non-contact with the sliding tool, in which the original HDG surface remained after sliding. No significant difference can be observed in the appearance of the specimens in the SEM images depending on aging time, as all the test specimens display similar areas of both contact and non-contact with the sliding tool. From the analytical results of Al and O obtained by EPMA, although segregation of Al-based oxides can be observed in the non-contact areas with the tool, the intensities of Al in the contact areas are lower than those in the non-contact areas. In the contact areas with the tool, even the Al intensity level of the

HDG aged for 3 000 h was similar to that of the HDG aged for 24 h, suggesting that the Al-based oxides which existed on the HDG surface before sliding were mechanically plowed by the sliding tool.

4.2. Surface observation and analysis of tools after sliding

The tool surfaces before and after sliding were analyzed by EPMA. Figure 8 shows the analytical results of Al and O on all the tool surfaces which were in contact with HDG. An area with high intensities of Al and O was observed on the tool surface after sliding, when the intensities of Al and O on the tool before sliding were taken into account as background. Considering both the results of analysis of the tool surface and the HDG surface as mentioned above, the Al-based oxides which existed on the HDG surface were removed by the sliding tool and then adhered to the tool surface. Distributions of Al and O were observed only at the head end of the tool surface after sliding on the HDG aged for 24 h, and the distributions of Al and O extended to the tail end of the tool as aging time increase. The distributions on the tools after sliding on the HDG aged for 3 000 h correspond to the entire area in contact with the HDG surface. This difference could be derived from the difference in the amount of Al-based oxides which originally existed on the HDG surface.

In other words, because the amount of Al-based oxides on the surface of the HDG aged for 24 h was smaller, all the Al-based oxides on the specimen were removed by the head end of the tool and adhered to the tool, after which the exposed metallic Zn came into contact with the tool surface without adhering Al-based oxides. However, the HDG aged for 3 000 h had a larger amount of Al-based oxides on its surface. Therefore, when this material was used in test specimens, the Al-based oxides were removed and adhered to the tool in the same manner, but due to the larger amount of Al-based oxides which originally existed on the HDG surface, the Al-based oxides adhered to not only the head end, but also up to the tail end, essentially covering the entire tool surface, and this condition of coverage with Al-based oxides could lead to the decrease in the friction coefficient.

Figure 7. SEM images of surface of HDG after sliding, showing specimens aged for (a) 24 h, (b) 360 h and (c) 3 000 h after production. The EPMA intensity mapping of (d-f) Al and (g-i) O of each SEM image are shown at the right. The black arrow on the right side of (i) shows the direction of tool movement.

Figure 8. EPMA intensity mapping of (a-d) Al and (e-h) O on tool surface, (a, e) before sliding and after sliding on HDG aged for (b, f) 24 h, (c, g) 360 h and (d, h) 3 000 h. The black arrow on the right side of (d) and (h) shows the direction of tool movement.

4.3. Sliding model

Figure 9 shows a schematic sliding model which describes the effect of the Al-based oxide layer on the frictional properties of HDG based on the results of this study. An Al-based oxide layer, which segregates as aging time increases, exists on the HDG surface before sliding. A thicker Al-based oxide layer exists in the contact area with the temper roll and a thinner oxide layer could exist in the non-contact area. During sliding, the Al-based oxides on the HDG surface are plowed by the micro-ridges of the tool surface, which were introduced by polishing with polishing paper, and those oxides adhere to the tool surface like covering the tool surface. Therefore, the friction coefficient decreases because the shearing force decreases due to metal-to-oxide contact, which has a lower adhesion force than metal-to-metal contact [20]. Because the tool surface can be fully covered with the Al-based oxide by sliding, the friction coefficient reaches saturation at around 0.09 independent of the aging time over 1 200 h, as shown in figures 2 and 4.

Figure 9. Schematic illustration of sliding model for HDG.

5. Conclusions

In order to stabilize and improve the press formability of HDG and to obtain clues for the development of a new lubrication treatment for HDG, the effect of aging time on the friction coefficient and segregation behavior of Al-based oxides was investigated.

- (1) The Al-based oxides on the HDG tended to increase as aging time increased. The Al-based oxides tended to segregate on areas where contact with the temper roll occurred.
- (2) The friction coefficient of the HDG tended to decrease with an increase of aging time. However, the friction coefficient became saturated at around an aging time of 1 200 h.
- (3) The Al-based oxides on the HDG surface were plowed by the tool during sliding and accumulated on the tool surface. The tool surface was then covered with the Al-based oxides

by subsequent sliding.

- (4) The friction coefficient of the HDG decreased because the interface changed from metal-tometal contact, which is characterized by higher adhesion force, to metal-to-oxide contact, which has lower adhesion force.
- (5) The tool surface could be fully covered with the Al-based oxide by sliding on HDG aged for over 1 200 h, and the friction coefficient reached saturation at this aging time.
- (6) Focusing on controlling the adhering materials to the tool surface by Al-based oxides on HDG can be clues to develop a new lubrication treatment system for HDG with a thin film.

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