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Galvanizability and Corrosion Properties of ZnMgAl Hop-Dip Coating with Shot Blasting Pre-Treatment

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We investigated the wetting behaviors of ZnMgAl ternary alloy coatings using the sessile drop method, varying the composition. The static contact angles of various ZnMgAl droplets on a steel substrate, measured after 10 seconds, ranged from 85.2° to 115.7°, significantly higher than the 35.7° observed for standard galvanized coating (GI). Additionally, the ZnMgAl droplets did not spread easily over time. We hypothesize that surface pre-treatment is essential for enhancing the surface and interface properties of the ZnMgAl coating. To study the effects of pre-treatment through shot blasting on microstructures and corrosion resistance, we employed field emission-scanning electron microscopy (FE-SEM), synchrotron X-ray diffraction (SXRD), surface profile analysis, and salt spray tests (SST). The quality of the coated layer could be adjusted by altering the impact force of shot balls on the hot-rolled substrate. The corrosion resistance of the ZnMgAl coating improved due to the formation of a dimple-like structure on the surface. This organized surface structure is crucial for creating pockets that retain post-treated chromate materials.

Keywords: *ZnMgAl coating, Shot blasting, Corrosion resistance*

1. Introduction

In the last few decades, numerous investigations have been focused on the improvement of corrosion resistance of hot dip galvanized steel by addition of alloying elements in the galvanizing bath. It was revealed that Mg and Al had significant effects on the corrosion resistance of steel coatings. In Japanese industry, the Mg and Al contents in Zn bath varies from 0.1 to 3.0 wt% and 0.2 to 11.0 wt%, respectively (hereafter "wt%" is presented simply as "%") [1-4]. In contrast, European researchers developed the ZnMgAl coatings including a little amount of alloy elements with 1.0 ~ 2.0%Mg and 0.4~2.0%Al [5-8]. Recently, we have developed the hot-dip alloyed steel sheet having the coating composition of Zn-3.0%Mg-2.5%Al. It has an excellent corrosion resistance than general galvanized steel sheet because of having both the sacrificial ability of Zn and the passive properties of Al. Especially, we used the shot blasting pre-treatment as roles of removing scale before continuous pickling process on hot-rolled strip and inducing method of compressive stress on steel substrate [9].

In this work, the wetting properties of ZnMgAl coating with changing contents will be discussed including the effect of shot blasting pre-treatment on corrosion resistance and galvanizability of ZnMgAl.

2. Experiment

2.1 Methods of Sessile Drop Test

Wettability of the ZnMgAl droplets was characterized by measuring the contact angle of a liquid droplet, more specifically a sessile drop method, on a steel substrate. The evolution of droplet curvature was verified by optical imaging as a function of time. The experiment was carried out with changing droplet compositions. Nominal coating compositions were Zn-0.2%Al (GI), Zn-1.7%Mg-1.7%Al, Zn-3.0%Mg-2.0%Al, Zn-3.0%Mg-2.5%Al and Zn- 3.0%Mg-6.0%Al, respectively. The sample substrate was used a typical carbon steel as a conventional quality grade (C 0.04%). The annealing cycles for the sample substrate and droplet including individual ambient

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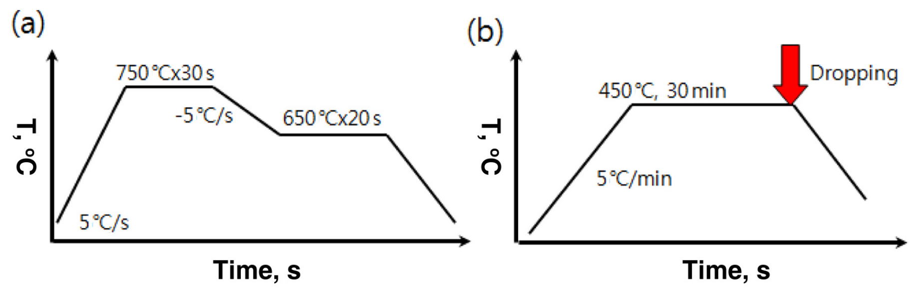


Parameter	Value
Annealing Temp.	750℃
Atmosphere	15%H_2-N_2
Dew Point (℃)	-40℃

Parameter	Value
Wetting Temp.	450℃
Atmosphere	100%H_2
Dew Point (℃)	-40℃

Fig. 1. Parameters of experimental sessile drop. (a) Annealing cycle of the sample substrate, (b) Annealing cycle of the liquid metal droplet

condition are described in Fig. 1. The temperature of droplet and substrate was equal when the contact was initiated.

2.2 Materials

The ZnMgAl coatings in this study were produced by hot dip galvanizing of hot-rolled steel on the production line of POSCO #1CGL at Pohang. The test coils having tensile strength of 540 MPa were used. The hot-rolled coils were continuously transferred to the shot blasting machines, so that the approximately 50% of the total amounts of mill scale was removed. The shot blasting pre-treatment was performed by changing the wheel speed of shot blaster from 0 to 1800 rpm (rotating per minute) and the material of the short ball (SAE-S110) is low carbon steel. After that the strip was passing through two short chambers filled with HCl. The pickled fresh surface of steel sheets was annealed in the reduction furnace and subsequently put into the bath. The detail composition for galvanizing bath was 94.5% of Zn, 3.0% of Mg and 2.5% of Al. The average coating weight 190 g/m^2 for both sides. Sample sheets were post-treated by roll coating with chromate materials.

2.3 Characterization methods

Cross-sections of test samples were prepared and analyzed with FE-SEM. The FE-SEM experiments were studied by using the back-scattered electron mode to clearly distinguish the various metallic phases. For the FE-SEM investigations, a Zeiss Supra 55 scanning electron microscope was used. The synchrotron x-ray diffraction (SXRD) analysis in order to investigate the crystal structure was performed in Pohang Accelerator Laboratory (PAL). X-ray energy and wavelength were 8.8 keV and 0.14089 nm, respectively. The diffraction profile was measured along the out of plane direction of momentum transfer, q_z. The surface roughness and profiles were measured by 3D surface profiler, Veeco NT-8000. The measuring area was 3.5 mm(L) × 4.7 mm (w). Coating amount and exact compositions of coatings were analyzed by Inductively Coupled Plasma (ICP) spectroscopy. Solutions were prepared by dissolving the coating materials with the mixture of 35% HCl and distilled water (HCl : water = 3 : 1). In order to compare the surface appearance, the brightness of sample surface was measured by TRI-Glossmaster while maintaining the same incidence angle of 60º.

2.4 Corrosion test

To investigate of the corrosion behaviors taking place on the ZnMgAl, a standard salt spray testing system, SUGA STP-200 was used. As specified in the standard a 5% sodium chloride solution, made up to pH 6.7 by adding sodium hydroxide. The average amounts of sprayed solution were controlled 1.5 ± 0.5ml/h. The testing chamber was kept at a constant temperature of 34.5 ± 0.5 ºC. The tested specimens were evaluated for corrosion resistance according to ISO9227 [10].

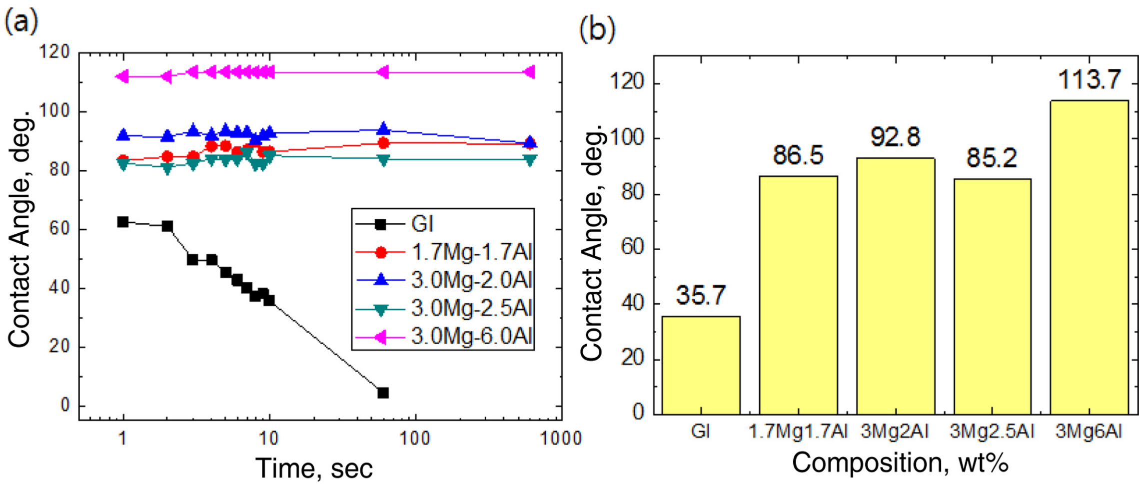


Fig. 2. (a) Evolution of contact angle at different droplet compositions, (b) Static contact angle of droplet after 10 secs exposure on substrate

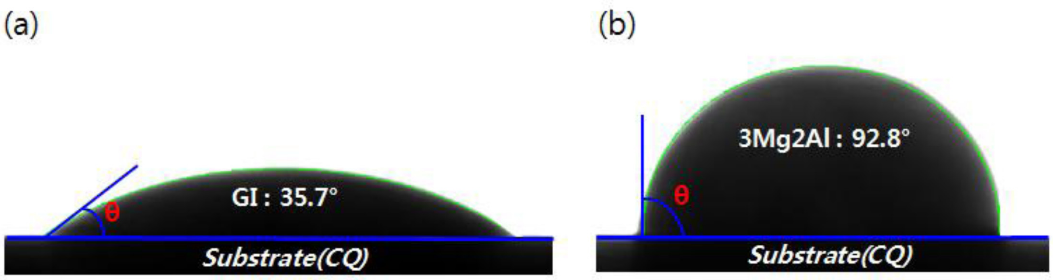


Fig. 3. Comparison of contact angle after 10 secs exposure on substrate at different droplet compositions (a) GI: 35.7° (b) Zn-3.0%Mg-2.0%Al: 92.8°

3. Results

3.1 Wettability of Zn-Mg-Al Coatings

Fig. 2a indicates the evolutions of contact angle as a function of time. The considerable difference of wetting kinetics between GI and ZnMgAl coating materials is clearly shown. In the case of GI, the contact angle is rapidly decreased while the contact time increases gradually. A descending slope of GI as depicted in Fig. 2a indicates progressed wetting between a droplet and a substrate, also shows the enhanced reactivity due to the growth of inter-metallic compounds at the interface. In contrast, the contact angles of ZnMgAl coatings are shown having higher values than it of GI. The proceeding angles are not changed subsequently and kept nearly motionless values. The measuring results of contact angles after contacting time of 10 secs show in Fig. 2b. The contact angle of GI droplet and that of ternary alloyed Zn-3.0%Mg-6.0%Al is measured 35.7° and 113.7°, respectively. The example of real optical images GI and ZnMgAl droplets illustrates in Fig. 3. The comparison between the results of ZnMgAl and GI materials generally means that the Mg and Al addition to the galvanizing bath deteriorates wettability of steel substrate. On the other hand, the contact angle of Zn-3.0%Mg-2.5%Al droplet is slightly smaller than others of having different ZnMgAl contents in experiment. For that reason, we believe that the Zn-3.0%Mg-2.5%Al would be a promising content for improving wetting behaviors of ternary alloy system.

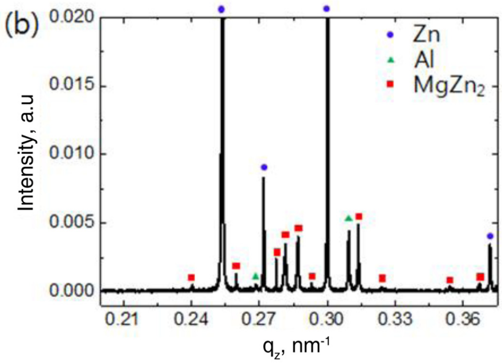
3.2 Microstructure of Zn-Mg-Al Coatings and Influence of Shot Blasting

Fig. 4a shows the experimental conditions of shot blasting pre-treatment and temperature of strip into the bath. The measured x-ray diffraction profile of Zn-3.0%Mg-2.5%Al coating is shown in Fig. 4b. Analyzing result of the Bragg peaks clearly indicates Zn, Al and $MgZn_2$ crystal phases, Mg is not existed as a single phase but it makes an inter-metallic compound with Zn as forming a meta-stable $MgZn_2$. The presence of Mg_2Zn_{11} was not observed which was expected from the equilibrium [11]. The dependence of microstructure on the wheel-speed of shot blasting machine and strip temperature to the bath is illustrated in Fig. 4c, d and e, respectively. Regardless of experimental condition, cross-sectional analysis in microstructure of Zn-3.0 %Mg-2.5%Al coatings indicates that Mg and Al containing Zn alloy coated layer consists of Zn single phase, Zn/$MgZn_2$ binary eutectic and Zn/Al/$MgZn_2$ ternary eutectic phases. From Fig. 4d, we also observe that the micro-structural size of Zn/$MgZn_2$ binary eutectic increases with increasing impact force of shot treating and strip

(a)

No	Shot Blasting Wheel Speed(rpm)	Temperature of strip into the bath(°C)
1	1600	445
2	1800	480
3	0	480

(b)



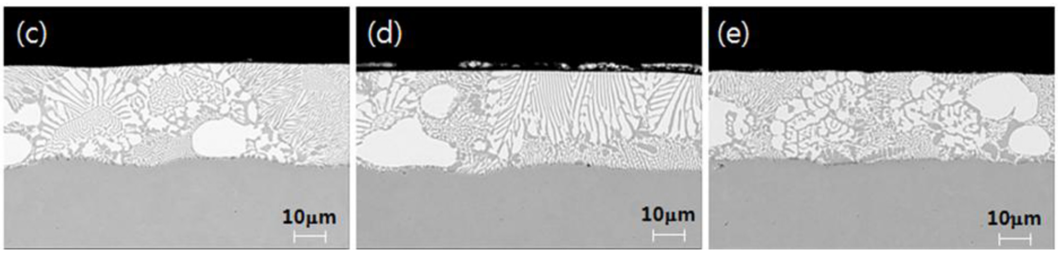


Fig. 4. (a) Table of experimental conditions, (b) Synchrotron x-ray diffraction pattern of Zn-3.0%Mg-2.5%Al coating, (c), (d), (e) Cross sectional microstructure of Zn-3.0 %Mg-2.5 %Al coating by changing conditions according to 1, 2 and 3

(a)

No	Brightness (G. U)	Surface roughness		Coating Weight (g/m^2)	Composition(wt%)				Chromate Coating Weight (mg/m^2)
		Ra(μm)	Rz(μm)		Zn	Al	Mg	Fe	
1	96-103	1.3	17.3	193	93.9	2.77	3.26	0.03	50.6
2	135-142	1.6	18.4	199	93.9	2.80	3.30	0.03	53.9
3	115-122	0.8	12.4	182	94.0	2.74	3.26	0.04	40.5

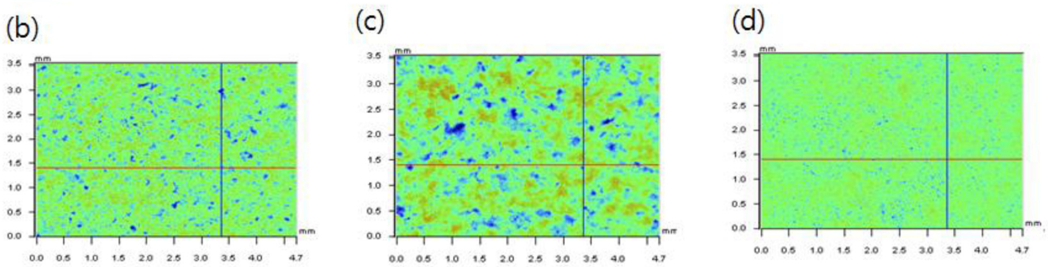


Fig. 5. (a) Table of experimental results (Brightness, Surface roughness, Coating weight, Coating composition and Chromate coating weight), (b), (c), (d) 2-D Surface roughness profile according to 1. 2 and 3 experimental condition

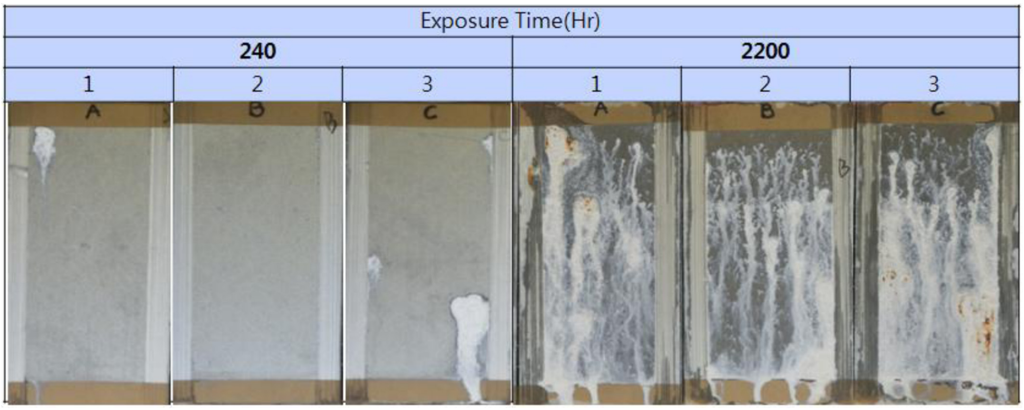


Fig. 6. Salt spray test results of hot-dip galvanized Zn-3.0%Mg-2.5%Al sheets

temperature into the bath. We can speculate the enhancement of inter-atomic interaction through the compressive stress of the strip and temperature to grow a binary eutectic microstructure of ZnMgAl coatings. When strip goes into the pot, the compressive stress is relieved, making the micro-structure larger.

Fig. 5 illustrates the surface roughness and profiles according to experimental conditions. The intense impact force can modify the roughness profile from smooth to rough surface. For this reason, the surface appearance can be improved brightly by increasing the impact force and strip temperature because the amount of light to non-directionally scattered reflection from modified rough surface would be increased consequently. Fig. 5a shows the results of surface brightness with changing the experimental conditions.

3.3 Corrosion Properties of ZnMgAl Coatings

Fig. 6 shows the result of corrosion by SST. The white and red rusts do not appear for 200 hours and 2000 hours after salt spray exposure, respectively. It is obvious that the anti-corrosion property of strongly impacted sample by shot blasting is considerably improved. The amount of chromate is slightly larger than that of non-shot blasting or slightly treated sample. The dimple-like structure formed on surface can be a containing pocket of the post-treated chromate materials from the result of Fig. 5a.

4. Conclusions

The wettability of ternary alloy ZnMgAl hot dip coating and the effect of shot blasting pre-treatment on galvanizability have been studied. Conclusions drawn from this study are summarized below:

· The addition of Mg and Al as alloying elements to the galvanizing bath deteriorates wetting properties of steel substrate.

· The wetting angle of Zn-3.0%Mg-2.5%Al droplet is slightly smaller than those of having different compositions of ZnMgAl coatings.

· The microstructures of Zn-3.0%Mg-2.5%Al coating are composed of Zn single phase, Zn/$MgZn_2$ binary eutectic and Zn/Al/$MgZn_2$ ternary eutectic phases.

· The dimple-like surface structure formed by pre-treated impact of shot ball is helpful to enhance the corrosion resistance and surface appearance of ZnMgAl coatings.

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