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Thesis for the Degree of Master of Engineering

Effect of Hot-stamping on the Phase Change of Coating Layer in High-strength Steel Plate

by

Jong-Hoi Woo

Department of Materials Science and Engineering

Graduate School of Industry

Pukyong National University

February 2021

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**(고강도강판 코팅층의 상변화에 미치는
핫 스탬핑의 영향)**

Advisor : Prof. Ki Woo Nam

by

Jong-Hoi Woo

A thesis submitted in partial fulfillment of the requirements for the degree
of Master of Engineering

Department of Materials Science and Engineering

Graduate School of Industry

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A Dissertation by

Jong-Hoi Woo

Approved by

Chang-Yong Kang

(Chairman)

Eun-Jong Oh

(Member)

Ki-Woo Nam

(Member)

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Abstract

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Jong-Hoi Woo

Department of Material Science and Engineering, Graduate School,
Pukyong National University

In this study, the effect of hot-stamping forming process on the properties change of compound layer in Al-Si coated high-strength steel sheet with different thickness was investigated. The coating layer in hot-stamped high-strength steel sheet was composed of a diffusion layer and a compound layer. As the hot-stamping temperature and holding time for forming increased, the thickness of the compound layer in the Al-Si coated high-strength steel sheet increased. The thickness of sample did not affect the thickness of the compound layer. The compound layer in hot-stamped high-strength steel sheet with Al-Si coating layer consists of $\text{Al}_{15}\text{Fe}_2$ and Fe_2Si phases. In the compound layer, Al and Si were irregularly distributed.

Keyword: hot-stamping; coating layer; diffusion layer; high-strength steel sheet; heating time; heating temperature

1. Introduction

Recently, transportation equipment related industries are trying to solve problems such as the regulation of fossil fuel use as an environmental problem, and resource saving due to fuel exhaustion. [1,2] The most efficient method to solve this problem is to improve fuel efficiency by lowering the weight of transportation equipment.

To date, strategies to improve fuel efficiency in the transportation equipment industry are to improve the efficiency of transportation equipment components, reduce rolling resistance, and lower the weight of transportation equipment. [3,4] Improvement in component efficiency and fuel efficiency by the reduction of rolling resistance have reached their limit. Therefore, the lowering the weight of transportation equipment is the most efficient method to improve fuel efficiency.

Currently, various methods are being used to attempt to reduce the weight of transportation equipment. That is, materials for transportation equipment components are replaced by materials with higher strength and toughness than conventional materials, or ferrous alloys with high specific gravity are replaced by non-ferrous alloys with high specific strength. These studies are being actively conducted. [5-8] However, if the desired

conversion is to a high-strength material or a material having a high specific strength, material development having such properties is also important. However, various optimal manufacturing process technologies must be developed according to manufacturing the components of transportation equipment using the developed materials. In addition, to improve the product reliability and achieve the reduction of cost in manufacturing the components, development of this optimal manufacturing process technology is urgently required. Since as the strength of steel sheet increases, elongation decreases, it is difficult for the hot-stamping method to mold components. [9-11] Therefore the hot-stamping method overcomes this problem, and prevents cracks occurring during the molding of coated high-strength steel sheet, thereby ensuring high-strength and formability at the same time. [12-14]

Therefore, this study was carried out to obtain basic data for the development of the optimal process required for manufacturing components by the hot-stamping method. That is, the coated SABC 1470 steel sheet was heated at different temperatures and times. Then, after the PHS products were manufactured by the hot-stamping method, the thickness and microstructural change of the coating layer according to the change of the heating temperature and the holding time were investigated.

2. Samples and experimental methods

2.1 Sample and hot-stamping molding

The sample used in the study was used by coating the SABC 1470 steel sheet with a coating solution of 40 % Al – 10 % Si–Fe. The hot-stamping was performed at heating temperatures of (890, 910, and 950) °C with the coated steel sheet, and the holding time at each temperature was (180–850) s.

2.2 Microstructure observation

The microstructure changes of the base metal and the coating layer according to the heating temperature and the holding time for hot-stamping were observed by optical microscopy and scanning electron microscopy (SEM).

2.3 Thickness measurement of coating layer

The thickness change of the coating layer according to the changes of heating temperature and holding time was measured by hardness test and line profile analysis of Electron Probe Micro Analysis (EPMA). Measurement by the hardness test was measured at constant interval from the surface of the coating layer using a micro Vickers hardness tester, and the thickness was

measured 5 times, and averaged. In addition, the analysis of EPMA (S-2400) was measured by examining the concentration change of Al, Si, C, and Fe by line analysis inside the sample surface.

2.4 Microstructure observation of the coating layer (alloy layer)

The phases in the coating layer (alloy layer) of the hot-stamping sample were observed using X-ray diffraction and transmission electron microscopy (TEM). X-ray diffraction (Rigaku D/Max-IIA) was analyzed at 0.04° intervals in the Bragg angle range $2\theta = (20-100)^\circ$ using Mo- $K\alpha$ characteristic X-ray. The compound analysis was conducted using the peak corresponding to each phase from the diffraction curve. TEM (Hitach, 200 kV) observation was carried out by polishing the hot-stamped sample, and making a thin film by jet-polishing.

3. Experimental results and discussions

3.1 *Microstructure observation*

Figure 1 shows the microstructure of SABC 1470 steel sheet coated for hot-stamping observed by optical microscopy, in which A is the coating layer, and B is a SABC 1470 steel sheet composed of martensite, pearlite, and ferrite structures.



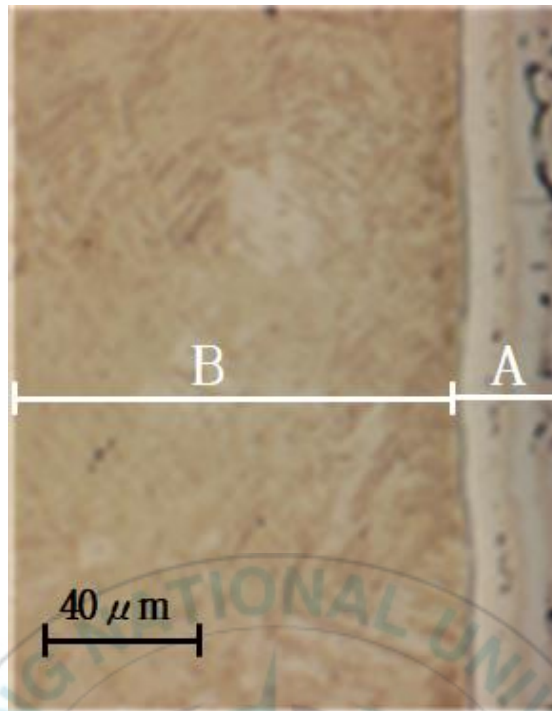


Figure 1. Optical micrograph of the SABC 1470 steel sheet with coating layer.

3.2 Effect of hot-stamping on the microstructure of the coating layer

Figure 2 shows the microstructure of the sample subjected to hot-stamping after heating the coated SABC 1470 steel sheet at 910 °C for 500 s. The microstructure is composed of (a) a compound layer, (b) a diffusion layer, and (c) a SABC 1470 steel sheet having martensite structure. From this result, it can be seen that when the coated steel sheet is hot-stamped, the coating layer is composed of a compound layer and a diffusion layer. In addition, the compound layer as well as the diffusion layer must be strictly managed, because it acts as a cause of crack in the hot-stamping process.

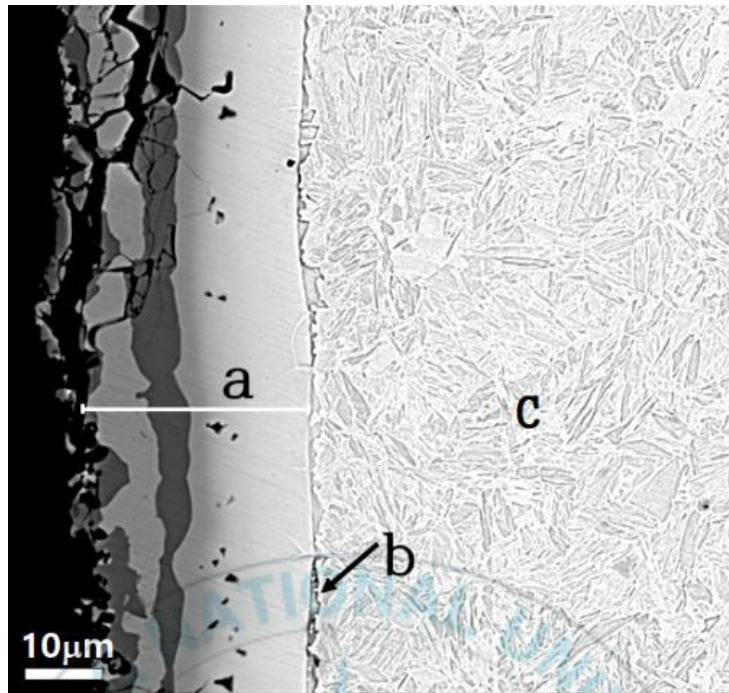
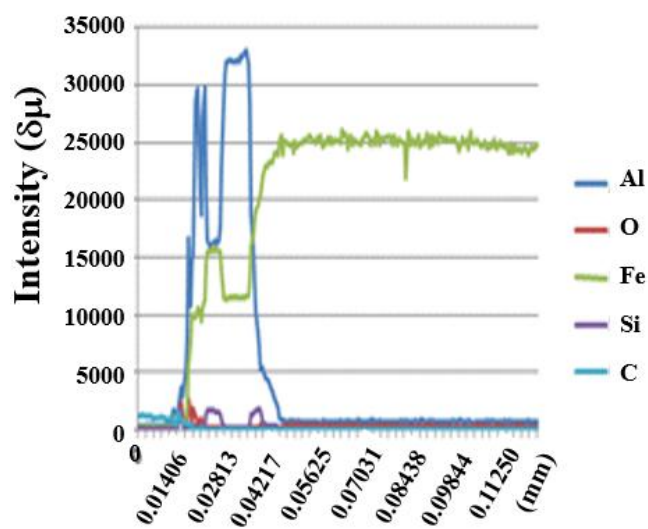
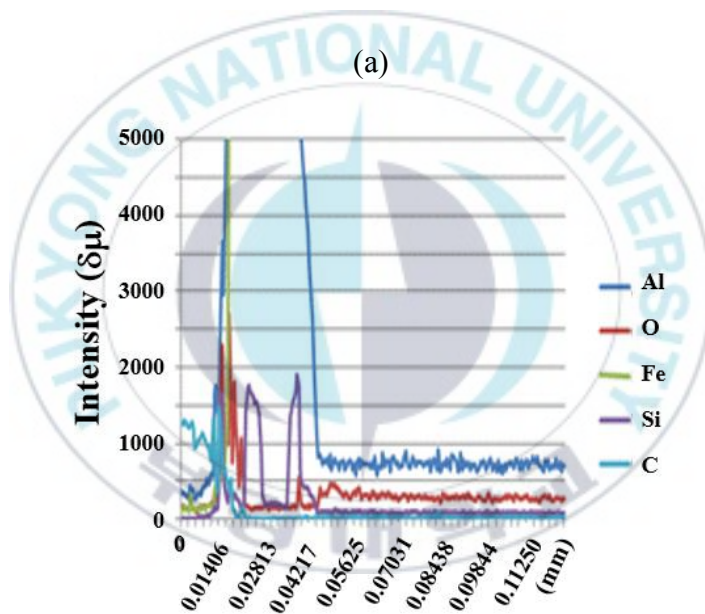


Figure 2. SEM micrographs of the hot-stamped steel sheet at 910 °C for 500 s.

Figure 3 shows the distribution of main elements in the compound layer and the diffusion layer by EPMA after the coated SABC 1470 steel sheet had been hot-stamped. The sample was hot-stamped after heating at 910 °C for 500 s. Line profile was performed from the surface to the inner side. In the compound layer of a), Al and Si are irregularly distributed, and a very thin diffusion layer was formed, while (b) shows the enlarged vertical axis of (a). This was expanded to understand in detail the distribution of major elements in the compound layer and the diffusion layer. The diffusion layer shows that Si, which was present in the coating layer before hot-stamping, hardly diffuses, and the diffusion layer is mainly made by the diffusion of Al. This is because Al has a lower melting point than Si, and at the same temperature is more thermally activated.



(a)

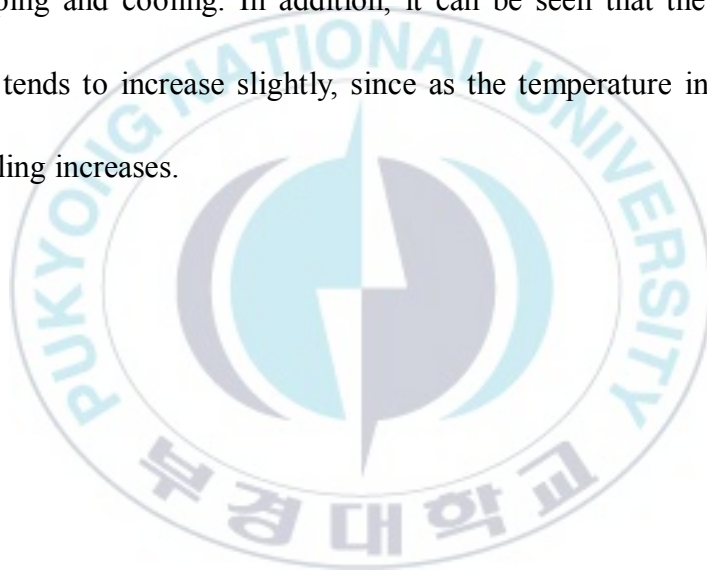


(b)

Figure 3. (a) EPMA line profile of the hot-stamped sample, (b) enlarged vertical axis of (a)

Figure 4 shows the coating layer thickness according to the hot-stamping temperature. The coated SABC 1470 steel sheet was heated for 500 s at different temperatures. All samples confirmed the formation of the compound layer and the diffusion layer, regardless of the hot-stamping temperature. In addition, it can be seen that as the temperature increases, the thicknesses of the compound layer and the diffusion layer increase. The reason is that as the temperature increases, Al, which was present in the coating layer before hot-stamping, diffuses more rapidly into the base metal. The hot-stamping temperature of 890 °C is a martensite structure with a small amount of pearlite and a ferrite structure. On the other hand, it can be seen that both the hot-stamping temperatures of (910 and 950) °C have martensite structure. In addition, it is evident that the size of the martensitic lath is slightly larger in the case of 950 °C than at 910 °C. The temperature at 890 °C is higher than the A3 transformation point of SABC1470 steel, so during heating, it has a single-phase austenite structure. However, the transfer to mold after heating and the mold temperature decreasing during hot-stamping made small amounts of pearlite and ferrite structures.

Therefore, during forming and cooling, only the austenite structure is transformed into martensite structure, and thus pearlite and ferrite are made at the martensite structure. On the other hand, (910 and 950)°C are single-phase austenite structures when forming, even if the temperature decreases during the transfer process for hot-stamping after heating. Austenite was transformed into martensitic structure during both hot-stamping and cooling. In addition, it can be seen that the martensitic lath size tends to increase slightly, since as the temperature increases, the supercooling increases.



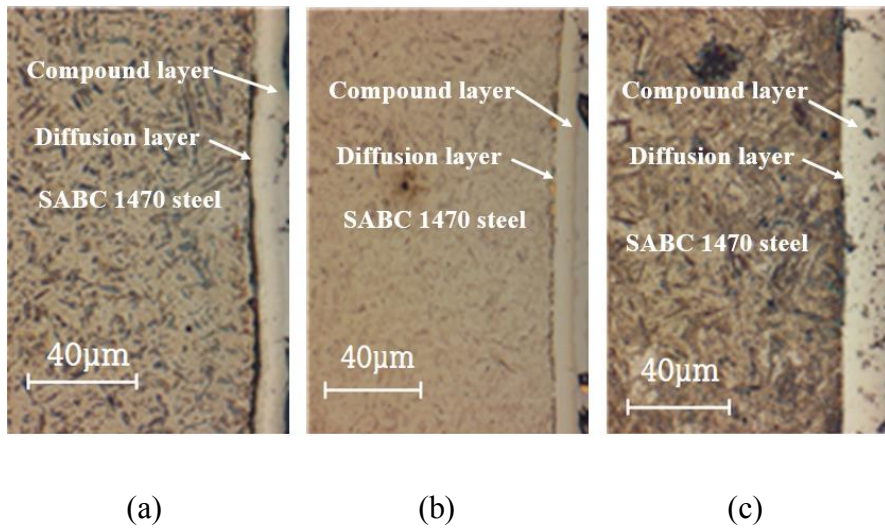


Figure 4. Coating layer according to different hot-stamping temperature at heating time of 500 s at (a) 890, (b) 910, and (c) 950 °C.

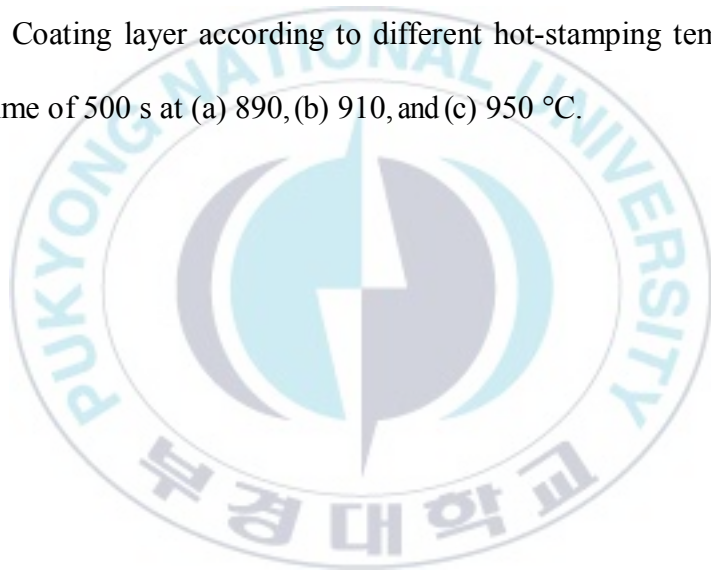


Figure 5 shows the observation of the coating layer with different heating time at a hot-stamping temperature of 910 °C. All samples confirm the compound layer and the diffusion layer, regardless of the hot-stamping time. As the heating time increases, the thickness of the coating layer becomes increases. This is because the longer the heating time, the more Al and Si in the coating layer before hot-stamping diffuse into the base metal. Regardless of the heating time, all three samples have martensitic structure, and as time increases, the martensitic lath size slightly increases, [15-17] as shown in Fig. 3. It was found that the hot-stamped samples consist of a compound layer, a diffusion layer, and a base metal; and as the heating temperature and heating time increase, the thicknesses of the compound layer and the diffusion layer increase.

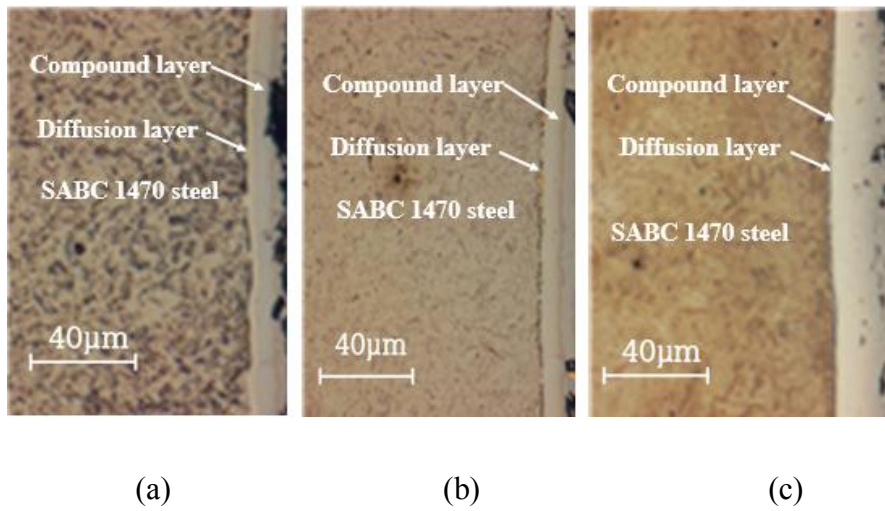
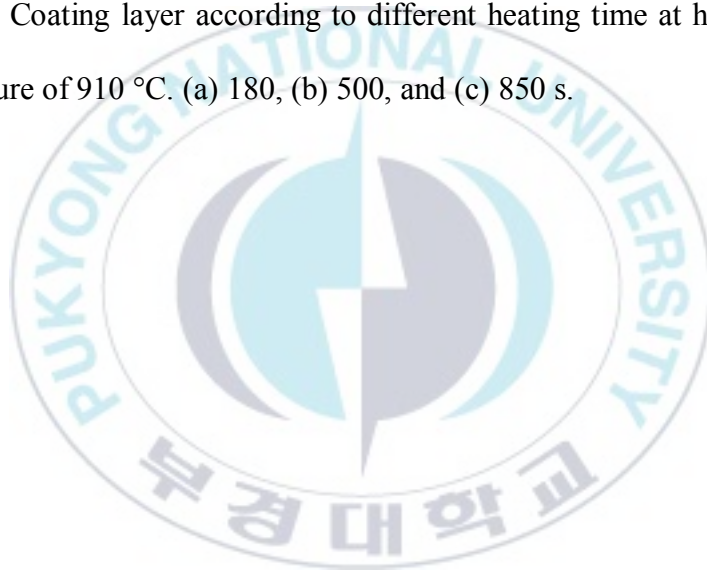


Figure 5. Coating layer according to different heating time at hot-stamping temperature of 910 °C. (a) 180, (b) 500, and (c) 850 s.



3.3 Compound of coating layer

In the SABC 1470 steel sheet coated with 40 % Al – 10 % Si–Fe solution, a compound layer and a diffusion layer were formed in the coating layer before formation by reaction and diffusion of Al and Si in the coating layer during the heating process for hot-stamping. It was found that the concentration of the main elements of the coating layer was irregularly distributed. Therefore, the components of the coating layer composed of the compound layer and the diffusion layer were investigated.

Figure 6 shows an analysis of the elements of a hot-stamped sample after heating at 910 °C for 500 s. A bright-field image (BF) using TEM shows the coating layer (compound layer and diffusion layer), and the distribution of main elements was mapped by EDS. In addition to Fe, Al, Si, and C can be identified in the compound layer and diffusion layer of the bright-field image.

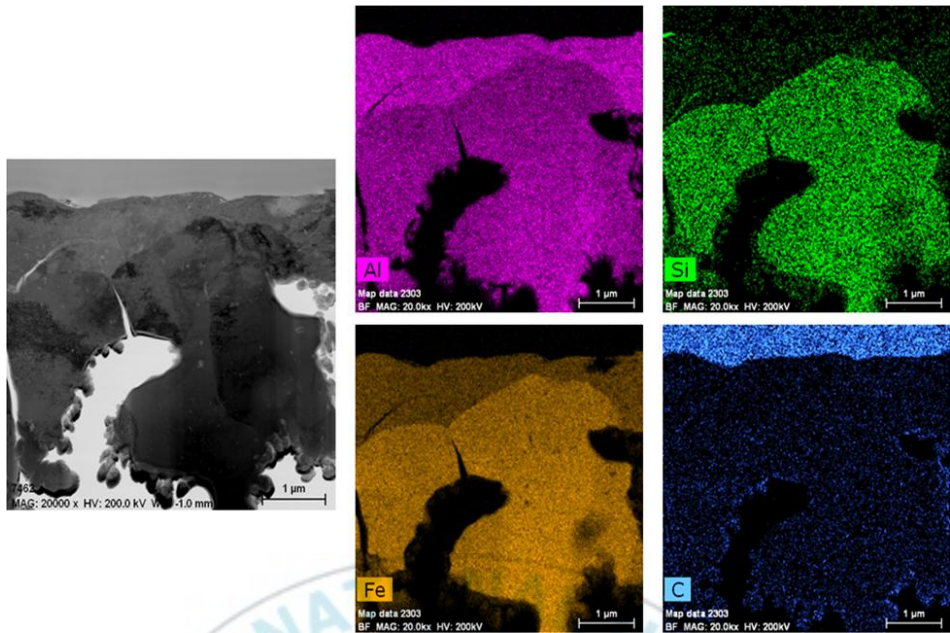
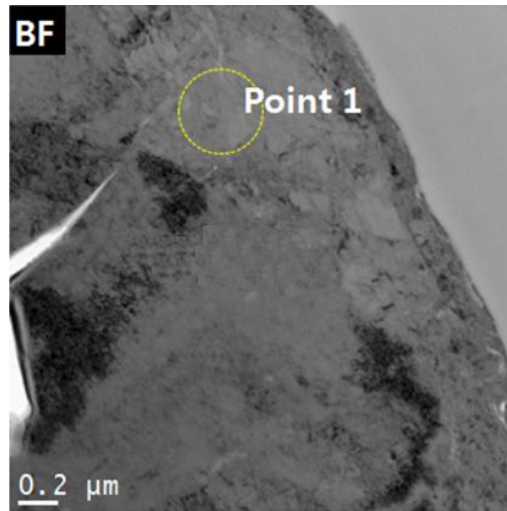


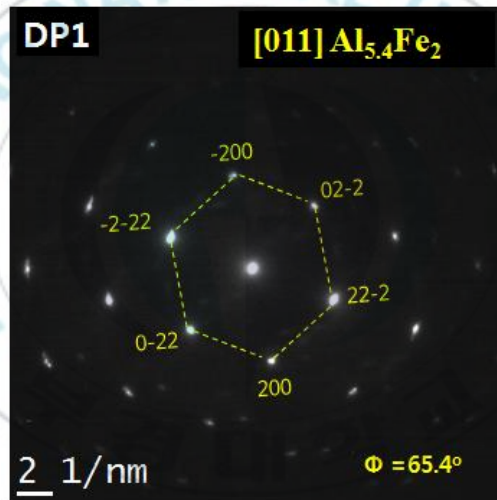
Figure 6. Bright-field image (left), and elemental mapping (right) of the coating layer in hot-stamped steel sheet.

Figure 7 shows the TEM imagery to analyze the phase of the coating layer (compound layer and diffusion layer) of the hot-stamped steel sheet, in which a) shows the coating layer (plating layer) in a bright-field image, while b) shows the Selected area electron diffraction patterns (SADP) of the compound (point 1 of (a)) present in the bright-field image and its analysis. From this observation, the $\text{Al}_{5.4}\text{Fe}_2$ compound was confirmed.





(a)



(b)

Figure 7. TEM micrographs of coating layer in hot-stamped steel sheet. (a) Bright-field image, and (b) SADP and indexing of SADP.

Figure 8 shows the diffraction curve and analysis obtained by X-ray diffraction of the coating layer of the hot-stamped steel sheet after heating at 910 °C for 500 s. This was investigated by another method for the coating layer compound of the hot-stamped steel sheet. In the coating layer, a compound having the structure of $\text{Al}_{5.4}\text{Fe}$ and Fe_2Si (the composition of the plating solution) was present. Therefore, Al, which was present in the coating layer before hot-stamping, was obtained as a compound of $\text{Al}_{5.4}\text{Fe}$ structure, and Si was obtained as a compound of Fe_2Si structure. In general, when a steel sheet coated with a plating solution containing Al and Si is heat-treated at a high temperature of 850 °C or higher, compounds such as FeAl , Fe_3Al , and Fe_2Al_5 are obtained. [15-17] However, the hot-stamping of this study obtained compounds of $\text{Al}_{5.4}\text{Fe}$ structure and Fe_2Si structure.

The above results show that when the SABC 1470 steel sheet coated with 40 % Al – 10 % Si–Fe solution is hot-stamped, the compound layer can obtain a compound of $\text{Al}_{5.4}\text{Fe}_2$ and Fe_2Si .

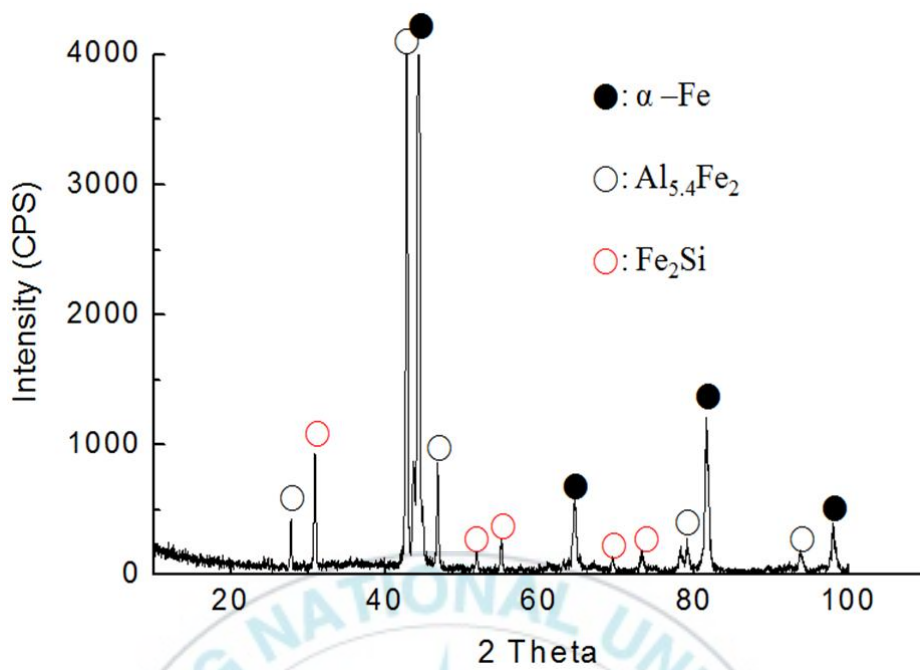


Figure 8. X-Ray diffraction patterns of coating layer after hot-stamping.

4. Conclusions

In this study, the SABC 1470 steel sheet coated with 40 % Al – 10 % Si–Fe coating solution was hot-stamped by change according to the temperature and time, and the coating layer was investigated. The conclusions obtained are as follows:

- 1) The coating layer of the hot-stamped steel sheet consisted of a compound layer, and a diffusion layer.
- 2) As the heating temperature and the heating time increased, the thickness of the diffusion layer increased. However, the compound layer, which is the coating layer before forming, did not change.
- 3) The diffusion layer was obtained by diffusion of Al in the coating layer.
- 4) The coating layer composed of the compound layer and the diffusion layer obtained a compound of $Al_{5.4}Fe$ and Fe_2Si structures.

References

1. Perera. F. S. Pollution from Fossil-Fuel Combustion is the Leading Environmental Threat to Global Pediatric Health and Equity: Solutions Exist. *Int. J. Environ. Res. Public. Health* **2018**, 15, 16. <https://doi.org/10.3390/ijerph15010016>
2. Brauser, S.; Pepke, L.A.; Weber, G.; Rethmeier, M. Deformation behaviour of spot-welded high strength steels for automotive applications. *Mater. Sci. Eng. A* **2010**, 527, 7099-7108. <https://doi.org/10.1016/j.msea.2010.07.091>
3. Wood, G.M. Lightweight Materials for Transportation. *Materials Technology* **1993**, 8, 221-223. <https://doi.org/10.1080/10667857.1993.11784987>
4. Fremgen, C.; Mkrtchyan, L.; Huber, U.; Maier, M. Modeling and testing of energy absorbing lightweight materials and structures for automotive applications. *Journal Science and Technology of Advanced Materials* **2005**, 883-888. <https://doi.org/10.1016/j.stam.2005.07.007>
5. Kim, M.J.; Cho, H.H.; Kim, S.H.; Nam, S.M.; Lee, S.H.; Moon, M.B.; Han, H.N. Effect of Zr addition on phase transformation and precipitation in B-added hot stamping steel. *Met. Mater. Int.* 2013, 19, 629-635. <https://doi.org/10.1007/s12540-013-4001-y>
6. Nakasato, F.; Takahashi, M. Effects of boron, titanium, and nitrogen on the hardenability of boron-treated steels for heavy machinery. *Met. Technol.* 1979, 6, 102-105. <https://doi.org/10.1179/030716979803276679>

7. Shen, Y.; Hansen, S.S. Effect of the Ti/N ratio on the hardenability and mechanical properties of a quenched-and-tempered C-Mn-B steel. *Met. Mater. Trans. A* 1997, 28, 2027–2035.
<https://doi.org/10.1007/s11661-997-0159-6>
8. Suh, D.W.; Oh, C.S.; Han, H.N.; Kim, S.J. Dilatometric analysis of austenite decomposition considering the effect of non-isotropic volume change. *Acta Mater.* 2007, 55, 2659–2669.
<https://doi.org/10.1016/j.actamat.2006.12.007>
9. Lee, C.W.; Choi, W.S.; Cho, Y.R.; De Cooman, B.C. Microstructure evolution of a 55 wt.% Al–Zn coating on press hardening steel during rapid heating. *Surf. Coat. Technol.* 2015, 281, 35–43.
<https://doi.org/10.1016/j.surfcoat.2015.09.041>
10. All  y, C. ; Dosdat, L.; Clauzeau, O.; Ogle, K.; Volovitch, P. Anticorrosion mechanisms of aluminized steel for hot stamping. *Surf. Coat. Technol.* 2014, 238, 188–196.
<https://doi.org/10.1016/j.surfcoat.2013.10.072>
11. Dosdat, L.; Petitjean, J.; Vietoris, T.; Clauzeau, O. Corrosion Resistance of Different Metallic Coatings on Press-Hardened Steels for Automotive. *Steel Res. Int.* 2011, 82, 726–733.
<https://doi.org/10.1002/srin.201000291>
12. Lee, M.G.; Kim, S.J.; Han, H.N.; Jeong, W.C. Application of hot press forming process to manufacture an automotive part and its finite element analysis considering phase transformation plasticity. *Int. J. Mech. Sci.* 2009, 51, 888–898.
<https://doi.org/10.1016/j.ijmecsci.2009.09.030>

13. Hayashi, K.; Nishibata, T.; Kojima, N.; Kajihara, M. Decarburization of 0.21C-1.3Mn-0.2Si Steel for Hot Stamping at Various Heating Temperatures. *Solid State Phenomena* 2011, 172-174, 882-892. <https://doi.org/10.4028/www.scientific.net/SSP.172-174.887>
14. Hamamoto, S.; Omori, H.; Asai, T.; Mizuta, N.; Jimbo, N. Yamano, T. Steel sheets for highly productive hot stamping. Kobelco Technology Review 2017, 35, 39-44.
15. Suehiro, M.; Maki, J.; Kusumi, K.; Ohgami, M.; Miyakoshi, T. Properties of Aluminum-coated Steels for Hot-forming. Nippon Steel Technical Report, 2003, 88, 295 – 415.
16. Seong Guk Son, Yeonjung Hwang, Chang Wook Lee, Ji Hong Yoo, Minsu Choi Effect of Hot Stamping Heat Treatment Temperature on Resistance Spot Weldability of Al-10% Si Coated 30MnB5 Steel Korean J. Met. Mater. 2019; 57, 778-786. <https://doi.org/10.3365/KJMM.2019.57.12.778>
17. Çavuşoğlu, O.; Çavuşoğlu, O.; Yılmazoğlu, A.G.; Üzel, U.; Aydın, H.; Güral, A. Microstructural features and mechanical properties of 22MnB5 hot stamping steel in different heat treatment conditions. Journal of Materials Research and Technology. 2020, 9, 10901-10908. <https://doi.org/10.1016/j.jmrt.2020.07.043>

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