World Academy of Science, Engineering and Technology
International Journal of Chemical and Materials Engineering
Vol:18, No:7, 2024

The Effect of Bath Composition for Hot-Dip Aluminizing of AISI 4140 Steel

Aptullah Karakaş, Murat Baydoğan

Open Science Index, Chemical and Materials Engineering Vol:18, No:7, 2024 publications.waset.org/10013688.pdf

Abstract**—**In the HDA process, Al or Al-Si molten baths are mostly used. However, in this study, three different Al alloys such as Al4043 (Al-Mg), Al5356 (Al-Si) and Al7020 (Al-Zn) were used as the molten bath in order to see their effects on morphological and mechanical properties of the resulting aluminide layers. AISI 4140 low alloyed steel was used as the substrate. Parameters of the HDA process were bath composition, bath temperature, and dipping time. These parameters were considered within a Taguchi L9 orthogonal array. After the HDA process and subsequent diffusion annealing, coating thickness measurement, microstructural analysis and hardness measurement of the aluminide layers were conducted. The optimum process parameters were evaluated according to coating morphology such as cracks, Kirkendall porosity and hardness of the coatings. According to the results, smooth and clean aluminide layer with less Kirkendall porosity and cracks were observed on the sample, which was aluminized in the molten Al7020 bath at 700 °C for 10 minutes, and subsequently diffusion annealed at 750 °C. Hardness of the aluminide layer was in between 1100-1300 hardness of Vickers (HV) and the coating thickness was approximately 400 µm. The results were promising such that a hard and thick aluminide layer with less Kirkendall porosity and cracks could be formed. It is therefore, concluded that Al7020 bath may be used in the HDA process of AISI 4140 steel substrate.

Keywords**—**Aluminum alloys, coating, hot-dip aluminizing, microstructure.

I. INTRODUCTION

HOT-dip aluminizing (HDA) is one of the several aluminizing methods to form a wear-, corrosion- and oxidation-resistant aluminide layers on the surface. In this method, the substrate is dipped into a molten aluminum bath, hold in the bath for several minutes, and cooled down to the room temperature in air. A subsequent annealing after the HDA process is generally performed. The main advantage of HDA is its very low investment cost in comparison with other aluminizing methods such as chemical vapor deposition (CVD), pack aluminizing and metalizing. Structure and thickness of the coating depend on the several parameters such as temperature, chemical composition of bath, dipping time and physical and mechanical properties of coating material. The coating microstructure generally comprises an outer layer with a composition similar to that of the molten bath and an inner intermetallic layer, which is very hard and brittle. According to literature review, researches are mostly based on process temperature and time and used as bath composition pure aluminum or Al-Si 11% alloys [1]-[3]. The diffusion coating could be produced by calorizing, cladding, welding, electrodeposit, hot dipping, spray metallizing, laser process, and vapor deposition. However, HDA and pack cementation calorizing are the most important commercial processes for aluminizing of steel. HDA is a process with lower investment cost compared to other aluminizing methods and due to this advantage, it is frequently used in the industry and there are many studies in the literature [1]-[4]. Since brittle iron aluminide (Fe2Al5) phase is formed on the surface after HDA and a brittle surface cannot be used in engineering applications, diffusion annealing is required to transform the brittle iron aluminide phase into ductile iron aluminide phase. One of the main reasons why the pack aluminizing method is preferred is that the ductile iron aluminide phase (FeAl) is formed directly on the surface after coating. [4]. Two types of HDA are commercially significant which are type 1 that uses aluminum-silisium alloy (5 to 11 % Si) and type 2 that uses pure aluminum for the coating. Silisium decreases the temperature of the bath because of eutectic temperature and get narrower coating layer. The manufacturers of aluminized steel products had realized that the HDA process must exercise control of the coating reaction in order to limit the growth of a brittle intermetallic layer during dipping. The excessive thickness of this layer became an area of concern for post-dip forming operations of the steel product. If the layer is too thick, it could crack and peel off during fabrication. The dipping time and the temperature of the molten aluminium bath have to be closely monitored in order to control the thickness of the interlayer [5]. In this study, three different aluminum alloys which are Al4043 (Al-Mg), Al5356 (Al-Si) and Al7020 (Al-Zn) were used as the molten bath in order to see the bath chemical composition effect on coating thickness and hardness with Taguchi orthogonal array [6].

II. EXPERIMENTAL

Three different Al alloys which are Al4043 (Al-Mg), Al5356 (Al-Si) and Al7020 (Al-Zn) used as bath material and were melted in graphite crucible. AISI 4140 specimens with the size of 30 mm diameter and 10 mm thickness were dipped in bath with three different dipping times as 10, 20 and 30 minutes, bath temperatures as 650, 700 and 750 °C and heat treated for diffusion annealing at 650, 700 and 750 °C temperatures after aluminizing according to Taguchi orthogonal array as indicated in Table I. Taguchi method makes possible to work with less specimens and nine different processes were sufficient for L9 matrix. Before HDA processes, surface of specimens was

Aptullah Karakas is with Istanbul Technical University, Türkiye (e-mail: aptullah.karakas@repkon.com.tr).

grinded with 320 grit number sandpaper and sandblasted with Al_2O_3 particles. Finally, the specimens were cleaned in ultrasonic bath with ethanol 2 minutes. After HDA processes, specimens were prepared with metallography equipment and coating thicknesses were measured and morphologies were examined with Olympus BX53M optical microscope. Hardness measurements were applied by 200 g load (HV0,2) for 5 seconds on coated layers with Emco Test Durascan 20.

TABLE I
TAGUCHI ORTHOGONAL ARRAY

Specimen Code	Bath Composition (Al)	Bath Temperature (^{O}C)	Dipping Time (min)	Diffusion Temperature (^{O}C)
L1	4043	650	10	650
L2	4043	700	20	700
L3	4043	750	30	750
L4	5356	650	20	750
L5	5356	700	30	650
L6	5356	750	10	700
L7	7020	650	30	700
L8	7020	700	10	750
L9	7020	750	20	650

III. RESULTS

The cross-section micrographs 200X magnification captured by optical microscope are shown in Fig. 1 and coating thickness and hardness values were indicated in Table II. There are iron aluminide layers, top aluminum layers and Kirkendal voids are seen in micrographs. The effect of different bath chemical compositions on coating thickness, coating hardness and coating morphology depending on different bath temperature, immersion time and diffusion annealing temperatures were investigated. All micrographs contain iron aluminide layers, large or small amount of Kirkendal voids and some of them retain aluminum from HDA process. Kirkendal voids creation is related with different diffusion mobility of atoms and it is affected directly by temperature, time and chemical composition [7]. As for another morphological property of HDA, saw blade structure is generally observed after HDA processes and it could be disappeared after diffusion annealing. The specimens aluminized in Al4043 bath did not show saw blade effect although diffusion annealing temperatures same for three different bath materials. This could be related with crystallographic direction of iron aluminide layer, because of Fe_2Al_5 growth on direction of <111>. It is possible that during aluminizing process with Al4043 bath, <111> direction could not play active role because of different chemical compositions [8].

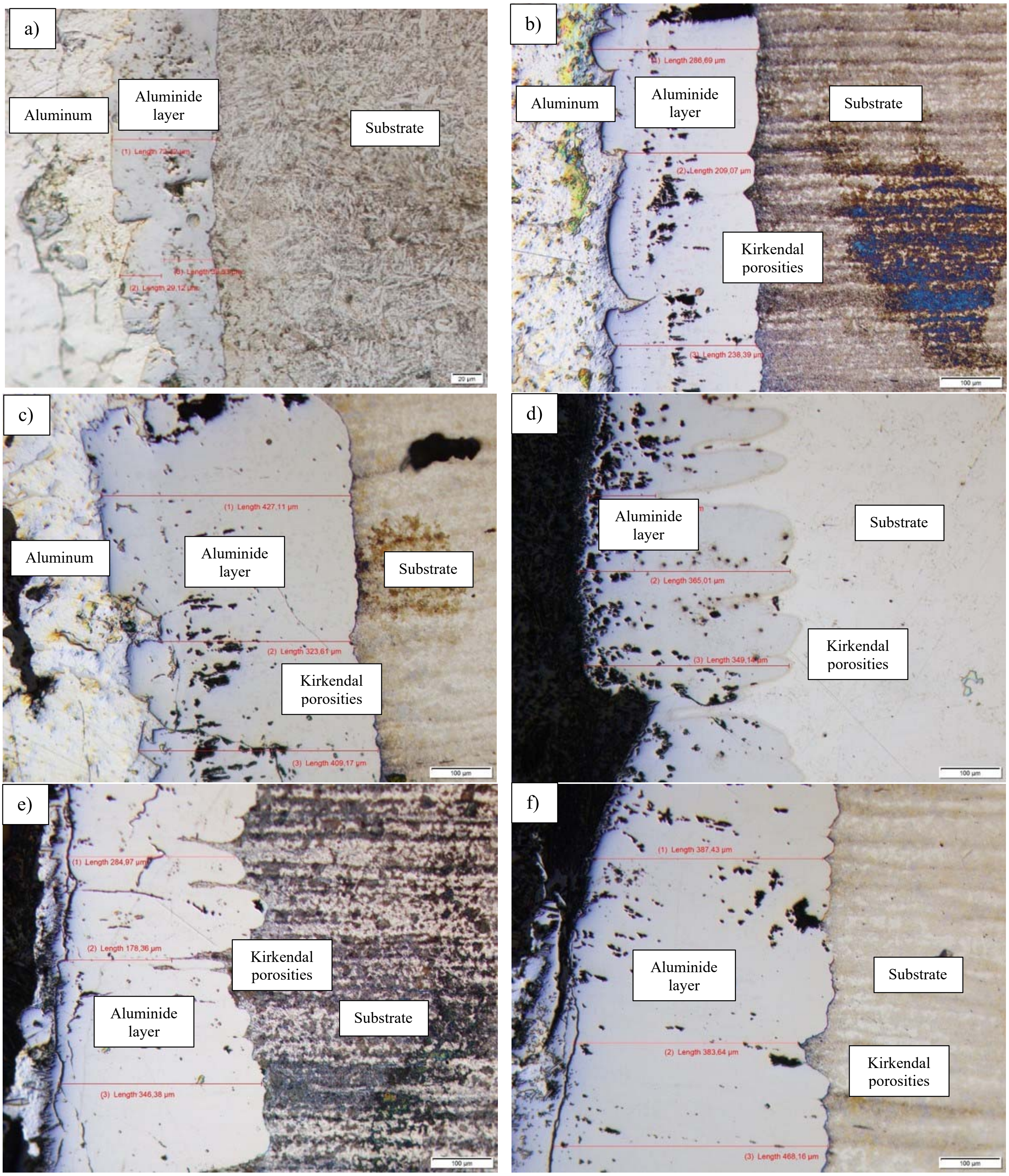
Throughout the HDA process, molten aluminium wets the surface of the steel substrate and diffuses into steel to form Fe-Al intermetallic compounds at the coating/substrate interface This intermetallic layer dominates the performance of substrate material, and its thickness affects the resulting properties. These intermetallic compounds are highly brittle; therefore, it is always intended to keep the thickness of the interlayer to a minimum to achieve better mechanical properties. Al-rich iron aluminide phases such as Fe_2Al_5, $FeAl_3$ and $FeAl_2$ have high hardness values around 1000-12000 HV and behave brittle, as for Fe-rich iron aluminide phases such as FeAl and Fe_3Al have hardness values around 500-600 HV and behave ductile. Ductile behavior of Fe-rich iron aluminide phases is more resistant to crack formation than Al-rich iron aluminide phases. From L1 to L9, specimens have almost same hardness values and most probably those aluminide layers consist of Al-rich iron aluminide phases because of high hardness values. Diffusion annealing temperatures which are 650, 700 and 750 °C were not sufficient to phase transition from Al-rich iron aluminide phase to Fe-rich iron aluminide phase. Coating thickness values are highly dependent on bath temperature, because the higher bath temperature the thicker the coating thickness. The diffusion annealing temperature value has a dominant effect on the coating thickness, because L4 has a greater coating thickness value than L5 and L8 has a greater coating thickness value than L9, but L5 and L9 bath temperatures are higher than L4 and L8. This result showed that diffusion annealing temperature is more important than bath temperature in related with coating thickness. The specimens, which were aluminized in Al4043, have aluminum top layer left after diffusion annealing processes, but there were not observed any aluminum top layers on the surface of the specimens which were aluminized in Al5356 and Al7020. The reason could be related with chemical compositions of the bath alloys, Al4043 consists of mainly Al and Si metals and diffusion capability of this alloy could be less than other bath alloys. This phenomenon could be associated with Si metal, because coating thickness after Type 1 HDA method is less than Type 2 method according to ASTM A463.

TABLE II
COATING THICKNESS AND HARDNESS VALUES OF SPECIMENS FROM L1 TO L9

Specimen Code	Coating Thickness (μm)	Coating Hardness (HV)
L1	70-100	1000-1200
L2	240-280	1000-1200
L3	410-430	1000-1200
L4	350-380	1000-1200
L5	290-340	1000-1200
L6	400-450	1000-1200
L7	280-310	1000-1200
L8	450-480	1000-1200
L9	310-330	1000-1200

IV. CONCLUSION

Kirkendal voids are observed on all specimens, but L1 specimen is least one. L1 specimen also have thinnest coating layer, therefore, HDA process parameters of L1 specimen are Al4043 bath material, 650 °C dip temperature, 10 minutes dipping time and 650 °C diffusion annealing temperature look most promising route of this study. In this study, it could be concluded that three different aluminum alloys look candidate for HDA bath material, but only coating morphology and hardness values are not satisfactory. The performance tests such as wear test, oxidation test or erosion test according to intended usage area must be applied in order to compare with ASTM A463.


a)
Aluminum
Aluminide layer
Substrate
(2) Length 29,12 µm
20 µm
b)
(1) Length 286,69 µm
Aluminum
Aluminide layer
Substrate
(2) Length 209,07 µm
Kirkendal porosities
(3) Length 238,39 µm
100 µm
c)
(1) Length 427,11 µm
Aluminum
Aluminide layer
Substrate
(2) Length 323,61 µm
Kirkendal porosities
(3) Length 409,17 µm
100 µm
d)
Aluminide layer
Substrate
(2) Length 365,01 µm
Kirkendal porosities
(3) Length 349,14 µm
100 µm
e)
(1) Length 284,97 µm
(2) Length 178,36 µm
Kirkendal porosities
Aluminide layer
Substrate
(3) Length 346,38 µm
100 µm
f)
(1) Length 387,43 µm
Aluminide layer
Substrate
(2) Length 383,64 µm
Kirkendal porosities
(3) Length 468,16 µm
100 µm

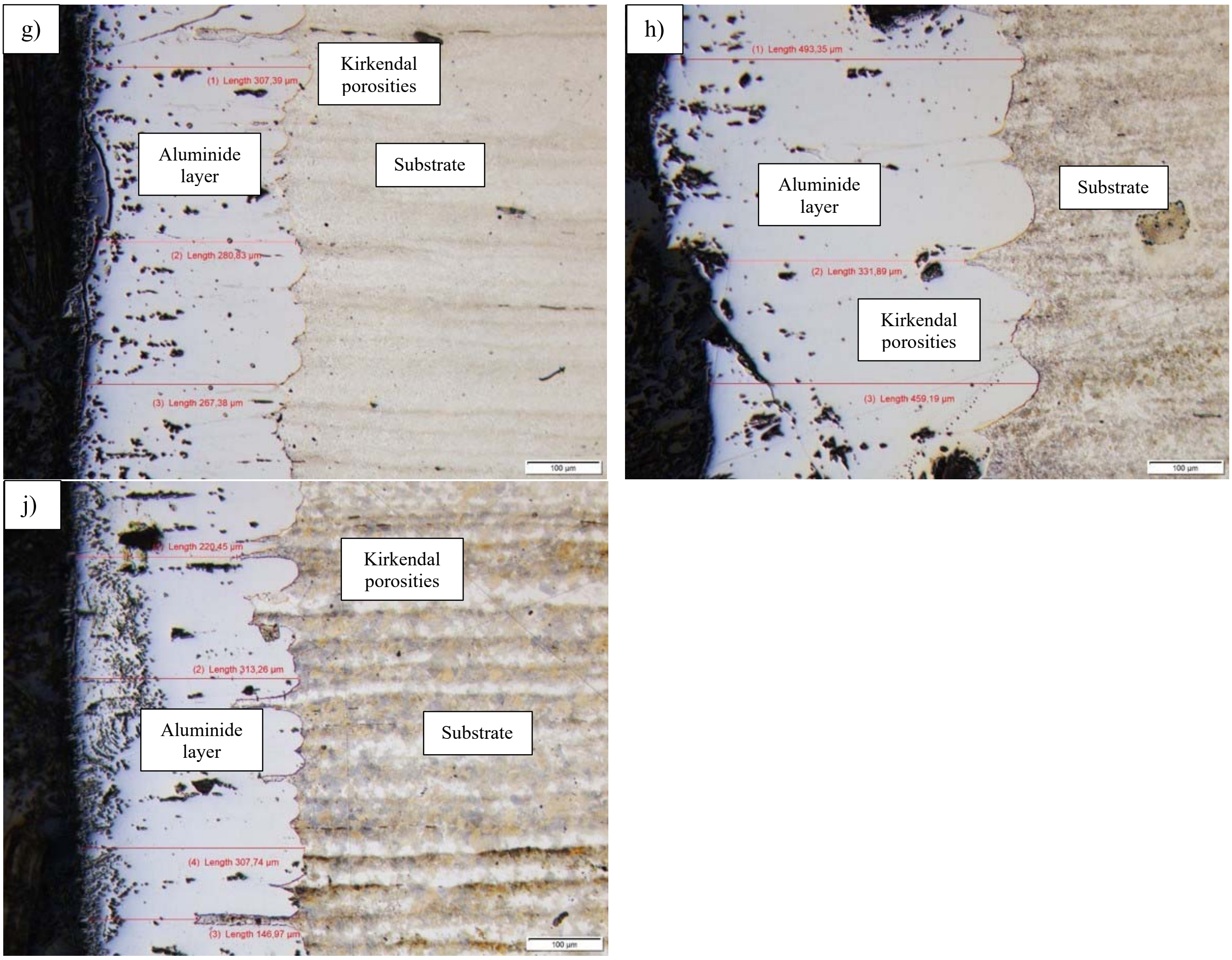


Fig. 1 Micrographs: (a) L1, (b) L2, (c) L3, (d) L4, (e) L5, (f) L6, (g) L7, (h) L8 and (j) L9 specimens

ACKNOWLEDGMENT

The authors would like to thank to Repkon Machine and Tool Industry and Trade Inc. which financially supported this study.

REFERENCES

[1] A. Bahadur, “Aluminum coatings for steel,” Materials and Manufacturing Processes, vol. 11, no. 2, pp. 225–232, 1996.
[2] R. Mola, T. Bucki, and K. Wcisło, “Characterization of Coatings on Grey Cast Iron Fabricated by Hot-dipping in Pure Al, AlSi11 and AlTi5 Alloys,” Archives of Foundry Engineering, vol. 14, no. 1, pp. 85–90, Mar. 2014.
[3] M. V. Akdeniz, A. Mekhrabov, and T. Yilmaz, “The role of Si addition on the interfacial interaction in fe-Al diffusion layer,” Scripta Metallurgica et Materialia, vol. 13, pp. 1723–1728, Jul. 1994.
[4] J. R. Davis, ASM Handbook Vol5, Surface Engineering of Carbon and Alloy Steels, 2nd ed., vol. 5. ASM International, 1994.
[5] P. P. Dey, S. Sahu, P. S. Banerjee, and M. Ghosh, “A review on metallurgical features of hot-dip aluminized steel,” Engineering Research Express, vol. 5, no. 1, Mar. 2023.
[6] R. Rajendran, P. Hariharan, N. Gowrishankar, U. Jaikrishna, and A. Rajadurai, “Surface Modification of EN 8 Steel by Aluminizing and Nitriding for,” 2009.
[7] Y. Yürektürk and M. Baydoğan, “Characterization of ferritic ductile iron subjected to successive aluminizing and austempering,” Surf Coat Technol, vol. 347, pp. 142–149, Aug. 2018.
[8] K. Kishore, S. Chhangani, M. J. N. V. Prasad, and K. Bhanumurthy, “Microstructure evolution and hardness of hot dip aluminized coating on pure iron and EUROFER 97 steel: Effect of substrate chemistry and heat treatment,” Surf Coat Technol, vol. 409, Mar. 2021.

Aptullah Karakaş is PhD student at Department of Metallurgical and Materials Engineering in Istanbul Technical University in İstanbul/Türkiye, equally, R&D Engineer at Materials Technologies Department in Repkon Machine and Tool Industry in İstanbul/Türkiye.

Murat Baydoğan is Professor at Department of Metallurgical and Materials Engineering in Istanbul Technical University in İstanbul/Türkiye.