

# Study of the behavior of a high temperature aluminum coated steel

Sabrina Mendil<sup>1,2</sup>, Kamel Taïbi<sup>2</sup>, Said Azem<sup>1</sup>

<sup>1</sup>Département de Génie Mécanique, Université Mouloud Mammeri de Tizi-Ouzou, 15100. Algérie

[smendil@yahoo.fr](mailto:smendil@yahoo.fr),  
[saidazem@mail.ummto.dz](mailto:saidazem@mail.ummto.dz),

<sup>2</sup>LSGM, Faculté du Génie Mécanique et du Génie des Procédés, Université USTHB de Bab-Ezzouar, 16311. Algérie.

[kameltaibi@yahoo.fr](mailto:kameltaibi@yahoo.fr)

**Abstract**---This work studies the feasibility of obtaining steel aluminide coatings on mild steel substrate by aluminizing treatment (HDA) especially at the temperature of 800°C and 1000°C. The mild steel was coated for 90 min in air. Detailed optical, scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDS) and electron backscatter diffraction (EBSD) investigations were performed to characterize the phases identified during aluminizing. After aluminizing, three layers were formed, external aluminum layer, intermetallic layer and steel substrate. The results showed that the intermetallic layer growing toward the steel substrate possessed a tongue-like morphology which delimited by a strong concentration of pearlite at the temperature of 800°C. But this morphology tends to disappear and takes on a planar aspect after aluminizing at 1000°. However, the interface intermetallic layer /external aluminum layer evolved under an irregular profil with a tongue-like morphology in the temperature of 800°C and 1000°C. The intermetallic layer of the hot dipped was composed of minor monoclinic FeAl<sub>3</sub> and major orthorhombic Fe<sub>2</sub>Al<sub>5</sub> according to the EDS and EBSD analysis at the temperature of 800°C and 1000°C. But at the interface intermetallic/steel, the intermetallic compounds formed in this level are FeAl and Fe<sub>3</sub>Al of cubic crystal structure at the temperatures of 1000°C and 800°C respectively. These intermetallic compounds which appear in very thin layer, at the interface, are tangent toward the steel.

**Keywords:** Mild steel, Hot-dipping, intermetallic Compounds, Aluminide layer, EBSD.

## I. INTRODUCTION

Aluminizing in aluminum melt is an effective method used to form a thin layer of aluminum on the surface of steel substrate for improving oxidation and corrosion resistance [1-4]. Aluminizing is usually carried out by the hot-dip process in which the component to be

coated is treated in an aluminum molten bath. The interface between the substrate and the intermetallic layer appeared tongue-like morphology in most cases. In the process, Al diffuses into steel to form intermetallic compound between the outer aluminum coat and the substrate steel [5, 6]. This compound layer may be composed of one or more of the intermetallic compounds between iron and aluminum, like Fe<sub>2</sub>Al<sub>5</sub> and Fe<sub>3</sub>Al. This compounds are very brittle in nature [5, 6], and cause cracking and peeling during bending and machining [7]. Thus, the present paper is to present the results of an experimental study on the effects of time aluminizing during aluminizing at temperature of 800°C on the microstructure of Fe-Al coated steel substrate.

## II. Experimental

### A. Materials

The substrate material used for the investigation was a medium carbon cubic steel with a nominal composition Fe-0,4C (wt %). Commercial grade pure aluminum with a purity of 99,5 % was used as the molten aluminum bath.

### B. Sample preparation

Aluminum ingot was melted in a graphite crucible in a resistance furnace, and the melt was maintained at 800°C and 1000°C. After being degreased in 100 g/l sodium hydrate solution at 50 °C for 5 min, rinsed with water, and then descaled in a 20% hydrochloric acid solution, rinsed with water again, and pretreated in a potassium salt solution at 92 °C for 2 min, the steels substrates were immersed in the molten aluminum bath for a period of time, 180min. Then the samples were taken out from the melt and cooled in air.

### C. Microstructure and property characterization

After hot-dip aluminizing, Microstructure and chemical composition of the cross-section of the coated specimens were analyzed using scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) and back scattered diffraction (EBSD). The different phases of the surface layers were determined with EBSD technique analysis.

TABLE I. EDAX RESULTS

	Fe (At%)	Al (At%)	Phase
A	98.32	/	Fe
B	72.71	23.35	Fe <sub>3</sub> Al
C	25.75	74.25	Fe <sub>2</sub> Al <sub>5</sub>
D	00.17	99.83	Al
E	21.86	78.14	FeAl <sub>3</sub>
F	47.40	52.60	FeAl

### II. Result and discussion

By aluminizing the samples in pure aluminum, typical microstructure of samples aluminized of medium carbon steel in the temperature on 800°C and 1000°C, for 90min dipping times, are shown in Figure 1 and 2(a and c). Three distinct regions which could be easily identified in these microstructures include; the outer aluminum layer, the intermetallic compound layer, and the substrate steel. It can be observed that the thickness of intermetallic layers increases with increasing temperature. It can also be observed that the morphology of intermetallic layer appears irregular with a finger like orientated towards the iron in the temperature of 800°C but this morphology tend to become planar and uniforme at the temperature of 1000°C.

Figure (1a) and (2c) can be divided into three areas with different brightness, the bright area at the high side is steel, the dark one at the low side is Al, and the grey one with tongue like pattern located in between is iron aluminides. The intermetallic layer (about 3-4 mm thick) formed by Al diffusion into the steel substrate. The intermetallic layer mainly consisted of the brittle compound Fe<sub>2</sub>Al<sub>5</sub>. The composition found with EDX point analyses was 74.25 at.% Al, 25.75 at.% Fe. In the external layer showed in Figure (1b and 2d), there are ramifications in forms of crystals globular and lamellar ramifications which correspond to intermetallic compounds of FeAl<sub>3</sub>. The composition found with EDX point analysis was 21.86% at Fe and 78.14 at% Al. which dispersed in the aluminum matrix.

But in the transition zone between the intermetallic layer and the steel substrate, a thin layer of the compound Fe<sub>3</sub>Al could be detected at 800°C. An EDX point analyses was 23.35at% Al, 72.71at%

Fe. But at 1000°C, the phase formed at the interface is FeAl. An EDX analysis was 47.40%At Fe and 52.60%Al. These intermetallic compounds were detected by EBSD analysis. The EDAX analysis at different positions in the microstructure is concluded in Table 1.

Additionally, cracks and voids could be observed in the intermetallic layer (Figure (1a and 2(c, d)) which were caused by grinding and polishing indicating the brittle nature of the aluminized layer. The voids were also attributed to the Kirkendall effect. The different diffusion rates between Fe and Al caused a net flux of vacancies to form voids at the interface between the aluminide layer and the steel substrate [9].

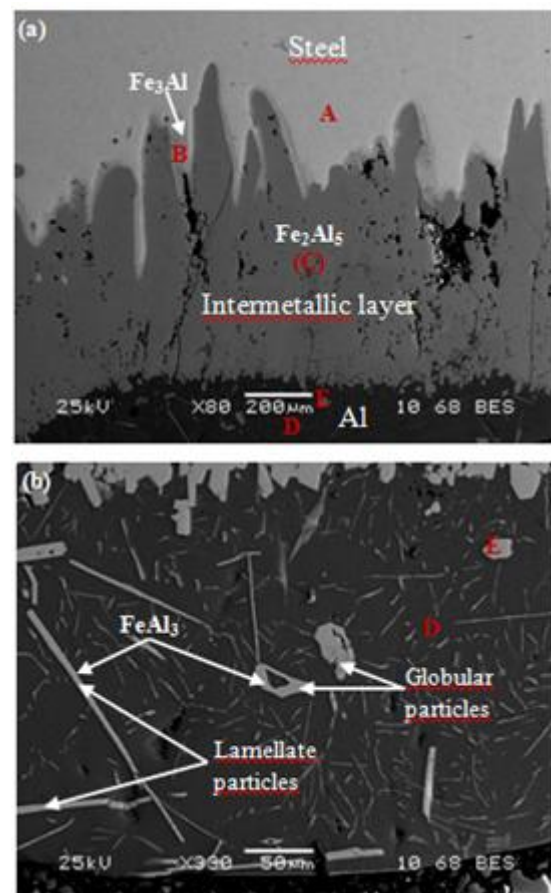


Fig.1. cross section micrographs of steel aluminized for 90mn at 800°C, (a) 200µm, (b) external layer.

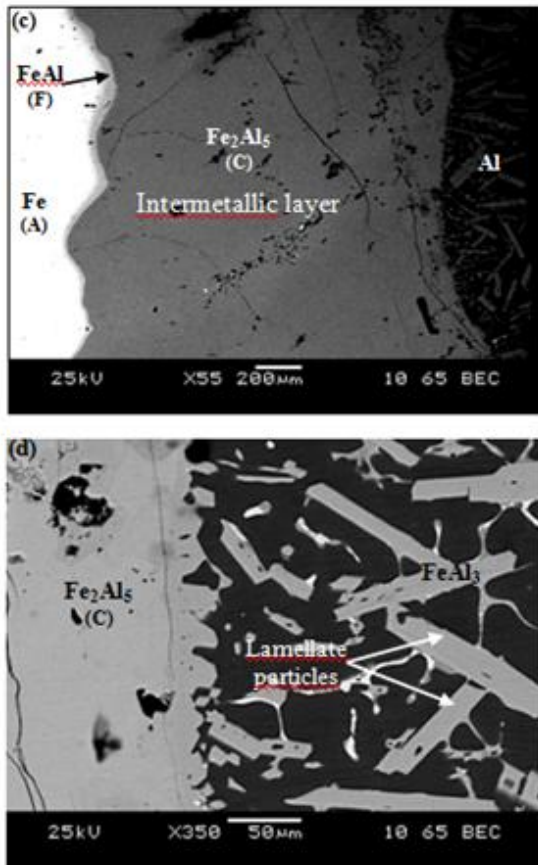


Fig.2. cross section micrographs of steel aluminized for 90mn at 1000°C, (c) 200µm, (d) external layer

applied for identifying each crystallographic structure of the intermetallic phases and the phase distributions in the aluminide layers after hot-dipping for various times. Figure (4(a, b, c)) are the experimental lines of Kikkuchi and their indexed results of the A, B and C phases in the steel

was revealed that the crystallographic structures of the A, B and C phases; are cubic; iron of the steel, space group(Oh) [m3m],  $a=2.87 \text{ \AA}$ , cubic  $\text{Fe}_3\text{Al}$ , space group (Oh) [m3m],  $a=5.78 \text{ \AA}$  and orthorhombic  $\text{Fe}_2\text{Al}_5$ , space group[D2h]mmm,  $a=7.656 \text{ \AA}$ ,  $b=6.415 \text{ \AA}$ ,  $c=4.218 \text{ \AA}$ , respectively, as showed in figure (3).

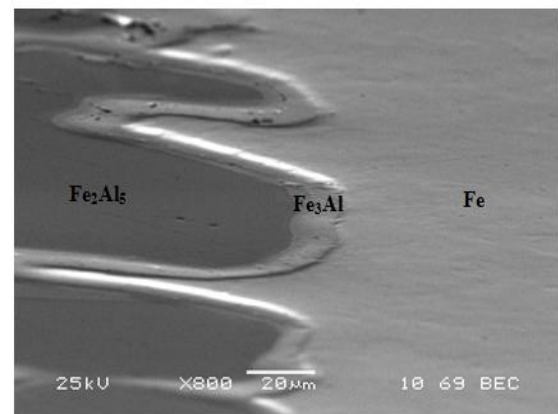


Fig.3. EBSD analysis showing the phase distribution of specimens aluminized in air at 800°C for 90mn

In the intermetallic layer, the aluminum intensity appears more important by comparing it has that of iron, which is confirmed with analyses EDS and EBSD showing that the intermetallic layer is composed of intermetallics  $\text{Fe}_2\text{Al}_5$  and  $\text{Fe}_3\text{Al}$ . The point of intersection between iron and aluminum located has a distance from  $9 \mu\text{m}$  confirms the interdiffusion of these two elements. While approaching towards the heart of steel, the aluminum intensity decreases whereas that of iron tends has to increase, it is the phase rich in iron.

According to the chemical compositions and crystallographic structures of the intermetallic phases in the aluminide layers which were acquired by means of EDS, there were  $\text{Fe}_2\text{Al}_5$  and  $\text{FeAl}_3$  formed in the aluminide layer on 0.4%C steel after hot-dipping into a pure aluminum molten bath for various immersion times at  $800 \text{ }^\circ\text{C}$  and  $1000 \text{ }^\circ\text{C}$ . But at the interface intermetallic/steel, the intermetallic compounds formed in this level are  $\text{FeAl}$  cubic and  $\text{Fe}_3\text{Al}$  at the temperatures of  $1000 \text{ }^\circ\text{C}$  and  $800 \text{ }^\circ\text{C}$  respectively.

For a further verification of the phase constitution of the aluminide layers, EBSD analysis were

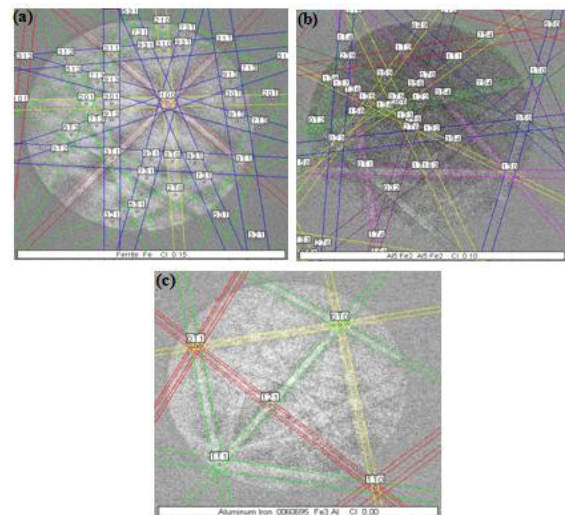


Fig.4. representative experimental EBSD pattern of different phases in the steel aluminized. (a) phase indexing as cubic Fe, (b) phase indexing as orthorhombic  $\text{Fe}_2\text{Al}_5$  and (c) phase indexing as cubic  $\text{Fe}_3\text{Al}$ .

But at the temperature of 1000°C as shown in figure (6c), It was revealed that the crystallographic structures of the F (figure 2c) phase is the cubic FeAl, space groupe Pm3m,  $a=2.9030\text{\AA}$ .

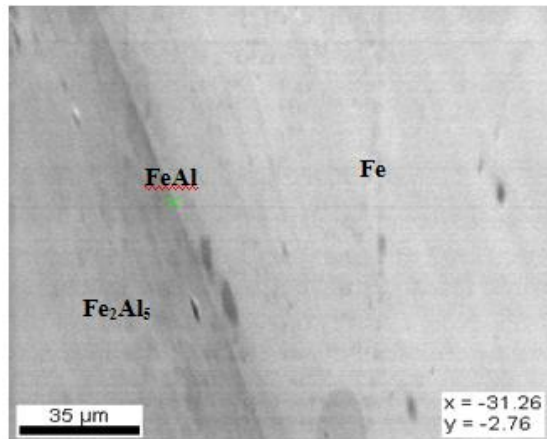


Fig.5. EBSD analysis showing the phase distribution of specimens aluminized in air at 1000°C for 90mn

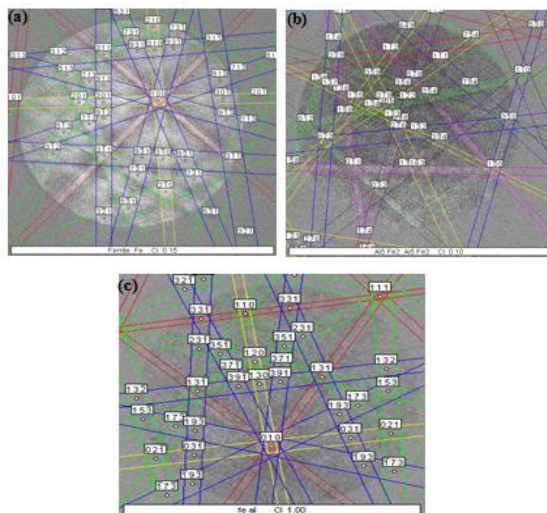


Fig.6. representative experimental EBSD pattern of different phases in the steel aluminized. (a) phase indexing as cubic Fe, (b) phase indexing as orthorhombic  $\text{Fe}_2\text{Al}_5$  and (c) phase indexing as cubic FeAl.

#### IV. CONCLUSION

Hot-dip aluminizing coating was investigated in the temperature from 800 °C and 1000°C in air for 90min. The results may be summarized as follows:

The thickness of the intermetallic layer in the steel substrate increase with increasing temperature and the morphology of intermetallic layer appear irregular with a finger like orientated towards the iron at 800°C, but this morphology tend to become planar and uniforme at the temperature of 1000°C.

The EDX and EBSD analysis revealed that the intermetallic layer was composed of minor monoclinic  $\text{FeAl}_3$  and major orthorhombic  $\text{Fe}_2\text{Al}_5$  at the temperature of 800°C and 1000°C. But at the interface intermetallic/steel, the intermetallics compounds formed in this level are FeAl cubic and  $\text{Fe}_3\text{Al}$  at the temperatures of 1000°C and 800°C respectively.

#### REFERENCES

- [1] Wang Deqing, Shi Ziyuan, Zou Longjiang. “A liquid aluminum corrosion resistance surface on steel substrate”, *Applied Surface Science* 214 (2003) 304–311.
- [2] wang Deqing. “Phase evolution of an aluminized steel by oxidation treatment”, *Applied Surface Science* xxx (2007) xxx–xxx
- [3] Deqing Wang, Ziyuan Shi. “Aluminizing and oxidation treatment of 1Cr18Ni9 stainless steel”, *Applied Surface Science* 277 (2004) 255-260
- [4] S. Sharafi, M.R. Farhang. “Effect of aluminising on surface microstructure of an HH309 stainless steel”, *Surface and Coatings Technology* 200 (2006) 5048 – 5051.
- [5] Shigeaki Kobayashi, Takao Yakou. “Control of intermetallic compound layers at interface between steel and aluminum by diffusion-treatment”, *Material Science and Engineering A* 338 (2002) 44-53.
- [6] Gul Hameed Awan, Faiz ul Hasan. “The morphology of coating/substrate interface in hot-dip-aluminized steels”, *Materials Science and Engineering A* 472 (2008) 157-165.
- [7] Tomohiro Sasaki, Takao Yakou, “Features of intermetallic compounds in aluminized steels formed using aluminum foil”, *Surface and Coating Technology* 201 (2006) 2131-2139.
- [8] Ningxin Zhang, Jaroslaw Wosik, Werner Fragner, Roman Sonnleitner, Gerhard E. Nauer, “Three-dimensional analysis of the growth of intermetallics phases between solid steel and molten aluminium”, *Intermetallics* 18 (2010) 221–225.
- [9] Chaur-Jeng Wang, Shih-Ming Chen, “The high-temperature oxidation behavior of hot-dipping Al–Si coating on low carbon steel”, *Surface and Coatings Technology* 200 (2006) 6601–6605.

- [10] Wei-Jen Cheng, Chaur-Jeng Wang, “Observation of high-temperature phase transformation in the Si modified aluminide coating on mild steel using EBSD”, *Materials characterization* 61 (2010) 467-473.
- [11] Wei-Jen Cheng, Chaur-Jeng Wang, “Growth of intermetallic layer in the aluminide mild steel during hot-dipping”, *Surface & Coatings Technology* 204 (2009) 824-828.
- [12] Wei-Jen Cheng, Chaur-Jeng Wang, “Effect of silicon on the formation of intermetallic phases in aluminide coating on mild steel”, *Intermetallics* 19 (2011) 1455-1460.
- [13] Shilei Han, Hualing Li, Shumao Wang, Lijun Jiang, Xiaopeng Liu, “Influence of silicon on hot-dip aluminizing process and subsequent oxidation for preparing hydrogen/tritium permeation barrier”, *international journal of hydrogen energy* 35 (2010) 2689-2693.
- [14] Sung-Ha Hwang, Jin-Hwa Song, Yong-Suk Kim. “Effects of carbone content of carbone steel on its dissolution into a molten aluminum alloys”, *Material Science and Engeneering A* 390 (2005) 437-443.
- [15] Z.D. Xiang, P.K. Datta. “Pack aluminisation of low alloy steels at temperatures below 700°C”, *Surface and Coatings Technology* 184 (2004) 108 – 115.
- [16] Teng-Shih Shih, Shu-Hao Tu. “Interaction of steel with pure Al, Al-7Si and A356 alloys”, *Materials Science and Engeneering A* 454-455 (2007) 349-356.
- [17] Hou Xiaoxia, Yang Hua, Zhao Yan, Pan Fuzhen. “Effect of Si on the interaction between die casting die and aluminum alloy”. *Materials Letters* 58 (2004) 3424- 3427.
- [18] Vikas Jindala, V.C. Srivastava. “Growth of intermetallic layer at roll bonded IF-steel/aluminum interface”. *journal of materials processing technology* 195 (2008) 88-93.
- [19] D.C. Lou a, O.M. Akselsen a, M.I. Onsøien a, J.K. Solberg b, J. Berget, “Surface modification of steel and cast iron to improve corrosion resistance in molten aluminium”, *Surface and Coatings Technology* 200 (2006) 5282 – 5288.
- [20] Wei-Jen Cheng, Chaur-Jeng Wang. “Study of microstructure and phase evolution of hot-dipped aluminide mild steel during high-temperature diffusion using electron backscatter diffraction”. *Applied Surface Science* 257 (2011) 4663-4668.
- [21] Wei-Jen Cheng, Chaur-Jeng Wang. “EBSD study of crystallographic identification of Fe-Al-Si intermetallic phases in Al-Si coating on Cr-Mo steel”. *Applied Surface Science* 257 (2011) 4637-4642.