# TRANSFORMATION OF INTERMETALLIC LAYER DUE TO OXIDATION HEAT TREATMENT ON HOT-DIPPED ALUMINUM COATED STEEL

# <sup>1</sup>Hishamuddin Hj. Husain, <sup>2</sup>Abdul Razak Daud, <sup>1</sup>Muhamad Daud, and <sup>1</sup>Nadira Kamarudin

<sup>1</sup>Malaysian Nuclear Agency, Bangi, 43000 Kajang, Selangor D.E. <sup>2</sup>Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor D.E. e-mail <hishamuddin@nuclearmalaysia.gov.my>

#### **ABSTRACT**

Heat treatment was introduced onto the aluminum coated low carbon steel to promote the formation of thin layer of oxide for enhancement of oxidation protection of steel. This process has transformed the existing intermetallic layer formed during hot dip aluminizing process. Experiment was conducted on the low carbon steel substrates with 10mm x 10mm x 2mm dimension. Hot dip aluminizing of low carbon steel was carried out at 750 °C dipping temperature in a molten pure aluminum for 5 minutes. Aluminized samples were heat treated at 600 °C, 700 °C, 800 °C, and 900 °C for 1 hour. X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and EDAX were used in investigation. From the observation, it showed the intermetallic thickness increased with the increase in temperature. The result of EDAX analysis revealed the existence of oxide phase and the intermetallics. The XRD identified the intermetallics as  $Fe_2Al_5$  and  $FeAl_3$ .

### **ABSTRAK**

Rawatan haba diperkenalkan ke keluli karbon rendah bersalut aluminium untuk mempromosikan pembentukan lapisan nipis oksida untuk meningkatkan perlindungan pengoksidaan keluli. Proses ini telah mengubah lapisan intermetallic sedia ada yang terbentuk semasa proses aluminizing panas. Eksperimen dilakukan pada substrat keluli karbon rendah dengan dimensi 10mm x 10mm x 2mm. Mematapkan aluminizing keluli karbon rendah dilakukan pada 750 °C suhu mencelupkan dalam aluminium tulen cair selama 5 minit. Sampel aluminized adalah haba yang dirawat pada 600 °C, 700 °C, 800 °C, dan 900 °C selama 1 jam. Difraksi sinar-X (XRD), Mikroskop elektron imbasan (SEM) dan EDAX digunakan dalam penyiasatan. Dari pemerhatian, ia menunjukkan ketebalan intermetallic meningkat dengan peningkatan suhu. Hasil analisis EDAX menunjukkan kewujudan fasa oksida dan intermetallik. XRD mengenalpasti intermetalkics sebagai Fe<sub>2</sub>Al<sub>5</sub> dan FeAl<sub>3</sub>.

Keywords: Low carbon steel, hot dip aluminizing and intermetallics.

# INTRODUCTION

Low carbon steel is important in structural applications due to its low cost. However, due to the lack of compact-scale formation on the steel surface, it cannot be used for oxidation resistance application, especially in oxidizing environment at elevated temperatures [1]. Surface coating is one of the techniques for improving oxidation resistance of steel. Aluminum coated steel has attracted many researchers due to their excellent mechanical properties and low price [2-6].

Aluminizing the steel surface is an effective method to form and maintain protective  $Al_2O_3$  scale, where the coating provides a reservoir of aluminum [7]. Aluminum owes its excellent corrosion resistance and its use as one of the primary metals of commerce to the barrier oxide film that is bonded strongly to its surface and that, if

damaged, re-forms immediately in most environments. On a fresh surface and then exposed to air, the barrier oxide film is only 1 nm (0.04 min.) thick but is highly effective in protecting the aluminum from corrosion [8].

This study was initiated to convert the aluminum surface into alumina through different heat treatments under dry oxidation environment. The oxidation heat treatment in dry air environment may produce thicker oxide layer on the surface. In this experiment, aluminum protective coating was used in protection of low carbon steel by means of hot dipping process. Then, the hot-dipped samples were heat treated in dry air environment at high temperature to enhance the growth of the oxide layer. Diffusion mechanism involved in these two processes produced and changed the intermetallic layer.

#### EXPERIMENTAL METHOD

Mild steel plates with 50mm x 10mm x 2mm dimension were used in the experiment as substrate. The oxide surfaces were removed by grinding with paper prior to cleaning by ultrasonic. The pure aluminum (99.9% from Aldrich Chemical Company Inc) melted in a graphite crucible at the temperature maintained to 750 degree Celsius. Then, the substrates were dipped into the molten aluminum for 5 minutes. The aluminized steel plates were cut into small pieces of 10mm x10mm x 2mm and cleaned by ultrasonic. The coated samples were heat treated in air at 600 °C, 700 °C, 800 °C, and 900 °C for one hour. Then, the oxidized samples surface was analyzed by using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and EDAX.

# **RESULTS & DISCUSSION**

Fig. 1 shows the formation of alumina onto aluminized steel at different treatment temperatures. Before treatment and At 600 °C treatment, the alumina formed is very thin and not clearly observed. At higher temperature, the alumina layer started to grow uniformly. Table 1 shows the elements detected by EDAX. As the temperature goes up, the aluminum layer starts to deplete. This is due to the heat treatment where some of the aluminum ions react with air forming alumina and some ions diffused futher into the subtrate forming the intermetallic. Fig. 2 shows that the thickness of intermetallic layer increase when treatment temperature increase.

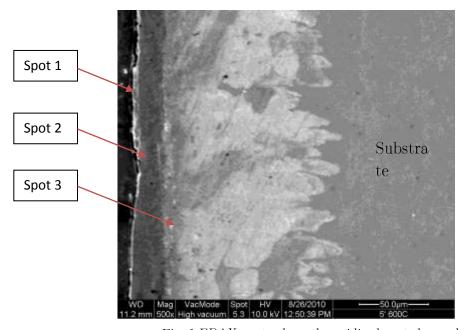


Fig. 1 EDAX spots along the oxidized coated samples

Table 1 EDAX analysis on heat treated samples at different conditions

Sample Element

	Spot 1	Spot 2	Spot 3
Treated at 600°C	Al, O	Al	Al, Fe
Treated at $700^{\circ}$ C	Al, O	Al, Fe	Al, Fe
Treated at $800^{\circ}$ C	Al, O	Al, Fe	Al, Fe
Treated at 900°C	Al, O	Al, Fe	Al, Fe

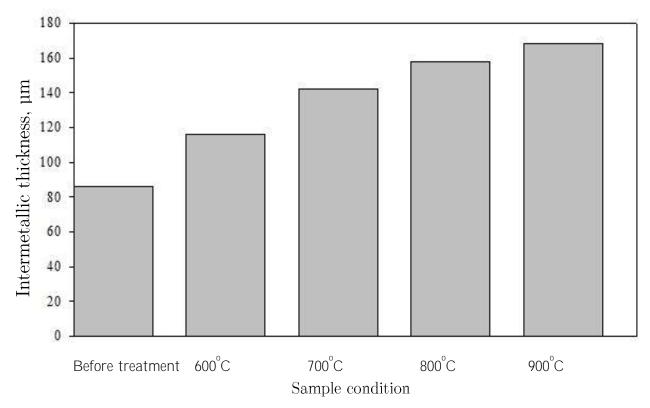


Fig. 2 The intermetallic thickness for samples before and after heat treated at 600 °C, 700 °C, 800 °C, and 900 °C

The XRD analysis (Fig.3) shows the existance of the intermetallic phase. These two major intermetallic phase i.e Fe<sub>2</sub>Al<sub>5</sub> and FeAl<sub>3</sub> peaks revealed is the intermetallic layer—below the alumina. Fe<sub>2</sub>Al<sub>5</sub> is the main intermetallic phase existed in the aluminized layer due to its large lattice interstice favoring a rapid influx of aluminum atoms to the growth front of its crystals at high temperature. As the temperature increase FeAl<sub>3</sub> phase started to grow. The columnar grains grew from Fe<sub>2</sub>Al<sub>5</sub> layer towards the surface aluminum layer when the temperature is higher than 800°C. These grains coarsened and increased with increasing in temperature and it has been suggested that the columnar grains are FeAl<sub>3</sub> [9].

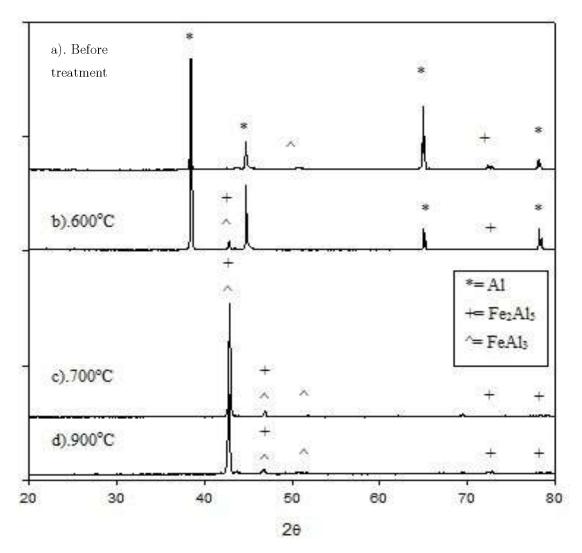


Fig. 3 XRD analysis for samples before and after heat treated at 600 °C, 700 °C, 800 °C, and 900 °C.

# **CONCLUSIONS**

From this study, it can be concluded that the intermetallic phases  $Fe_2Al_5$  and  $FeAl_3$  was formed in between the surface of aluminum and steel substrate. During heat treatment, some of the aluminum ions react with air forming alumina and some ions diffused futher into the subtrate forming the intermetallic. The thickness of the intermetallic layer increase with temperature.

# **ACKNOWLEDGEMENTS**

A deepest gratitude to Malaysian Nuclear Agency and Universiti Kebangsaan Malaysia (UKM-DLP-2011-012) for the supports and research facilities.

#### REFERENCES

1. Wang, C.J. 2006. The high temperature oxidation behavior of hot-dipping Al-Si coating on low carbon steel, Surface and Coating Technology 200: 6601-6605

- 2. Bouayad, A., Gerometta, C., Belkebir, A., and Ambari, A. 2003. Kinetic interactions between solid iron and molten aluminum. Materials Science and Engineering A363: 53-61
- 3. Kwok, C.T., Cheng, F.T., and Man, H.C. 2006. Cavitation erosion and corrosion behaviors of laser-aluminized mild steel. Surface and Coatings Technology 200: 3544-3552
- 4. Yajiang, L., Juan, J., Yonglan, Z., and Holly, X. 2002. Fine structure in Fe<sub>3</sub>Al alloy layer of a new hot dip aluminized steel. Bull. Mater. Sci. Vol. 25 No. 7: 635-639
- 5. Hwang, S.H., Song, J.H., and Kim, Y.S. 2005. Effects of carbon content of carbon steel on its dissolution into a molten aluminum alloy. Materials Science and Engineering A 390: 437-443
- 6. Deqing, W. 2008. Phase evolution of an aluminized steel by oxidation treatment. Applied Surface Science 254: 3026–3032
- 7. Wang, C.J., and Chen, S.M. 2006. Microstructure and cyclic oxidation behavior of hot dip aluminized coating on Ni-base superalloy Inconel 718. Surface & Coatings Technology 201: 3862-3866
- 8. ASM Handbook, Volume 13B, Corrosion: Materials. 2005. ASM International.
- 9. Kobayashi, S., and Yakou, T. 2002. Control of intermetallic compound layers at interface between steel and aluminum by diffusion-treatment. Materials Science and Engineering A338: 44-53