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TEXTURE ANALYSIS AND CORROSION TESTING OF ALUMINIZED CARBON STEEL C45

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Abstract: The texture of hot-dip aluminized carbon steel C45 has been studied. Hot-dip aluminized (HDA) samples were prepared by laboratory experiments. The structure and crystal orientation of the topcoat layer of an aluminized specimen was analyzed through X-ray diffraction. The outcome of XRD reveals that the phase formed after removing the excess aluminum from the top surface (ripples) of the coated sample was FeAl, a non-stochiometric intermetallic with a Fe/Al atomic ratio of 1/1, but it also contains some impurity atoms, which cannot be identified with XRD. These impurity atoms modify the lattice plane distance d values of the FeAl phase, and as a result, the reflections shift. Lattice orientation of such a phase was found to be (110). Corrosion testing of the hot air-exposed (at 700 °C and for several hours) aluminized specimens were also performed, which revealed the still quite high wet corrosion (that is the salt water exposure at ambient air) resistance of the so modified outermost surface zone consisting of iron aluminides formed during the prior high-temperature exposure.

Keywords: Aluminizing, XRD, poles figure, iron aluminides, corrosion resistance

INTRODUCTION

Protection against oxidation and in general corrosion is essential to avoid degradation of the structure's material. Concurrently considering the structure of the coated material and interlayer(s) formed between the substrate and coated material is of primary importance due to the need to perpetuate stable structure withstanding even

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in harsh environments at high temperature. Several researchers have studied the structure of aluminized carbon steel to depict the essential consistency of the coating layers. For example, the morphology structure at the coating/substrate interface was studied by Gul Hameed Awan and Faiz ul Hasan [1] and found on the XRD pattern that the interface was orthorhombic Fe_2Al_5 phase. Others, like Smiglewicz et al. [2] prepared samples of a specific Fe-Al binary alloy for studying its microstructure and phase changes at varying temperatures. They used a special VIM (vacuum induction melting) technique for producing the Fe-Al alloy casting/sample with 58 at.% aluminum. To modify the solidified sample microstructure (e.g., for grain refining), they also added small amounts (in total less than 0.2%) boron, carbon, and zirconium to the binary Fe-Al alloy. By XRD examinations, they identified the $FeAl_2$ phase present in the FeAl alloy prepared with the given composition, which coincides well with the phases appear in the Fe-Al equilibrium phase diagram [3]. Alboom et al. [4], while studying the structure of hot-dip aluminized steel and using several techniques (EDX, EBSD-Mapping, APT) combined with DFT calculations, detected two kinds of phases, $\theta(Fe_4Al_{13})$ and $\eta(Fe_2Al_{5+x})$ just beneath the aluminum cover layer.

In practice, several intermetallic layers that formed during any industrial HDA process, most often show a rather dis-ordered crystallographic structure. Nevertheless, the challenge from their exact identification point of view is that these intermetallic layers are usually fragile and never form an “ideal” stack of thermodynamically stable multilayers.

In our case, after preparing the aluminum-coated C45 steel specimens by the HDA process in one of our laboratories, the primary aim of our investigation was to measure the structure and crystal orientation of the coated sample’s inner interdiffusion Fe-Al layer employing the XRD facilities available at the Institute of Physical Metallurgy, Metalforming, and Nanotechnology. In addition to that, a few preliminary laboratory corrosion testing experiments were also performed to see the high-temperature oxidation resistance behavior of the aluminized C45 steel specimens, because the primary aim of such aluminizing surface treatment of carbon steels is to enhance the corrosion resistance.

1. Materials and Methods

Carbon steel C45 has been chosen considering their favorable mechanical properties and relatively cheap. Samples were off from a steel rod of 32 mm diameter, then cleaned, ground, and polished. For the hot-dip aluminizing route picked to aluminize carbon steel C45 samples, the molten bath was prepared in a graphite crucible with 98.01% aluminum content. Dipping time was set at 2.5 minutes, and the temperature of the commercially pure aluminum melt was 800 ± 2 °C. The disc shape steel samples were pickled in HCl solution (for de-scaling), then were ultrasonically degreased in acetone and stored in ethanol prior to aluminizing.

1.1. The XRD examinations

Due to the rather uneven original surface of the aluminized sample shown in *Figure 1*, first, it had to be prepared for the XRD analysis. The required flatness was achieved by grinding the top rippled surface of the sample; then, the specimen was mounted. X-ray diffraction was utilized to study the phase beneath the top surface of the aluminum and crystal lattice orientation at the top layer of coated aluminum. The applied Bruker D8 Advance type uses cobalt K-α radiation, and it was equipped with an Eulerian cradle. Besides the XRD phase analysis, the texture measurement was also performed on the sample to reveal the preferred orientation of the Fe-Al phase formed. The pole figure was measured up to a 75° tilting degree, and the absorption correction was carried out with ferritic steel powder.

1.2. Hot air corrosion testing

In this part of our studies, first, the hot-dip aluminized samples *(Figure 1)* were exposed to hot air (at 700 °C and for about 24 hours) in an electric resistance furnace in order to check the extent of the surface aluminum coatings transformation to the heat-affected and so modified surface zone mostly consisting of different iron aluminides [6].

Figure 1
Photos of three aluminized steel samples produced
by the laboratory hot-dip aluminizing at 2.5 min. dipping time

2. RESULTS AND DISCUSSION

After careful removal of the excess aluminum from the surface of the aluminized steel discs, the so flattened specimens could be examined by our XRD device. The result of phase analysis detected by XRD is depicted in *Figure 2*. It is revealed that the phase in the interdiffused top layer below the solidified outermost excess aluminum coverage is a non-stochiometric intermetallic FeAl, i.e., with a Fe/Al atomic ratio is very close to 1/1, but it also contains some impurity atoms that have crystal lattice distance that differs from FeAl, which cannot be identified with XRD. These impurity atoms modify the d values of the FeAl phase, and as a result, the reflections shift *(Figure 2)*. From the XRD results of the interdiffused layer, one could conclude that this aluminized layer contains an aluminum content that might reach around 50 at.%. This observation is also consistent with [2]. It is also apparent from the relative intensities of the FeAl phase that this phase is strongly textured. Texture measurements on the FeAl intermetallic phase revealed that the preferred orientation was (110) *(Figure 3)*. According to this, the vast majority of (110) planes of the FeAl phase are parallel with the sample's surface.

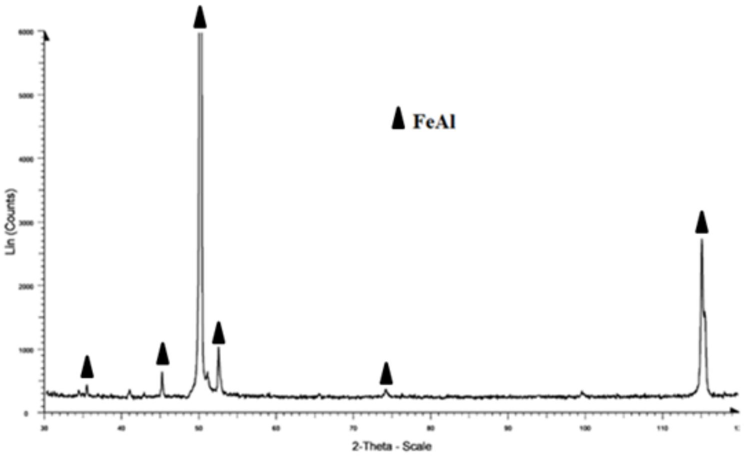

Figure 2

XRD-pattern of aluminized steel C45 interdiffused layer developed during 2.5 dipping time and recorded after removal of the topmost excess aluminum coverage

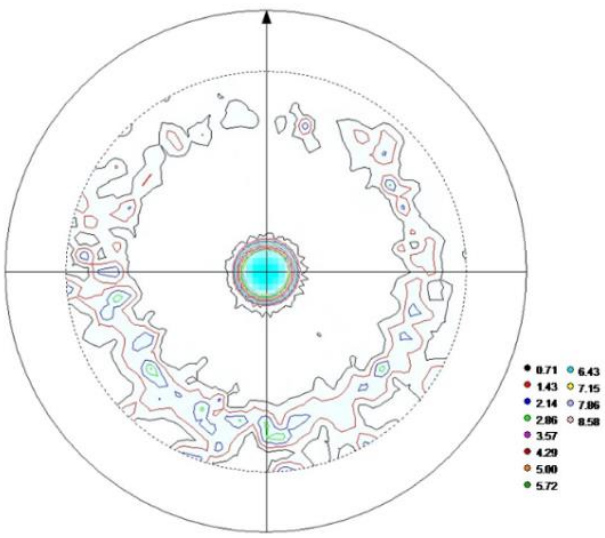

Figure 3

Pole figure of the FeAl intermetallic phase of {110}

High density appears in the middle of the pole figure, which means that the majority of the FeAl phase is oriented. It also can be observed that pole density is above 8 in the middle of the poles figure, which confirms that the developed texture is strong.

Carbon steel sheets aluminized in highly sophisticated and well-controlled industrial lines [6] have a surface aluminum coating of only around a few tens of micrometers. Under our laboratory conditions, however, the excess liquid aluminum was not shaved off either mechanically or using a so-called air knife. Hence, the disc-like steel specimen carried with it much excess adhering liquid aluminum that resulted in a somewhat uneven surface coverage (*Figures 1* and *4a*) after free (i.e., uncontrolled) cooling and solidification in ambient air. *Figure (4c)* shows the optical microscopy image of aluminized steel samples revealed three different zones; (1) steel substrate, (2) intermetallic, and (3) top aluminum.

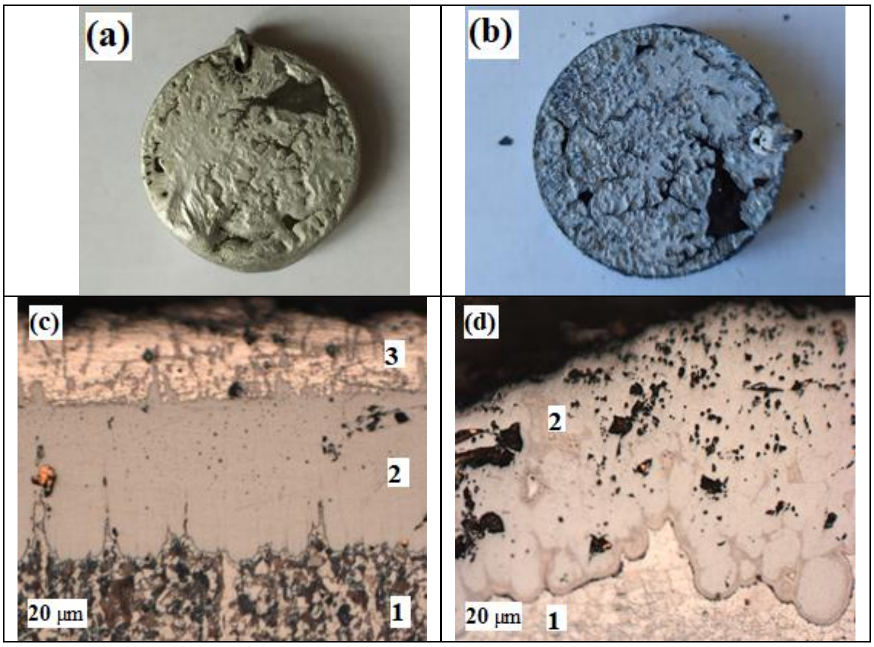

Figure 4

(a) Aluminized specimen (carbon steel C45 disc) with excess aluminum solidified on the top after removing the sample from the molten bath
(b) Aluminized specimen after hot air exposure at 700 °C for several hours showing a much-thinned aluminum surface coverage
(c) Aluminized specimen (carbon steel C45 disc) cross-section (OM image) revealing the layer structure of the hot-dip aluminized sample with 2.5 min dip time
(d) Partially oxidized intermetallic layer of the same sample after hot air exposure at 700 °C for 24 hours

After exposure to hot air inside the furnace, the topcoat surface, which is considered to be aluminum, has transformed (burn-off). Exposure to hot air causes the surface of aluminized steel to initiate a thin protective layer of aluminum oxide that can withstand against further oxidation, nevertheless, the aluminum beneath the aluminum oxide act as a reservoir to healing the top surface of the aluminum and produce aluminum oxide, hindering the oxidation progress inward steel substrate at least for a longer period compared to bare carbon steel. An aluminized sample is shown in *Figure (4a)* and its oxidized counterpart /i.e., after exposed to hot air for several hours/ is placed next to it in *Figure (4b)* just for visual comparison. As it is seen in *Figure (4b)* much of the original excess aluminum coverage not only thinned considerably but also developed a different surface morphology caused by the surface melting effect of the rather high temperature (~ 700 °C) that is a few tens degrees above the melting point of pure aluminum. *Figure (4d)* shows the optical microscopy image for the aluminized samples after exposure to hot air; the structure indicates that the aluminum top layer transformed to intermetallic, only two layers can be seen IM and substrate with some inclusions (oxide) in the intermetallic zone.

Otherwise, as it is well known [6], a relatively thin (<100μm) surface aluminum coatings at higher temperatures will diffuse inwards and form iron-aluminum intermetallic phases relatively fast. It means that in such cases and after several hours [5], it will be the alloyed aluminum surface of the initially aluminum-coated steel, which should resist any further attack of corrosion. And, indeed, it was observed after the neutral salt spray (standard NSS testing) exposure of 96 hours performed in one of our laboratories, that the originally white areas well covered with a thick aluminum layer did resist well the chemical attack of the neutral aqueous salt (NaCl) solution in the air at 35 °C. Moreover, even the originally bare-like small surface regions, where the outer surface must have consisted of iron aluminides, showed only some light brownish discoloration and formation of an only hardly detectable amount of rust-like surface corrosion products formed in the humid and wet salty air environment at around room temperature.

CONCLUSIONS

XRD phase analysis and texture analysis performed on the hot-dip aluminized C45 type steel specimen revealed that the formed FeAl phase is oriented in such a way that the (110) planes are parallel with the surface of the specimen. This result suggests that the FeAl intermetallic phase formed oriented instead of a random manner.

Exposing the hot-dip aluminized specimens to high temperature (700 °C in our case) in hot air must have caused the almost full conversion of the thinner (i.e., <100 μm) aluminum-coated areas to form iron aluminides in a few hours. Therefore, the high-temperature corrosion attack of air (i.e., via the iron oxide scale formation) can be mitigated, i.e., slowed down, as long as the aluminum content of the iron aluminides, something like an aluminum reservoir surface zone, can supply aluminum to form a thin protective aluminium oxide at the outermost surface of the HDA steel products.

As the wet corrosion resistance of commercially pure aluminum is quite high, and even higher than that of zinc, as long as the HDA coated carbon steel is covered with a coherent and sound aluminum layer, its wet corrosion resistance in neutral aqueous environments is also high; and such advantageous properties of the HDA coated carbon steels should contribute well to their even wider applications as that of today.

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REFERENCES

[1] Awan, G. H., ul Hasan, F. (2008). The morphology of coating/substrate interface in hot-dip-aluminized steels. *Mater. Sci. Eng. A*, Vol. 472, No. 1–2, pp. 157–165.

[2] Śmiglewicz, A., Jabłońska, M., Rodak, K., Tomaszewska, A. (2016). Study of the structure and thermal properties of intermetallics from Fe-Al system. *Acta Phys. Pol. A*, Vol. 130, No. 4, pp. 1004–1006.

[3] Kubaschewski, O. (1982). *IRON-Binary Phase Diagrams*. Berlin, Springer-Verlag.

[4] Van Alboom, A., Lemmens, B., Breitbach, B., De Grave, E., Cottenier, S., Verbeken, K. (2017). Multi-method identification and characterization of the intermetallic surface layers of hot-dip Al-coated steel: $FeAl_3$ or Fe_4Al_{13} and Fe_2Al_5 or Fe_2Al_{5+x}. *Surf. Coatings Technol.*, Vol. 324, pp. 419–428.

[5] Kobayashi, S., Yakou, T. (2002). Control of intermetallic compound layers at interface between steel and aluminum by diffusion-treatment. *Mater. Sci. Eng. A*, Vol. 338, No. 1–2, pp. 44–53.

[6] *Hot-dip aluminum-coated steel sheets, Nippon Steel Product Catalog*, https://www.nipponsteel.com/product/catalog_download/pdf/U034en.pdf.