Journal of Coating Science and Technology
http://www.lifescienceglobal.com/journals/journal-of-coating-science-and-technology

Fabrication of Iron Aluminide Coatings (Fe_3Al and $FeAl_3$) on Steel Substrate by Self-Propagating High Temperature Synthesis (SHS) Process

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Abstract: Iron aluminides (Fe_3Al and $FeAl_3$) coatings were fabricated on a steel substrate by self-propagating high temperature synthesis (SHS) method. Raw materials, Fe and Al powders, were mixed at two different stoichiometry ratios (3:1 and 1:3). The mixtures and the substrate were placed in a furnace at 950 °C to ignite the SHS process. Coating phases were investigated using X-ray diffraction (XRD) and Energy Dispersive Spectroscopy (EDS). The microstructure of the coatings was analyzed with optical microscopy (OM) and scanning electron microscopy (SEM). The results confirmed that it is possible to produce Fe_3Al and $FeAl_3$ coatings on steel substrate using SHS method. In addition, the results show that the coatings were composed of two different phases and their microstructures were non-porous and dense. Wear resistance of the coatings were higher than that of the substrate.

Received on 28-02-2017
Accepted on 30-08-2017
Published on 05-10-2017

Keywords: Iron Aluminide Coating, Fe_3Al, $FeAl_3$, Low carbon steel, Self-propagating high-temperature synthesis (SHS), SEM, XRD, Wear test.

DOI: https://doi.org/10.6000/2369-3355.2017.04.02.2

1. INTRODUCTION

Self-propagating high-temperature synthesis (SHS) is an extremely exothermic reaction among chemical components in the condensed phase, which is able to sustain the ignition wave to form a combustion wave. The combustion synthesis has desirable characteristics such as high coating yield and inherently low cost [1, 2]. This novel method can be used for coating materials which combines materials preparation with coating fabrication and deposition of the coating in a single step [3, 4]. The positive points of this method include the preparation of affordable and easy compact powder of raw materials, low-temperature process, process flexibility in controlling the microstructure and chemical composition of the products. Therefore, the main application of this process has been the production carbides, nitrides and aluminides which needs high temperature furnaces and relatively long processing times [5-7].

The iron aluminides Fe_3Al and $FeAl_3$ have been among the most widely studied intermetallics because of their low cost, low density, good wear resistance, ease of fabrication and resistance to oxidation and corrosion [8-12]. Conventional methods of processing iron aluminides, including casting, hot rolling, and powder metallurgy, have been investigated; however, these methods usually require a long time and high cost. In addition, other methods such as thermal spraying, PVD and CVD require special equipment and high cost [6]. Despite these conventional methods, SHS process has appropriate characteristics in coating fabrication. On the other hand, in the SHS process, the reaction is completed within seconds and, in principle, almost 100% of the reactant materials can be utilized for the coating layer [13-18]. The application of SHS process for coating substrate by Fe_3Al and $FeAl_3$ and its tribological, time and cost advantages have not yet been determined.

In this work, Iron aluminides (Fe_3Al and $FeAl_3$) coatings were produced on steel substrate through self-propagating high temperature synthesis (SHS) process. The optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive spectroscopy on the SEM (EDS) were used to investigate the phases of the produced coatings. Applying such analysis, different phases in the coating and interface were determined and the formation of them was discussed. A pin-on-disk tribometer was used to compare the wear resistance of the produced coatings and the substrate.

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E-ISSN: 2369-3355/17

2. EXPERIMENTAL PROCEDURE

Low carbon steel disks with 40 mm in diameter and 10mm in thickness were used as the substrate. Metal powders of iron and aluminum were weighted and dry mixed for 15 min in a low energy ball-mixing mill to have the molar ratios of 3:1 and 1:3, according to the stoichiometry of the reactions (1) and (2) which are the main reactions of the SHS procedure. At high percentages of aluminum, the occurrence of reaction (3) is inevitable and it assures the occurrence of the combustion synthesis, as discussed in our previous study [8].

$3\ Fe + Al = Fe_3Al$, $\Delta H° = -573.7$ (kJ.mol-1) (1)

$Fe + 3\ Al = FeAl_3$, $\Delta H° = -112.5$ (kJ.mol-1) (2)

$4\ Al + 3\ O2 = 2\ Al_2O_3$, $\Delta H° = -3340$ (kJ.mol-1) (3)

Prior to the coating process, the substrate was cleaned in an ultrasonic bath in acetone for 10 min. In order to generate a steady reaction wave in the SHS processing and reduce gases in the reactants, the reactant powder must be compacted. For this purpose, the mixture of reactants were cold-pressed under a 150 MPa uniaxial pressure. The compact pellet was stacked on top of the substrate disk without any pressure, then they were placed into the air furnace at 950 °C temperature to ignite the SHS reaction. The samples were then removed from the furnace and were allowed to cool in air. The mentioned uniaxial pressure and temperature should be applied to assure the occurrence of the SHS without any sputtering to the surrounding.

Coating phases were identified by X-ray diffraction. The composition of the coating layers formed by SHS reaction were determined by EDX and SEM analyses. The surface of coating samples were observed using optical microscopy and a Zeiss (LEO) 1450VP Scanning Electron Microscope (SEM). Prior to the OM, SEM and EDS examinations, the specimens were polished and etched with a solution of CH_3COOH 33%, HNO_3 33%, HF 1%, H_2O 33%. Specimens with dimensions of 19 mm × 19 mm × 4 mm were machined from the samples for tribological testing. Tribological tests were carried on a pin-on-disc tribometer under dry sliding condition to determine the mass loss with respect to time, as presented in detail elsewhere [8].

3. RESULT AND DISCUSSION

The metallographic images of the coatings were presented in Figure **1**. It can be seen that both of coatings consist of two 2 different phases which will be discussed in detail. The XRD patterns of the coatings were illustrated in Figure **2a** and **b**. The observed peaks in Figure **2a** and **b** revealed that $FeAl_3$ coating is composed of $FeAl_3$ and Al phase, while Fe_3Al coating is comprised of Fe and Fe_3Al phase.

The SEM images of the coatings were illustrated in Figure **3**. Different phases in the coating are marked by A, B, C and D regions. Moreover, the quantitative results of the EDS analysis of these regions were presented in Table **1**. Considering these analyses, it can be seen that the $FeAl_3$ coating (Region A and B) is composed of $FeAl_3$ and aluminum phase, while the Fe_3Al coating (Region C and D) is comprised of iron and Fe_3Al. The weight percentages of aluminum in the A and C regions in the coatings are about 58% and 18%, respectively, which indicates that the A region is $FeAl_3$ and the C region is Fe_3Al. As a result, it can be inferred from the analysis that iron aluminide phases are formed after SHS reaction. In addition, in the powder mixture with molar ratios of 1:3 (Fe: Al) which contains high percentage of aluminum, a small part of the existing aluminum in the compact reacted with oxygen during combustion synthesis which created a considerable amount of heat and a small percentage of Al_2O_3 at the edge of the $FeAl_3$ coating (Reaction E). Figure **4** represents the EDS analysis of this phase. It can be seen aluminum had reacted with oxygen during the process and a very thin layer of Al_2O_3 had formed at edges of the coating. This layer is highly fragile.

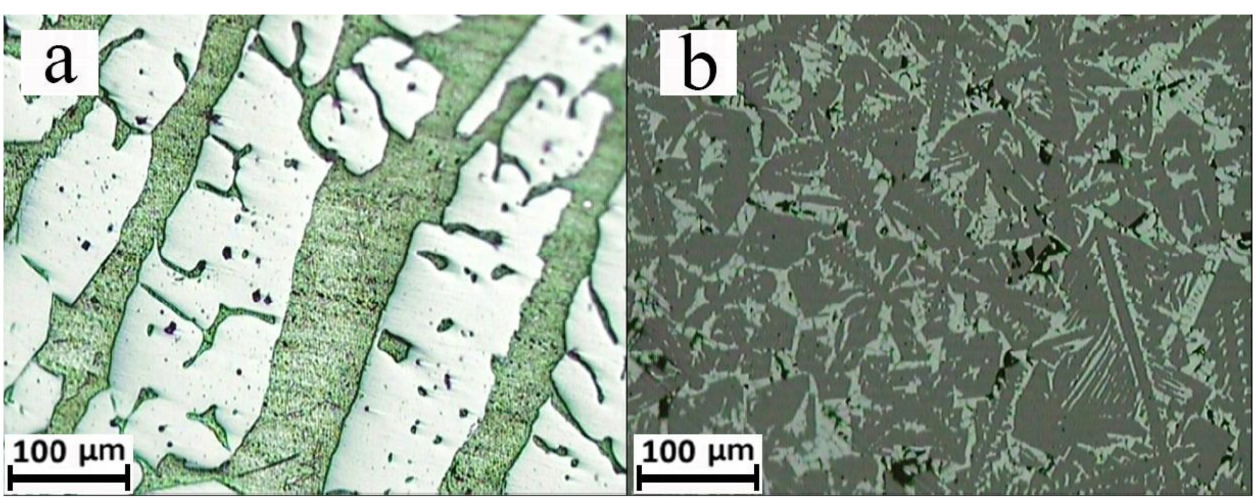

Figure 1: OM micrographs determining microstructure of **a**) $FeAl_3$, and **b**) Fe_3Al coatings.

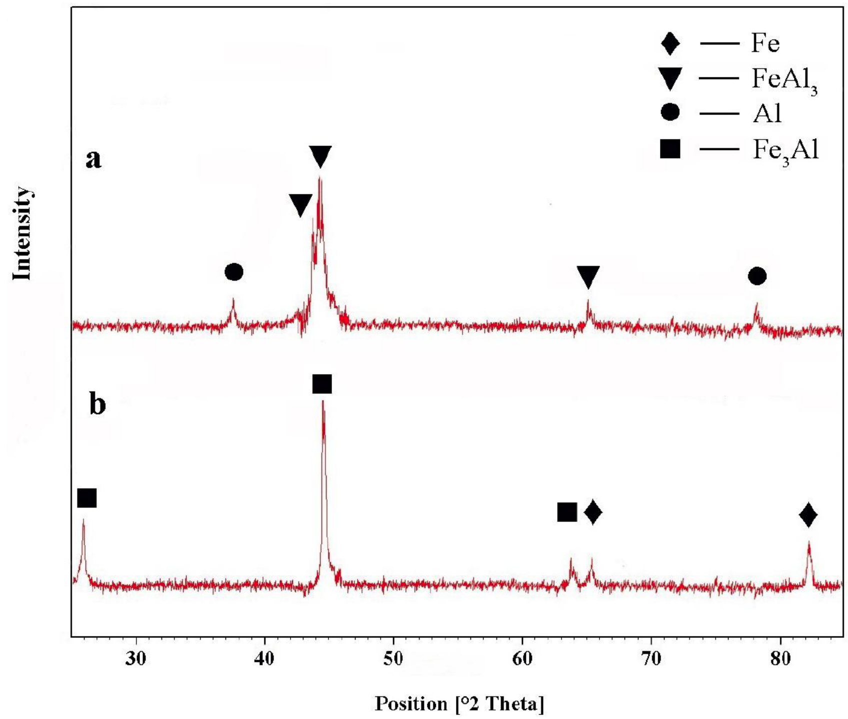

Figure 2: XRD patterns of **a**) $FeAl_3$ coating layer, **b**) Fe_3Al coating layer.

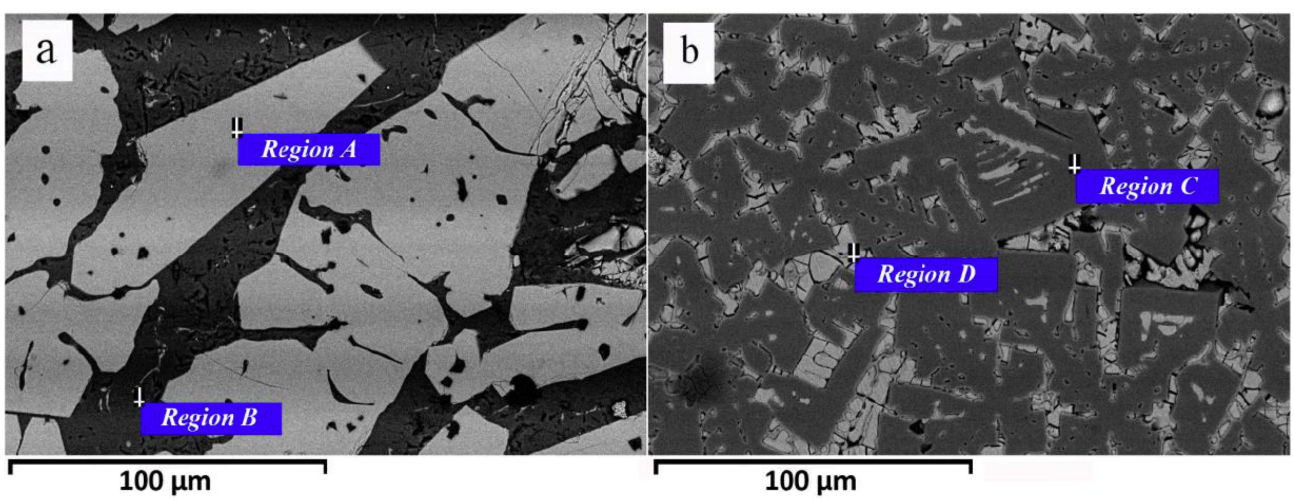

Figure 3: SEM images determining microstructure of **a**) $FeAl_3$ (Region A and B), and **b**) Fe_3Al coatings (Region C and D).

Table 1: The Quantitative EDS Results (wt. %) of Substrate, $FeAl_3$ and Fe_3Al Coatings (Region A, B, C, D and E)

Element	Segment				
	Region A	Region B	Region C	Region D	Region E
Fe	41.41	0.0	80.67	99.47	0.0
Al	58.33	100	18.24	0.0	62.8
C	0.0	0.0	0.60	0.13	0.0
Cr	0.12	0.0	0.23	0.21	0.0
Mn	0.14	0.0	0.26	0.19	0.0
O	0.0	0.0	0.0	0.0	37.2

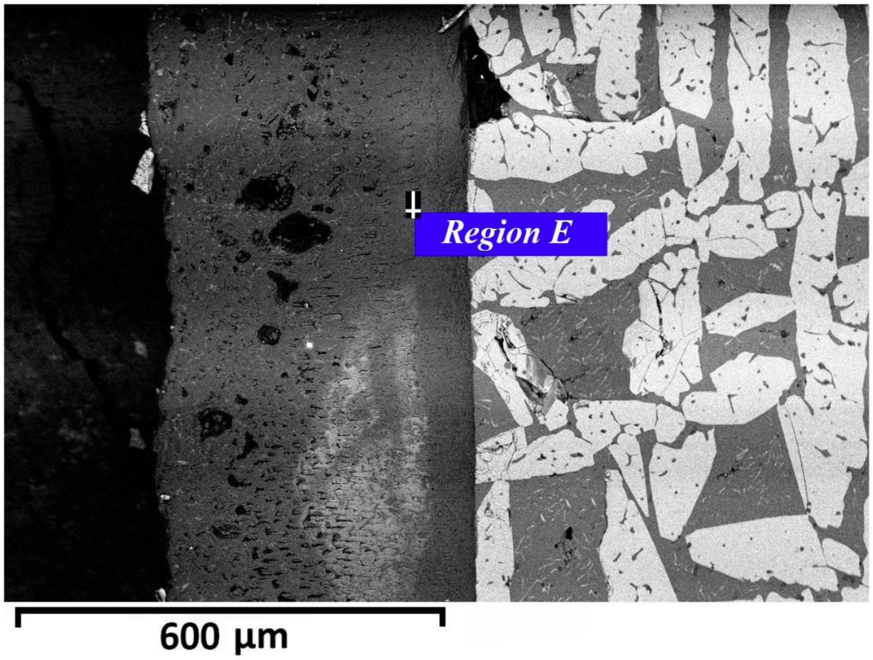

Figure 4: SEM image from the edge of $FeAl_3$ coating (Region E).

The third and fifth columns of Table **1** demonstrated the presence of pure aluminum and pure iron in B and D regions, respectively. It can be stated that very small fraction of initial aluminum and iron powder did not participate in reaction 1 and 2 and remained as a separate phase in the coatings. Quantitatively, the amount of these phases were much lower than iron aluminides phases.

With a cursory glance to the Fe-Al phase diagram, it can asserted that the stability of the FeAl phase is much more extensive than that of Fe_3Al and $FeAl_3$ phase [19]. Among all the iron-aluminum intermetallic compounds, FeAl has the widest range of weight percentage. Therefore, fabrication of a single phase FeAl coating is completely feasible [8]. In contrast, it is not practicable to prevent the formation of iron, aluminum and Al_2O_3 phases during the SHS reaction of Al and Fe powders in the air (at very low or high percentages of Al element). In fact, at high percentage of aluminum ($FeAl_3$) a small amount of aluminum in the powder mixture reacts with oxygen during the combustion synthesis which is formed a thin layer at the edge of the $FeAl_3$ coating. On the other hand, at lower percentage of aluminum, the amount of produced Al_2O_3 reduced. In Fe_3Al coating with very low percentage of aluminum, no Al_2O_3 phase is produced, but small fraction of Fe powder from raw materials did not react and remained as a phase in the produced coating.

The wear mass loss of Fe_3Al, $FeAl_3$ and low carbon steel substrate are illustrated in Figure **5**. It can be obviously asserted that wear mass loss of all coated samples was

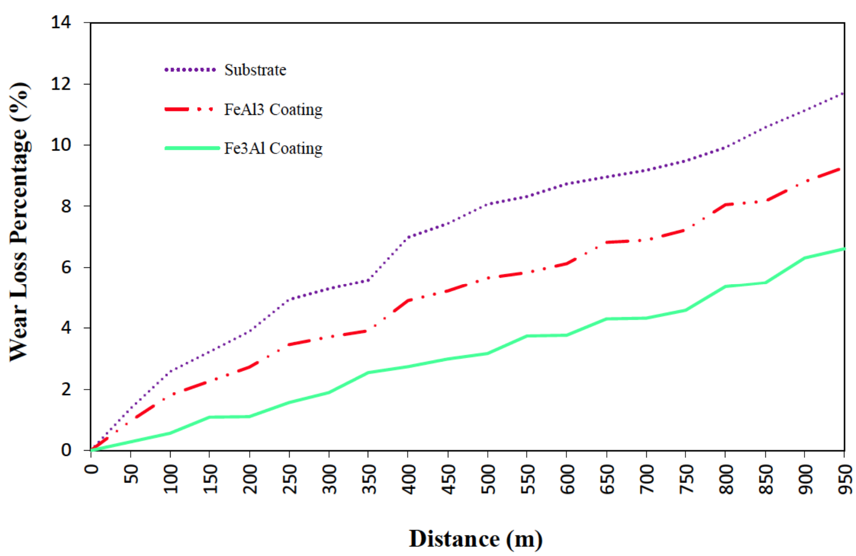

Figure 5: Comparison of wear mass loss of substrate, $FeAl_3$ and Fe_3Al coatings.

significantly lower than that of the substrate which shows that the coated layers have considerable adherence to the surface of the sample. The mentioned results were achieved just after one-step coating process.

Fabrication of iron aluminides coating by SHS process has other advantages including saving time and low cost. Unlike other fabrication methods such as casting, the required energy for the SHS reaction will be prepared by the raw materials. As a result, a low temperature furnace and just some seconds are enough to assure the ignition of SHS reaction.

4. CONCLUSION

It has been shown in this study that Fe_3Al and $FeAl_3$ coatings can be successfully fabricated on a low carbon steel substrate by the SHS process. Coatings have excellent adherence to the substrate due to the high temperature of combustion synthesis reaction and local fusion of substrate in the interface. The Fe_3Al coating layer was composed of iron and Fe_3Al phase, while the $FeAl_3$ coating layer was comprised of aluminum and $FeAl_3$ phase. The short ranges of stability of the Fe_3Al and $FeAl_3$ phases in the Fe-Al phase diagram led to the difficulty of controlling the products of SHS reaction and fabricating single phase coating. In $FeAl_3$ coating, a few percent of aluminum reacted with the oxygen of the air during SHS process which released a significant amount of heat and produced a thin layer of Al_2O_3 on the edges of the coating. The wear life of all coated specimens was more than the uncoated substrate. The Fe_3Al and $FeAl_3$ coatings had much better wear resistance compared to low carbon steel substrate.

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