

Investigation of Al₂O₃-Modified Aluminide on 304SS: Microstructural Evolution, Hardness and Growth Kinetic through Slurry Aluminizing

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Abstract. The microstructure and growth kinetic of alumina (Al₂O₃)-modified aluminide coating were investigated at 650°C, 680°C, and 700°C for various durations (4, 6, 8, and 10 hours) using the slurry aluminizing process. The heat-treated samples were analyzed through scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD) to assess microstructural evolution, elemental composition, and phases of the coating. SEM observations revealed a two-layer aluminide coating, comprising an Al-rich intermetallic (FeAl₃) and a Fe-rich intermetallic (FeAl). Microhardness tests showed that FeAl₃ had hardness values ranging from 880 to 990 HV, while FeAl, with values between 610 and 700 HV. The growth kinetics indicated that the thickness of the aluminide layers increased with both the aluminizing temperature and time, following a parabolic growth law. The activation energy for the growth of FeAl was 343.15 kJ/mol.

1.0 Introduction

The issue of material corrosion at high temperatures remains a critical challenge in thermal plant systems. Iron-based alloys are mainly used in the high-temperature regions of hot sections and combustion systems. Austenitic stainless steel is considered a promising choice for high-temperature applications due to its excellent corrosion resistance, strong mechanical properties, and thermal stability across a wide temperature range. This is primarily due to the presence of chromium, which forms a protective passive film that can shield the material and self-repair in the presence of oxygen. However, this chromium oxide loses its protective capability beyond certain temperatures. To improve oxidation and corrosion resistance, the surfaces of these alloys can be enriched with aluminium to form alumina [1].

Several methods have been developed for aluminizing steels, including hot dip aluminizing (HDA), pack cementation, chemical vapor deposition, physical vapor deposition, and slurry aluminizing. Among these, the slurry technique is favoured for its simplicity and cost-effectiveness, providing easy control over coating thickness along with good processability and compositional flexibility [2]. This method involves using aluminium to diffuse into or react with the substrate (iron) to create a surface layer of intermetallic phases. Diffusion coatings are known for their excellent adhesion to the substrate and uniform distribution [3]. The characteristics of the coating layers and their constituents are influenced by various factors, including composition, metal source, temperature, and time [4]. Understanding the formation and growth of iron aluminide layers in aluminized steel during service at 600–700°C is essential for evaluating their long-term performance. Information on aluminide coatings for austenitic steels has been reported in the literature [1,5,6].

The Fe-Al binary phase diagram reveals numerous intermetallic phases, including Fe₂Al₅, FeAl₃, FeAl, and Fe₃Al, which form depending on the temperature and time [7, 8]. Fe₂Al₅ and FeAl₃, which are Al-rich intermetallic compounds, emerge due to vacancies in the crystal structure and are known for their extreme brittleness and high rates of corrosion and oxidation [9]. Conversely, the Fe-rich

phases, such as FeAl and Fe₃Al, provide the steel surface with excellent corrosion and oxidation resistance. Researchers have investigated various methods to mitigate the brittleness of these intermetallic by adding alloying elements like Si and Zr to the aluminide coating, thus improving its high-temperature oxidation properties [10, 11]. This study focused on incorporating a specific ratio of alumina (Al₂O₃) into the aluminide coating to affect the morphology and growth kinetics of the intermetallic layers. The impact of growth in inward and outward directions on the microstructure and properties of the Al₂O₃-modified aluminide coating has yet to be thoroughly explored. The present study employed the slurry aluminizing technique to form Al₂O₃-modified aluminide coatings on 304 SS. The samples were analysed using various techniques to assess the microstructure, coating thickness, hardness, phase identification, and growth kinetics resulting from the interdiffusion between the coating and the substrate.

2.0 Materials and Method

The substrate used was 304 SS grade of austenitic stainless steel. The specimens were cut to dimensions of 10 mm x 10 mm x 2 mm, then manually ground, cleansed, and subjected to ultrasonic cleaning in acetone. Afterward, they were rinsed with distilled water and dried using a hot air dryer. The slurry consisted of 57% solvent binder system, made of distilled water and polyvinyl alcohol (PVA), and 43% additives comprised of 70% aluminium and 30% alumina (Al₂O₃). The composition formula used was in accordance with [12].

The slurry was prepared by heating distilled water to the glass transition temperature of PVA, which is 85°C. PVA powder and other additives (Al and Al₂O₃) were then mixed using a continuous magnetic stirrer. The prepared slurry was sprayed on the substrate with a Sagola gun. The coating process was controlled to ensure a total mass/area ratio of 15~30 mgcm⁻². The aluminizing process was conducted at 650°C, 680°C, and 700°C for 4, 6, 8, and 10 hrs in a carbolite furnace under an argon environment with a heating rate of 10°C min⁻¹, and finally, cooled in the furnace to room temperature. After the heat treatment, the samples were mounted in epoxy resin, ground with silicon carbide papers ranging from 240 to 2000 grits, polished into a mirror-like using alumina paste down to 0.05 µm, and etched with a standard Keller etchant for 3 to 5 seconds.

The cross-sections of material were analysed using a Hitachi TM 3000 SEM, an energy dispersive X-ray spectrometer (EDX), and X-ray diffraction (XRD Bruker Advanced D8) with Cu Kα radiation (λ = 0.15406 nm). XRD scans were conducted over a 2θ range of 10° to 90° to identify the phases present in the aluminized samples. Coating thickness was determined using ImageJ software, while microhardness measurements were performed on the coating samples with a 25 g load for 15 seconds using a LECO micro-indentation hardness tester (LM248AT). The activation energy (Q) of the intermetallic layers was calculated using the Arrhenius equation.

3.0 Result and Discussion

3.1 Morphology and phase analysis of coating

Figure 1 shows the SEM cross-section image of the aluminide coating samples heat treated at 650°C, 680°C, and 700°C for 4, 6, 8, and 10 h. The image revealed two-layer structure (outer and inner layer) present at the diffusion and interdiffusion zones following the aluminizing process. The interface between the coating and substrates was smooth and continuous for all the heat-treated samples. Based on the observations, it can be concluded that the addition of Al₂O₃ filler reduced the possibility of local melting and sintering of Al during the aluminizing process which is responsible for the smoothening of the interface between the coating and the substrate [13,14]. No traces of cracks were identified in the coating, which could be due to the smaller CTE mismatch between aluminide coating (17 × 10⁻⁶ K⁻¹) and stainless steel 304 (18 × 10⁻⁶ K⁻¹) [15].

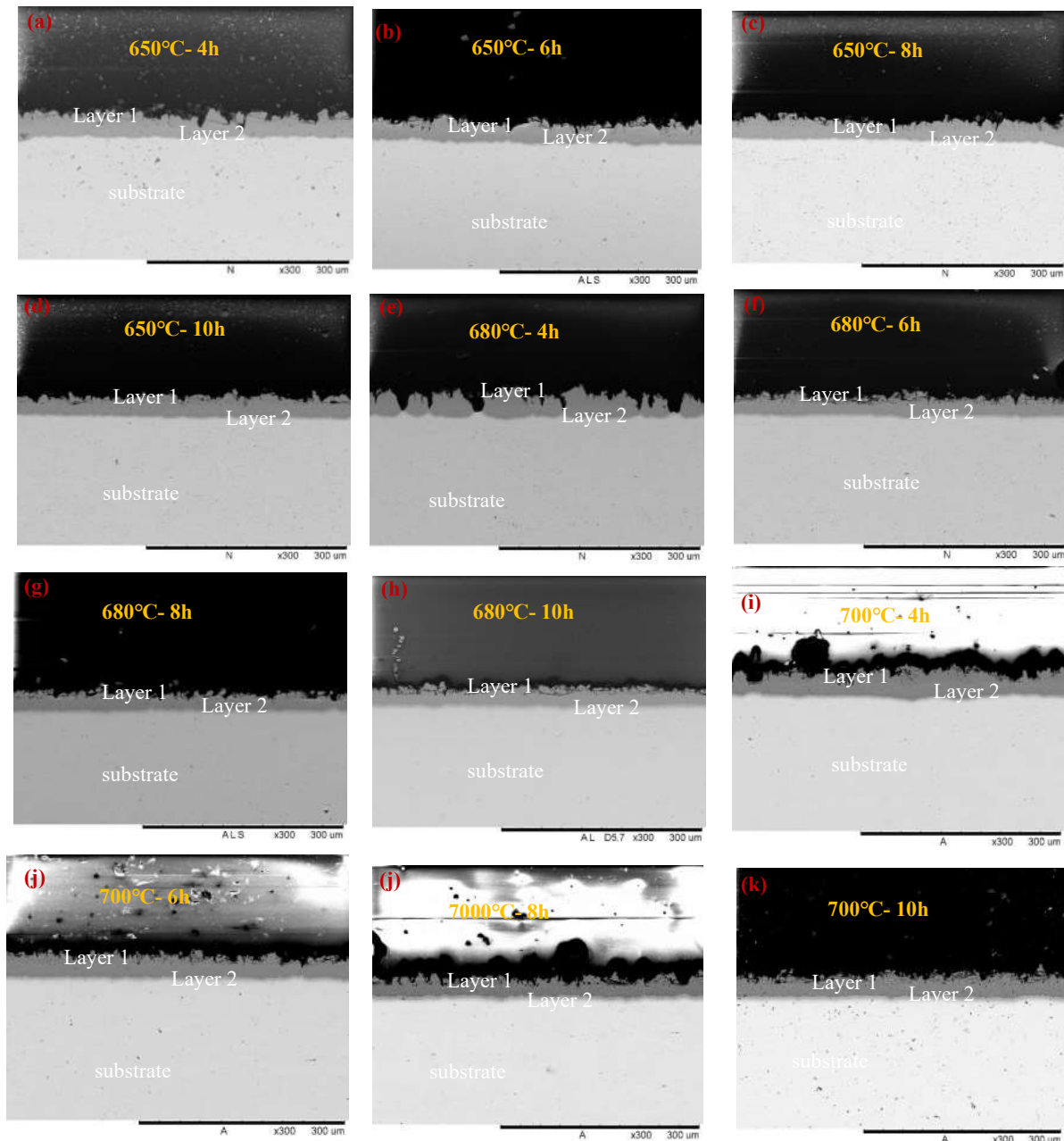


Fig. 1. SEM micrograph of Al_2O_3 -modified aluminide coating heat treated at 650°C, 680°C and 700°C for 4, 6, 8, and 10 h (300X).

The interdiffusion between the active Al and Fe determines the formation of the coating layers, as shown in Figure 1, resulting in the development of layer 1 and layer 2. Sample heat treated at 650°C mainly comprised of layer 1 with a very thin interface of layer 2. While for coatings formed at 680°C and 700°C, layer 2 grew significantly and became more uniform. The coating layers increase as the aluminizing temperature and time increases. The elemental composition of the layers was analyzed using EDX to identify the intermetallic phases, with chromium and nickel forming a solid solution as shown in Table 1. The EDX spot for both heat treatment temperatures were depicted in Figures 2 and 3.

As shown in Figure 1, layer 1 exhibited a higher concentration of aluminum, and EDX analysis identified this phase as FeAl_3 , an Al-rich intermetallic compound. In contrast, layer 2, which is closer to the substrate, contained a higher concentration of iron, identifying it as the Fe-rich intermetallic FeAl . In summary, the phase transformation in the aluminide coating begins with the formation of FeAl_3 , which, upon prolonged soaking, transitions to Fe_2Al_5 and eventually evolves into FeAl and Fe_3Al .

Table 1. EDX analysis of modified Al_2O_3 -aluminide coating on 304 SS heat treated at 650°C and 680°C for 10 h.

650°C				680°C			
Elements	spot 1	spot 2	spot 3	Elements	spot 1	spot 2	spot 3
C	39.2	42.38	40.83	C	35.09	37.73	40.79
Al	41.87	26.68	18.17	Al	45.36	33.19	2.99
Cr	3.56	6.15	8.19	Cr	3.44	4.4	10.59
Fe	15.37	19.82	30.48	Fe	16.11	19.08	42.58
Ni	-	4.96	2.33	Ni	-	5.59	3.05

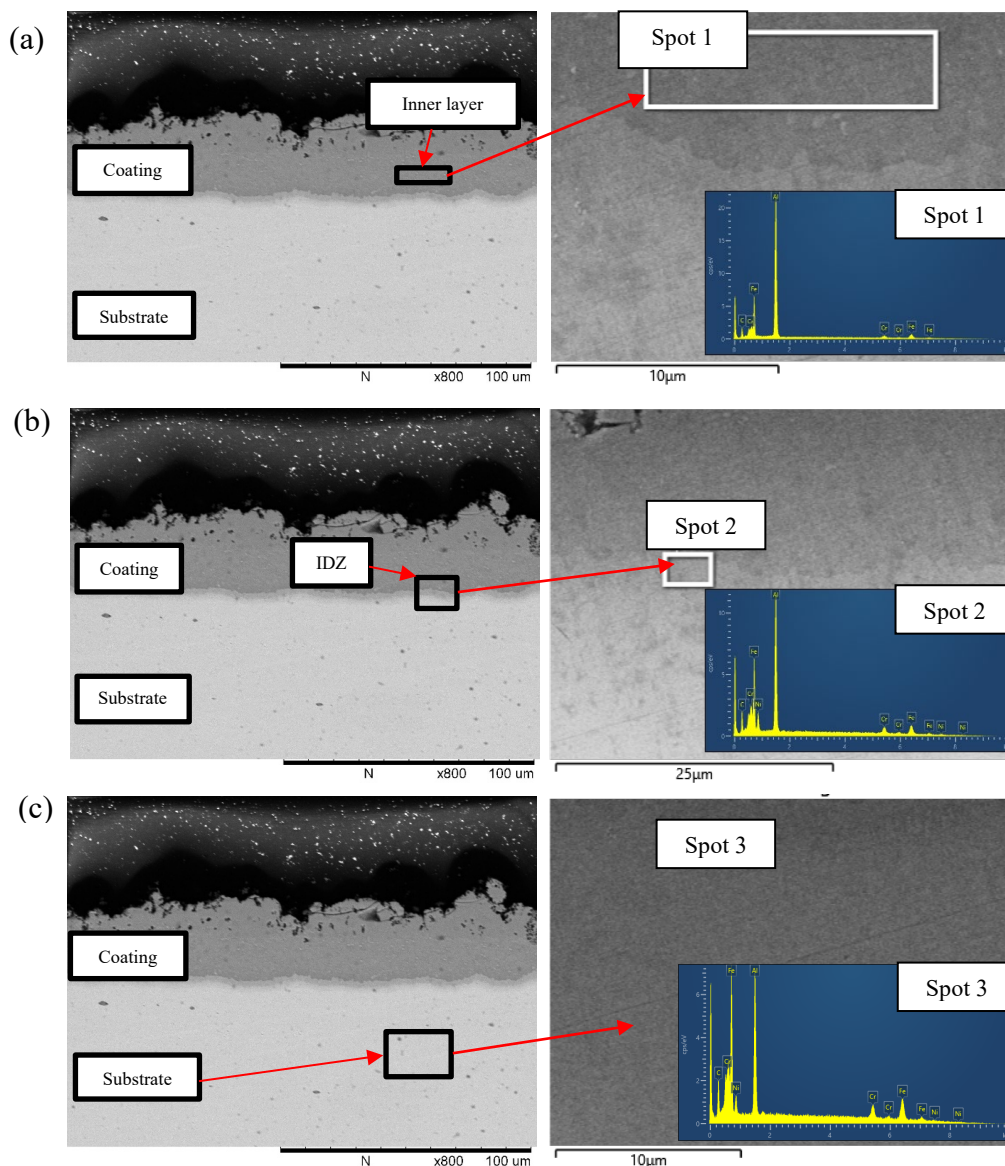


Fig. 2. EDX spot of Al_2O_3 -modified aluminide coating heat treated at 650°C for 10 h at various location (a) inner layer, (b) interdiffusion zone (IDZ) layer, and (c) substrate.

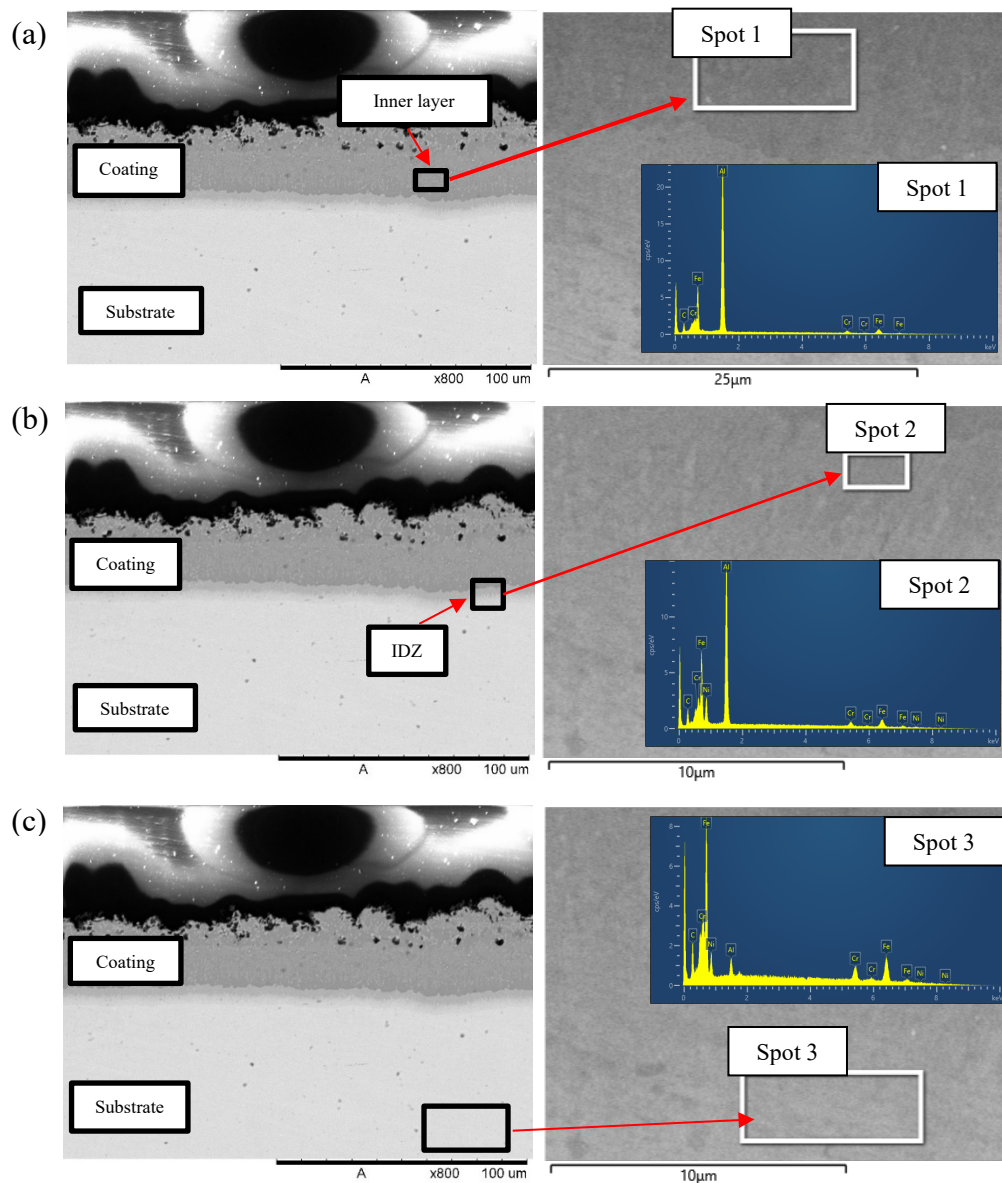


Fig. 3. EDX spot of Al_2O_3 -modified aluminide coating heat treated at 680°C for 10 h at various location (a) inner layer, (b) interdiffusion zone (IDZ) layer, and (c) substrate.

X-ray diffraction (XRD) analysis was employed to confirm the elemental composition of the phases analysed by EDX. Figures 4, 5 and 6 illustrate the XRD patterns of the aluminide coating heat treated at 650°C , 680°C , and 700°C for 4, 6, 8, and 10 hours. The results indicated that the aluminide layers' phase composition matched the EDX analysis. Specifically, the presence of the FeAl_3 phase (ICDD PDF number: 00-069-0296) was consistent across all heat-treated samples [7]. Additional phases identified included of FeAl (ICDD PDF number: 04-002-1309), Cr (ICDD PDF number: 04-007-8594), FeNi (ICDD PDF number: 00-003-1016), and Fe (ICDD PDF number: 00-034-0529).

The FeAl_3 phase was detected at all temperatures, while the formation of FeAl was observed at 680°C and both FeAl and FeAl_3 present at 700°C . The volume fractions of FeAl_3 and FeAl phases at 680°C for different durations were determined through Rietveld refinement. After 4 hours, the FeAl_3 phase was observed with a volume fraction of 42.3%. As the soaking time increased to 6 hours, the volume fraction of FeAl_3 was measured at 40.7%. At 8 hours, this volume fraction further decreased to 37.6%. When the soaking time was extended to 10 hours, both FeAl_3 and FeAl phases were detected, with volume fractions of 14.5% and 85.5%, respectively. These values are consistent with previous studies [16,17]. Overall, increasing soaking times led to the formation of FeAl , which supports earlier findings [7].

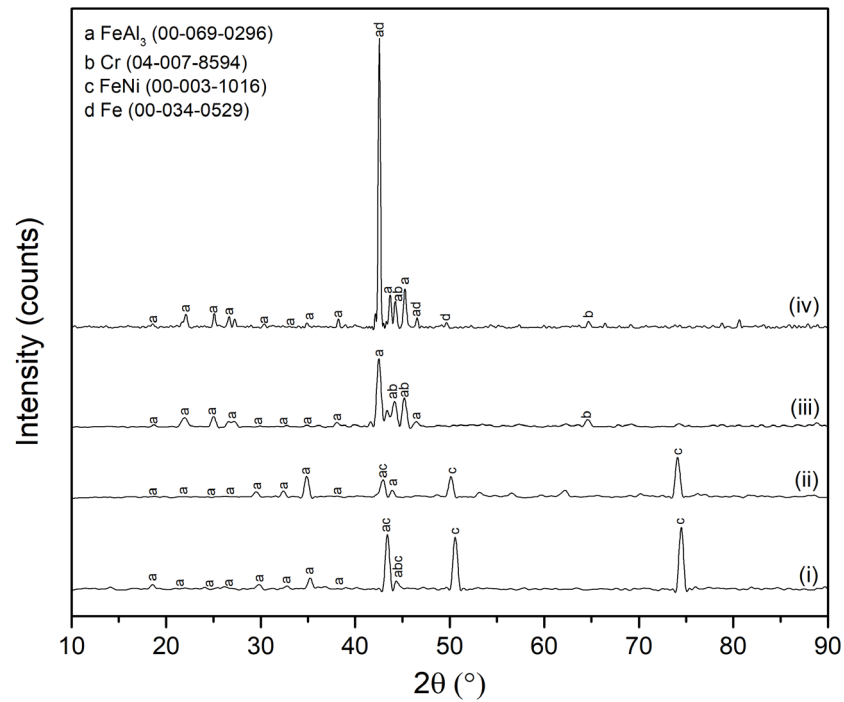


Fig. 4. XRD Pattern of Al_2O_3 -modified aluminide coating heat treated at 650°C for i) 4 hours, (ii) 6 hours, (iii) 8 hours, and (iv) 10 hours.

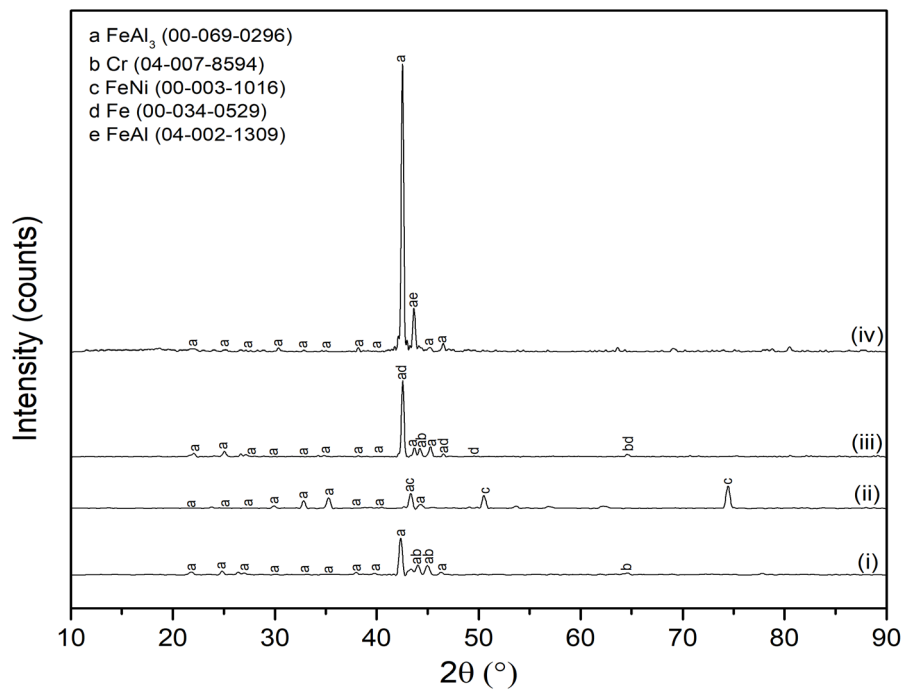


Fig. 5. XRD Pattern of Al_2O_3 -modified aluminide coating heat treated at 680°C for i) 4 hours, (ii) 6 hours, (iii) 8 hours, and (iv) 10 hours.

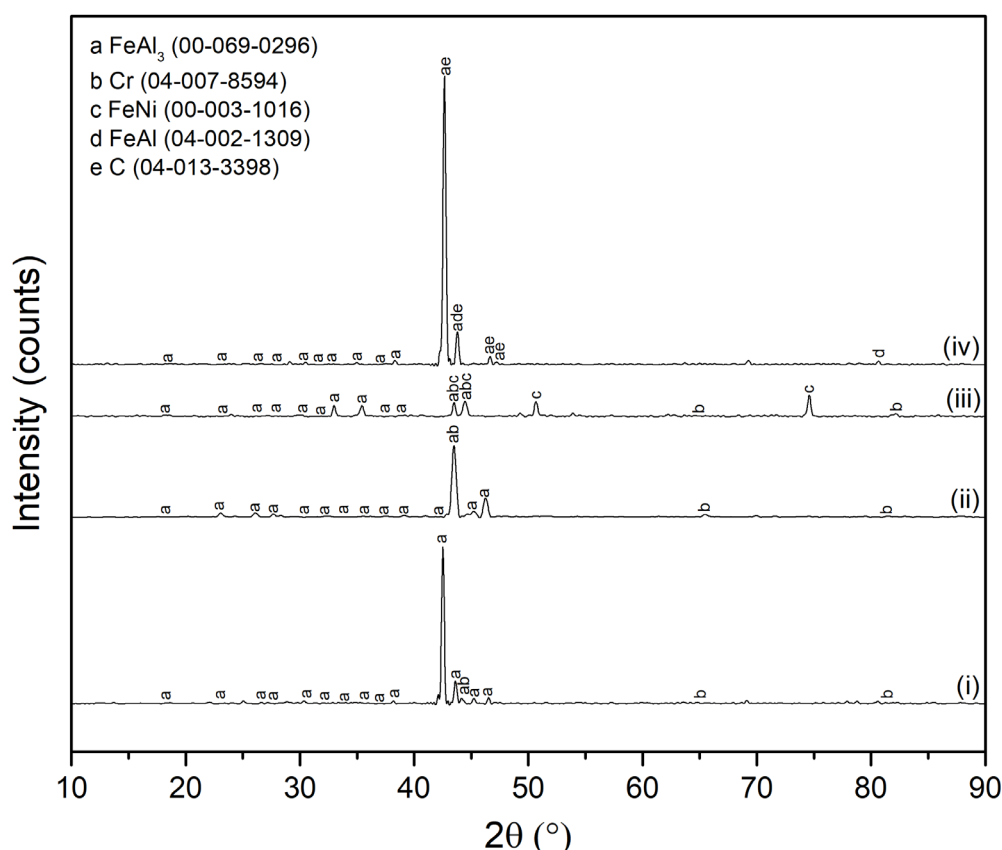


Fig. 6. XRD Pattern of Al_2O_3 -modified aluminide coating heat treated at 700°C i) 4 hours, (ii) 6 hours, (iii) 8 hours, and (iv) 10 hours.

3.2 Microhardness measurement

Figure 7 illustrates the hardness values of the coating layers in samples heat-treated at 650°C , 680°C , and 700°C for 4, 6, 8, and 10 hours. According to Figure 1 and the EDX/XRD analysis, layer 1 was identified as FeAl_3 across all aluminizing temperatures and soaking times. The hardness of FeAl_3 ranged from 880 to 990 HV, with previous studies reporting a hardness of 850 HV for FeAl_3 [18]. The highest hardness observed for FeAl_3 are attributed to the addition of Al_2O_3 in the aluminide coating. As shown in Figure 7a, the sample heat-treated at 650°C for 4 hours had the lowest hardness of 880 HV, while the sample heat-treated at 700°C for 10 hours achieved the highest hardness of 990 HV. The FeAl_3 phase exhibits the highest hardness due to its high aluminium content, classifying it as an Al-rich intermetallic compound. The intermetallic layer known as FeAl was formed at transition zone between FeAl_3 and the substrate. This layer located closer to the substrate and has higher concentration of Fe due to diffusion during the aluminizing process, forming Fe-rich intermetallic compound. The FeAl has hardness in the range of 600 to 720 HV.

Figure 7b shows the hardness of FeAl was 610 HV for the sample heat-treated at 650°C for 6 hours, while the sample heat-treated at 700°C for 10 hours exhibited the highest hardness (710 HV). Previous studies have reported that FeAl hardness ranges from 580 to 710 HV [19]. The observed increase in hardness is attributed to the higher aluminizing temperature and extended soaking time [13,20]. Fe-rich intermetallic compounds (IMCs), such as FeAl and Fe_3Al , are known for their superior chemical stability, toughness, corrosion, and wear resistance compared to Al-rich IMCs (Fe_2Al_5 and FeAl_3), making them more suitable as a protective layer for steel coatings [21].

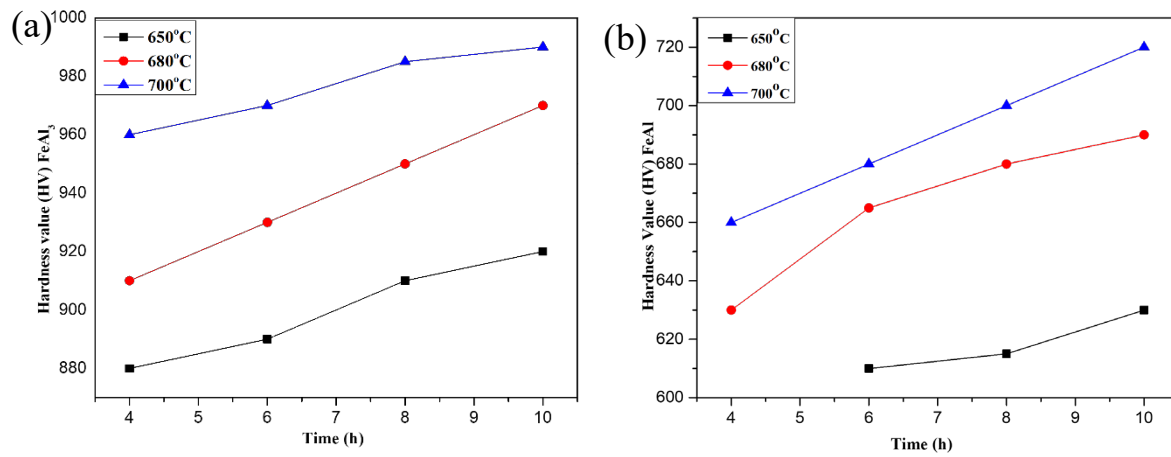


Fig. 7. Microhardness of modified Al₂O₃-aluminide coating for sample heat-treated at 650°C, 680°C and 700°C for 4, 6, 8, 10 h (a) FeAl₃, and (b) FeAl.

3.3 Growth kinetic of aluminide layer

Interdiffusion between the Al and Fe determines the growth rate of the aluminide layer. It has been proposed that the growth rate of the aluminide layer is a diffusion control [22,23]. The thickness of the intermetallic layer was assessed for all samples heat-treated at 650°C, 680°C, and 700°C for various soaking times, as illustrated in Figure 8(a) and (b). The thickness of the aluminide layer exhibits a linear relationship with diffusion time, as derived from Eq. 1. Figure 9 shows a plot of the square of the aluminide layer (X) against time (s), determining the growth rate (K). In addition, Figure 10 depicts a linear relationship between $\ln K$ and the reciprocal of temperature ($1/T$). The slope increases with increasing temperature indicating that the growth of the aluminized layers follows parabolic kinetics from Eq. (2 and 3) of Arrhenius equation to determine the activation energy (Q).

$$X^2 = Kt \quad (\text{Eq.1})$$

$$K = K_o \exp\left(\frac{-Q}{RT}\right) \quad (\text{Eq. 2})$$

$$\ln K = (\ln K_o) - Q/RT \quad (\text{Eq. 3})$$

where X (μm) stands for the coating thickness, K ($\mu\text{m}^2/\text{s}$) represents the growth rate constant, and t(s) is the aluminizing time.

K_o refers to the pre-exponential constant, Q represents activation energy (J), T(K) indicates absolute temperature, and R is the gas constant 8.314J/(K.mol).

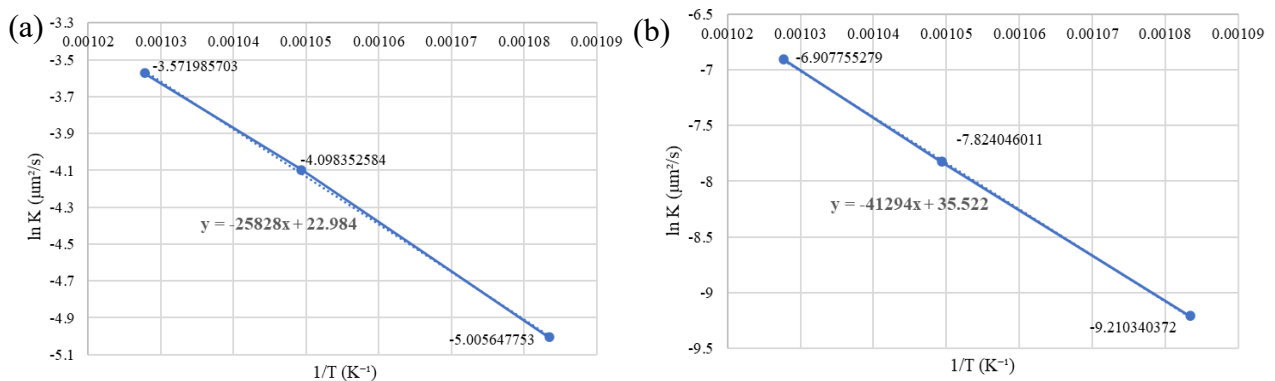


Fig. 8. Coating thickness of sample heat treated at 650°C, 680°C and 700°C against the heat treatment time (a) intermetallic aluminide layers, and (b) FeAl thickness.

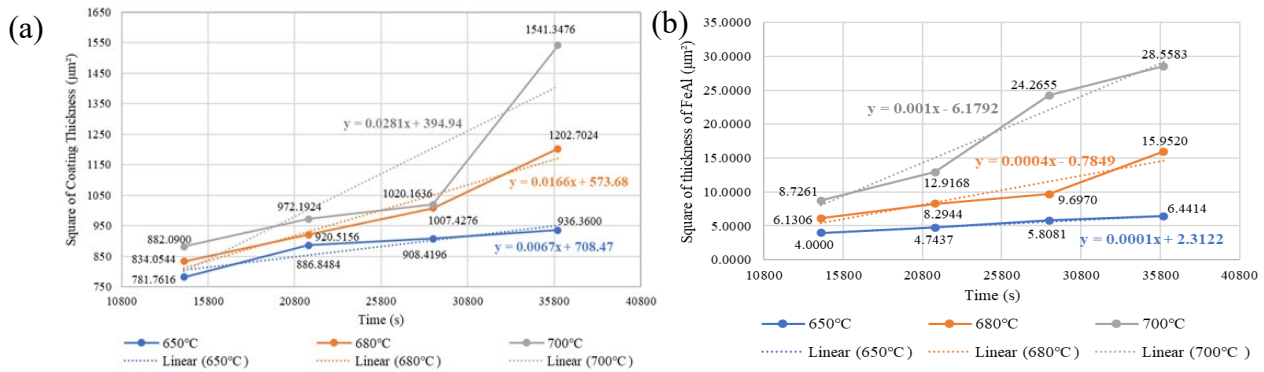


Fig. 9. Relationship between the square of intermetallic layers thickness and diffusion time for (a) intermetallic aluminide layers, and (b) FeAl.

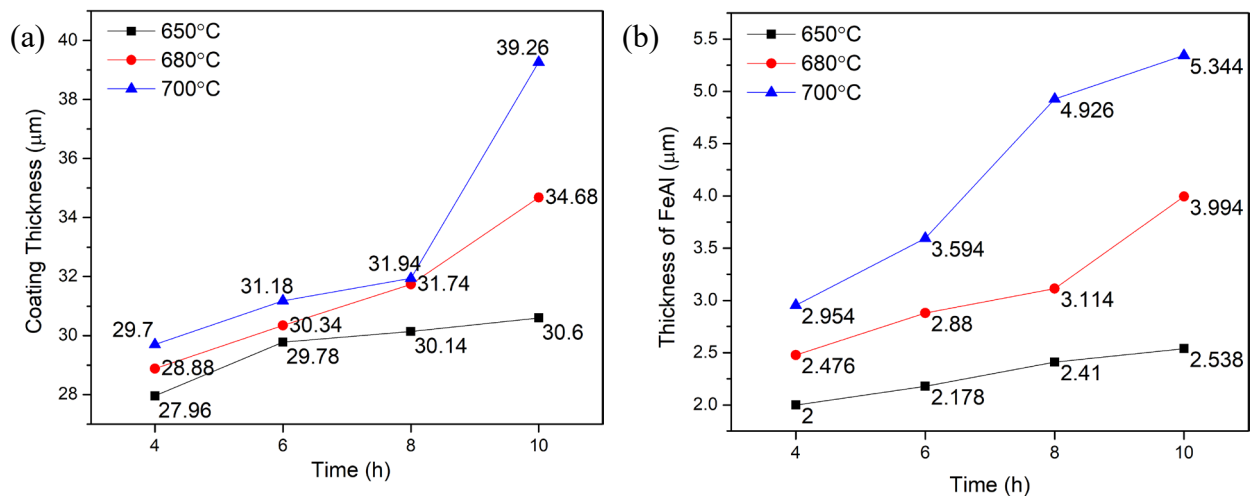


Fig. 10. Relationship between the $\ln K$ against the reciprocal of temperature ($1/T$) for (a) intermetallic aluminide layers, and (b) FeAl.

The activation energies (Q) of aluminide coating were determined based on Figure 10a. The aluminide layers have an activation energy of 214.63 kJ/mol, which contrasts with other findings: Prasanthi et al. [9] reported 184 kJ/mol., Yang et al. [8] recorded 691.95 kJ/mol, while Triani et al. [24] reported lower values of 83 kJ/mol and 34.5 kJ/mol, respectively. Additionally, the activation energy for FeAl growth was calculated from Figure 10b as 343.15 kJ/mol, higher than previously reported values as indicated in Table 2.

Table 2. The activation energy, Q (kJ/mol), for the growth of different aluminide layers (FeAl) reported in the literature and calculated in this work.

	Q FeAl (Kj/mol)
Literature	249.5[20]
	278 [25]
	250 [26]
	211 [27]
	180 [23]
This work	343.15

Variations in activation energy values are attributed to factors such as microstructure, prior surface conditions, carbon content, substrate, and other alloying elements affecting aluminide layer growth [20]. However, alloying elements have the most significant impact on activation energy. The addition of alumina in particular increased the activation energy.

4.0 Conclusions

The study demonstrates that the addition of Al_2O_3 effectively improves the aluminizing process by controlling diffusion, formation of intermetallic compound (FeAl_3 and FeAl), and enhancing hardness, while also providing the diffusion-controlled growth kinetics of the aluminide layers. From the findings, the results are summarized.

- Microstructure revealed the two-layer structures composed of Al-rich and Fe-rich intermetallic with a smooth interface between the coating and the substrate due to the addition of Al_2O_3 which reduced the activities of aluminium diffusion during aluminizing process and hence favoured the formation of FeAl .
- XRD and EDX results validated the existence of FeAl_3 and FeAl layers in the interface between the coating and 304SS.
- Microhardness measurements revealed that the FeAl_3 and FeAl had a hardness of 880- 990 HV and 610- 700 HV respectively. The sample heat treated at 650°C for 4 hours showed the lowest hardness value while the sample heat treated at 700°C for 10 hours displayed the highest hardness for FeAl_3 . A similar trend was observed for the FeAl at higher temperature and longer soaking time.
- The growth rate of the aluminide layer is a diffusion control that establishes a linear relationship between the aluminizing temperature and time, hence obeying the parabolic law. The activation energies for the growth of FeAl is 343.15 KJ/mol. The higher value of activation energy is due to the addition of alumina in the coating.

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