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# The Effectiveness of using Diffusion Coating on Steel Alloys (Steel-H25) to Resist Hot Corrosion Resulting from Sodium Chloride (NaCl) and Sodium Sulfate (Na<sub>2</sub>SO<sub>4</sub>)

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**Abstract** – This research aims to modify the surface components of Steel-H25 alloy using a surface engineering method through diffusion coating technology to obtain new alloys with high efficiency in resisting harsh environmental conditions. In this research, a harsh test was used for these produced alloys, which is the oxidation process at high temperatures and in a vapor atmosphere of a mixture of sodium chloride (NaCl) and sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) at a temperature of 900 degrees Celsius, then comparing it with the base alloy. The results showed in general that the alloys produced in this way are very efficient. While the results also showed that the single aluminum coating is highly efficient in resisting oxidation and hot corrosion testing and has provided better protection for a longer period compared to the uncoated alloy. This is due to its high adhesion to the formed Al<sub>2</sub>O<sub>3</sub> oxide skin layer as well as to the formed phases rich in aluminum, whether these phases are nickel-aluminum or iron-aluminum phases. The results also showed that the thickness of the coating and the weight gained by the alloy are subject to a parabolic relationship with time.

**Keywords** – Diffusion Coating, High-Temperature Oxidation, Hot Corrosion, Parabolic Relationship.

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## I. INTRODUCTION

Hot corrosion is a complex phenomenon from high temperatures interacting with corrosive materials such as chloride salts (NaCl). This type of corrosion is characterized by abnormally accelerated oxidation of the material, which leads to a deterioration in its mechanical properties and a significant reduction in its lifespan. Hot corrosion poses a major challenge in many industrial sectors, including aviation, power generation, and chemical processing, where components are exposed to harsh environments characterized by high temperatures and highly corrosive environments.

The hot corrosion process involves several interconnected mechanisms, as corrosive materials penetrate deep into the material, causing a series of chemical and electrochemical reactions that lead to the formation of unstable and porous oxidation layers, in addition to the formation of internal cavities and cracks that weaken the structure of the material. The dynamics of this corrosion can vary depending on the properties of the material and its microscopic structure, as well as the type and concentration of the corrosive material and the ambient temperature and pressure.

Hot corrosion can lead to an accelerated oxidation process that leads to the destruction and peeling of the protective oxidized layer on the surface of the alloy, making it vulnerable to subsequent corrosion reactions. Therefore, understanding hot corrosion mechanisms and developing corrosion-resistant materials becomes essential to ensure safe and reliable operational performance of high-performance engine components in various industrial applications [4].

## II. EXPERIMENTAL PART

The samples used, which are of different dimensions and sizes, were originally taken from the steel alloy known as (Steel-H25), and the chemical composition of the elements of this alloy is shown in Table (1). The samples were prepared and cleaned well, as the preparation process is considered one of the necessary means. Very suitable for the coating process.

Table 1. Chemical composition of the steel alloy known as (Steel-H25).

Component	C	N	Si	P	S	Cr	Mn	Fe	Ni	V	Mo
Wt. %	0.3	0.48	0.15	0.00	0.00	10.0	1.40	BAL	7.02	0.6	2.08

## III. EQUIPMENT AND MATERIALS USED

- The materials used are a mixture of different materials in specific weight ratios, including:
  1. A mixture of pure aluminum powder (30%).
  2. Ammonium chloride powder (NH<sub>4</sub>Cl) as a stimulant (4%).
  3. In addition to pure aluminum oxide (AL<sub>2</sub>O<sub>3</sub>) with a weight percentage of (66%), it works to prevent the mixture from clumping.
  4. The devices used are:
    - A tube made of heat-resistant alumina, open at one end and closed at the other.
    - Temperature control.
    - Grinding and polishing machine.
    - A sensitive electric balance with an accuracy of 0.0001gm.
    - X-ray diffraction (XRD) device.
    - Scanning electron microscope (SEM).
    - An optical microscope equipped with a camera.
    - Vacuum pump.
    - Electric ovens reach temperatures of (1150C0).
    - An image processing program for measuring thickness (Image J program).

## IV. SAMPLE PREPARATION FOR COATING

Preparing samples is one of the important things before starting the diffusion coating process. After cutting the samples, the sample preparation process took place in several stages.

1. Performing the process of grinding and polishing the samples to remove the oxides present on the surface. By using smoothing paper made of silicone with different degrees of softness, starting from (220 - 400 - 600 - 1000 - 1200) until reaching the final grade (2000) [10] [11].
2. Then the samples are washed with warm water and washing powder, then methanol to remove the stuck gr-

-easy fats, then with acetone for quick drying, then with distilled water, all to obtain a suitable surface for the coating process [12].

## V. COATING PROCESSES

The prepared samples were coated using one of the diffusion coating methods, which is the pack cementation method. This powder contains a mixture of the coating material, which consists of pure aluminum powder (30%), which is considered a source of coating, and ammonium chloride powder (4%) as an activating material that transfers aluminum from the mixture to the sample to be coated [14]. The remainder of the mixture is alumina powder ( $\text{Al}_2\text{O}_3$ ), which is used as a substance that prevents the mixture from clumping. Then the mixture is mixed for several hours, after which it is placed the sample to be coated is inside the pod, and it is tightly closed on both sides. After that, the sample is inserted into the vacuum electric furnace, where the coating process takes place at a temperature of ( $1000^\circ\text{C}$ ) and at time intervals of (6, 4, 2) hours, and after the coating process is completed. The powder is removed from the oven, and the sample is then removed from the powder and washed with warm water and washing powder, then methanol, then with acetone for quick drying, then with distilled water. Then all samples are kept in a special place until tests are conducted on them [3].

## VI. HOT CORROSION TEST

The hot corrosion test was conducted in a furnace specifically designed for this purpose using the cyclic oxidation method or thermal cycles at a temperature of ( $900^\circ\text{C}$ ), as shown in Figure (2). The test was conducted under atmospheric pressure; each cycle lasted five hours. This was done using an atmosphere of a mixture of sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) and sodium chloride ( $\text{NaCl}$ ) vapor.

## VII. TEST METHOD

A flask containing a mixture of sodium sulfate and sodium chloride with one liter of water was placed on an electric heater until it reached boiling point.

1. The vapor of the mixture was then transferred to the inside of the furnace at a temperature of  $900^\circ\text{C}$ .
2. This was done to test the behavior of uncoated samples and samples coated with different types of coatings, including the conventional aluminum coating (pack cementation).



Fig. 1. The system used in the cyclic oxidation process.

3. Weighing and measuring the dimensions of the samples: The samples were weighed before testing and their dimensions were recorded to determine the change that occurred after testing.
4. Placing the samples in a mold: The samples are placed in a mold that has cavities to hold them in place.
5. Inserting the samples into the furnace: The mold was inserted into the furnace after it reached the desired temperature (1000 C°)
6. Removing the samples from the furnace: The samples are removed from the furnace after five hours.
7. Cooling the samples: The samples are left to cool for half an hour.
8. Washing the samples: The samples are washed with water and detergent.
9. Drying the samples: The samples are dried completely.
10. Weighing the samples again: The samples are weighed again after washing and drying.
11. Repeating the process: The process is repeated cyclically for a maximum of 200 hours for testing.
12. Comparing the results: The results of the coated samples are compared with the results of the uncoated model to determine which coatings provided better protection. [14]

### VIII. MICROSCOPIC TEST

Samples were allocated for microscopic examination after the coating processes were completed. They were subjected to cold embedding, grinding, and polishing, and then a microscopic examination was performed to determine the thickness of the coating. As for the [15] process [16] and the structures that appeared as a result of the coating, it was done using the demonstration solution, Keller’s solution consists of hydrofluoric acid (1% - Hf), hydrochloric acid (1.5% - HCl), nitric acid (2.5% - HNO<sub>3</sub>), and the remainder is distilled water (95%), and to reduce hot corrosion and oxidation, the method used in our research is simple aluminized coating, or what is known as diffusion coating [17] [18].

Table 2. Components and ratios of Keller demonstration solution.

Components	HF	HNO <sub>3</sub>	HCl	Pure Water
Percentage	1%	2.5%	1.5%	95%

### IX. RESULTS AND DISCUSSION

Table (1) includes the results obtained from coating operations at this degree, as it includes the rates of weight gained and resulting from coating with aluminum at this degree (1000 °C) for the three coating times. Figures (2) and (3) show the presence of a parabolic relationship when drawing rates. The weight gained as a function of time as well as the thickness of the coating sequentially, which indicates that the diffusion process is what governs the coating process [10].

Table 3. Details of weight gain rates and thickness of coating produced in one stage (Coating only) at 1000 C° for steel alloy (Steel-H25).

Temp. C°	Time of Coating (h)	Av. Thickness of Coating (μm)				Av. Weight Gain (gm/cm <sup>2</sup> )
		Outer Layer	Inter Diffusion Zone	Inner Layer	Total	
1000	2	56.032	23.011	72.571	151.617	0.028756

Temp. C <sup>0</sup>	Time of Coating (h)	Av. Thickness of Coating ( $\mu m$ )				Av. Weight Gain (gm/cm <sup>2</sup> )
		Outer Layer	Inter Diffusion Zone	Inner Layer	Total	
	4	63.021	15.213	88.033	163.267	0.056534
	6	64.013	13.310	104.410	181.733	0.061123

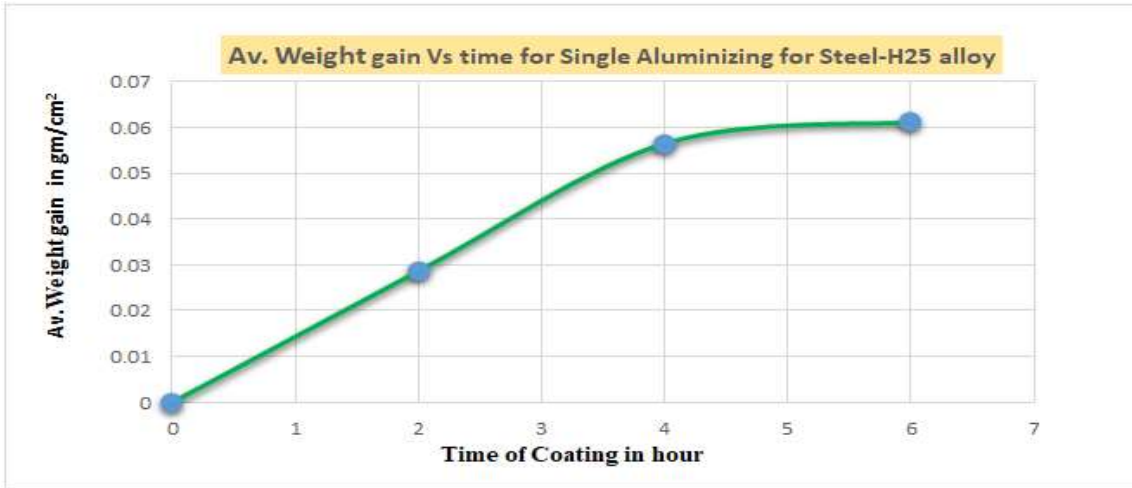


Fig. 2. The gained weight of steel alloy (Steel-H25) aluminizing at a temperature of (1000 C<sup>0</sup>) as a function of time.

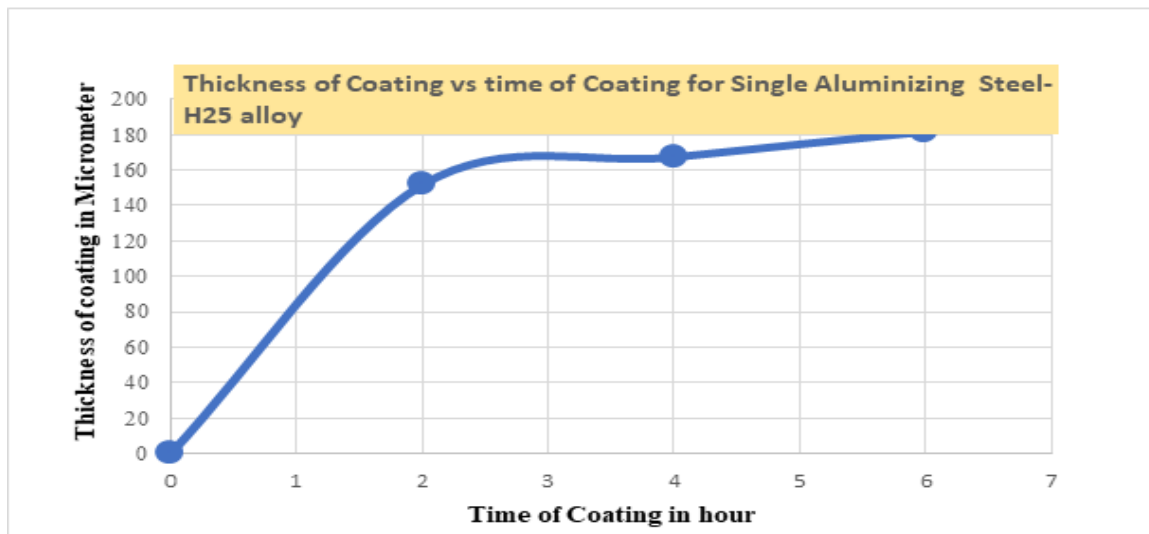


Fig. 3. Average coating thickness resulting from the single aluminum coating of steel alloy (Steel-H25) at temperature (1000C<sup>0</sup>) as a function of time.

Through the results bits of microscopic examination of cross-sections of samples coated for periods (6-4-2) hours, we have revealed several things:

- The resulting coating after the three time periods consists of several layers, and the thickness of the total coating layer is approximately 151.617  $\mu m$ , 163.267  $\mu m$  and 181.733  $\mu m$  for the periods (6-4-2) hours, respectively.
- It is also noted that the microstructure of the coatings produced over the three time periods is similar. It is also characterized by the presence of more than one phase in the coating layer, and the outer coating layer consists of phases rich in aluminum.

- It is also characterized by the presence of carbide deposits of different colors, including dark brown, light brown, and light yellow, which is affected by the type of rendering solution used.
- Because the alloy used in our research is iron-based, coating it with aluminum leads to the formation of one of the phases of the intermediate compounds (iron-aluminum) and one of the phases (nickel-aluminum). This was demonstrated by the results of X-ray diffraction (XRD) analysis, despite the many complications. In terms of the number of resulting phases, it confirmed the formation of the aluminum-rich phase (FeAl) and (Ni-Al) mainly in the three resulting coatings after the three time periods (6-4-2) hours had passed.
- The most important point from what was mentioned above is that the basic phase formed does not only change with increasing coating time, but also changes with changing temperature, coating powder concentration, or base alloy composition, and this has been confirmed by recent research [11] [17].

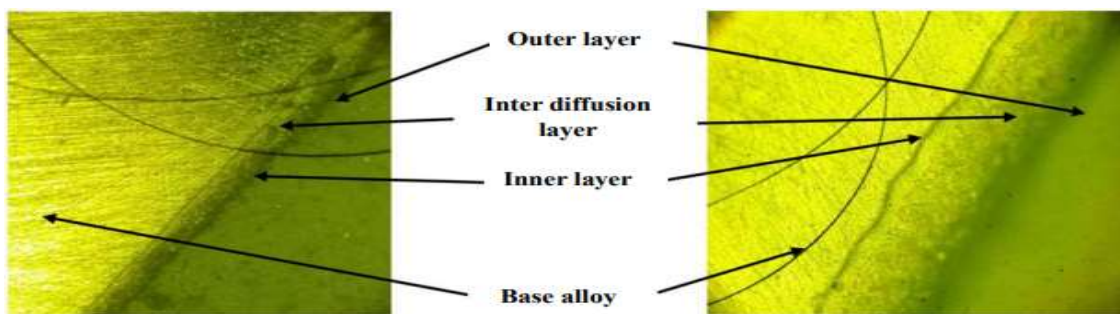


Fig. 4. Microscopic structure of samples coated in one stage (aluminizing only) at a temperature of (1000 °C) and for periods (2) and (6) for the steel alloy (Steel-H25).

Table (4) shows the results of the X-ray diffraction analysis of the sample coated for 6 hours, as it was shown from the diffraction pattern, Figure (5), the appearance of peaks for intermediate compounds belonging to the iron-aluminum phases, such as (FeAl), which appeared primarily, and (Fe<sub>4</sub>Al<sub>13</sub>) with strong peaks. However, several weak peaks are indicating the presence of what is believed to be the (AlCr) phase. The analysis also confirmed the presence of peaks for intermediate compounds dating back to (aluminum-nickel) such as (NiAl) and (NiAl<sub>3</sub>), and that such phases are possible to form at this temperature.

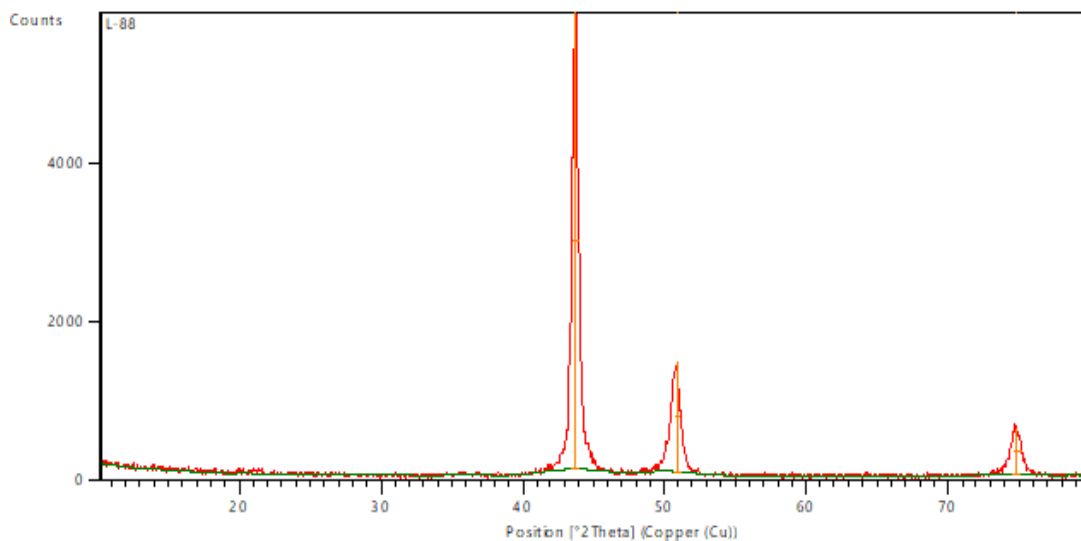


Fig. 5. X-ray diffraction (XRD) pattern of the sample coated in only one stage (Single Aluminizing at a temperature of 1000 °C for 6 hours for the alloy (Steel-H25)).

Table 4. X-ray examination results (XRD) for the sample coated in one stage (Single Aluminizing at a temperature of 1000 °C for 6 hours for the alloy (Steel-H25).

Al <sub>2</sub> Cr <sub>3</sub>	NiAl <sub>3</sub>	NiAl	Fe <sub>4</sub> Al <sub>13</sub>	FeAl	Phase Type
Vw	W	S	Vs	Vs	Relativity intensity
S = strong, W = weak, Vw = very weak, Vs = very strong					

- The results of the analysis were shown by the scanning electronic analysis device (SEM-EDS) of the coating layer at multiple magnification powers (100 kx). As shown in Figure (6), the clarity of the spread of aluminum on the surface of the alloy, is confirmed by Figure (7), which represents the energy dispersion spectroscopy (EDS), as we notice that there are many atoms for the elements, including aluminum, iron, and chromium, and the highest intensity of these peaks is the aluminum element, which will represent the element. The basis for the coating layer, is what appeared in the data in Table (5) for the quantitative and qualitative analysis using the scanning electronic device (SEM-EDS).
- It is worth noting that the percentage of aluminum was equal to 23.52 wt.%, while the percentage of iron was 27.32%, and this in turn supports the analysis of the 5.83%.

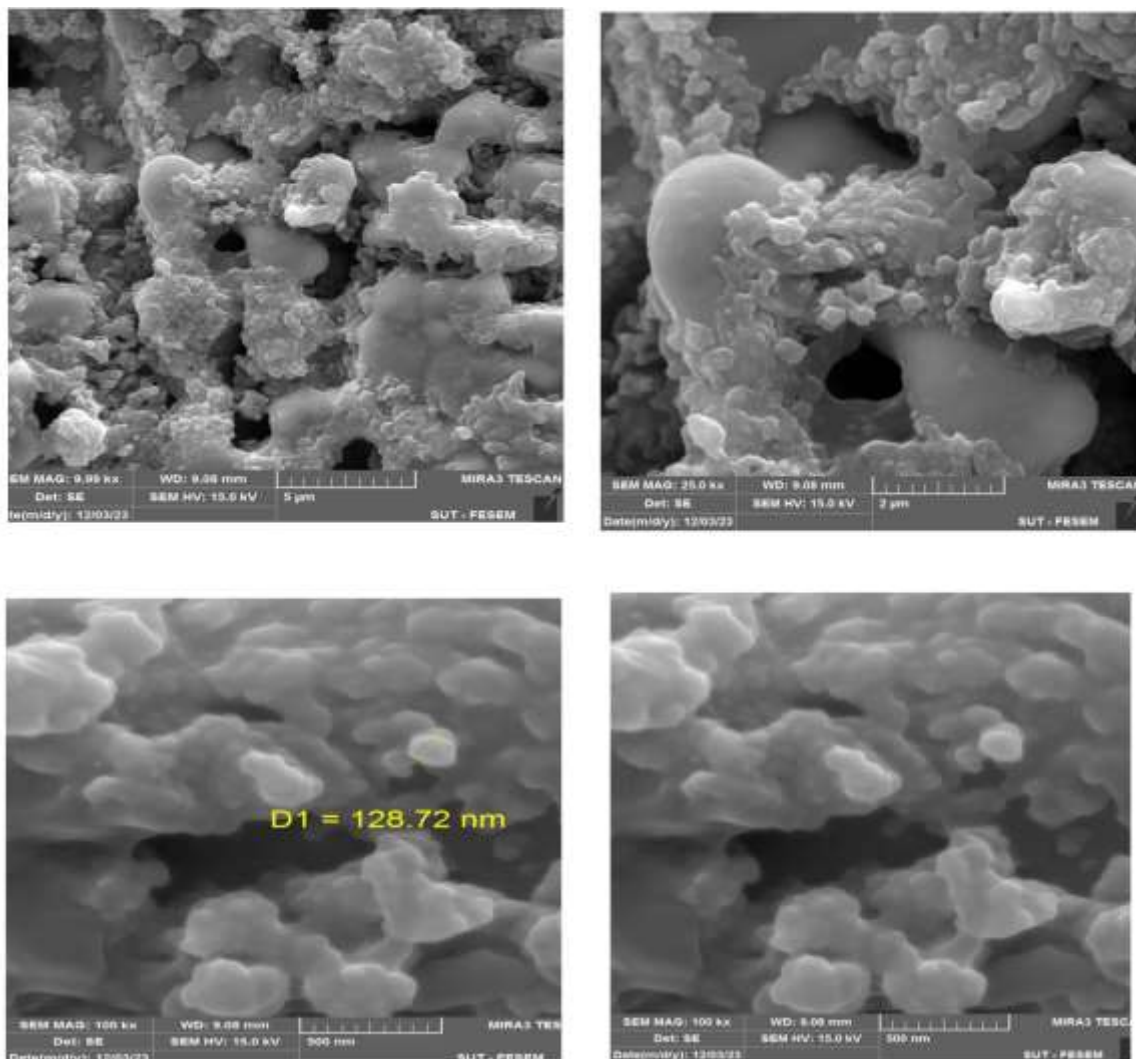


Fig. 6. Scanning electron microscopic structure (SEM) of steel alloy (Steel-H25) aluminized for (6) hours at a temperature of (1000 °C) and with different magnification powers.

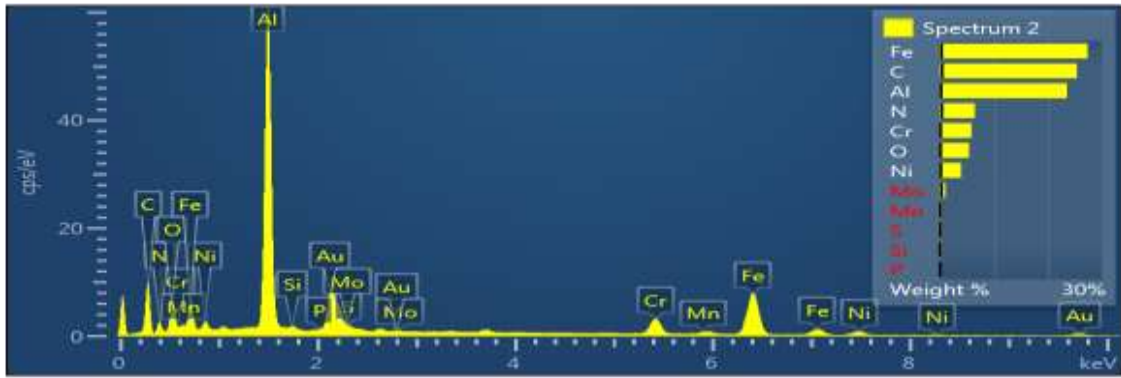


Fig. 7. Scheme of quantitative and qualitative examination with the scanning electronic device (SEM) of steel alloy (Steel-H25) aluminized for a period of (6 hours) at a temperature of (1000 °C).

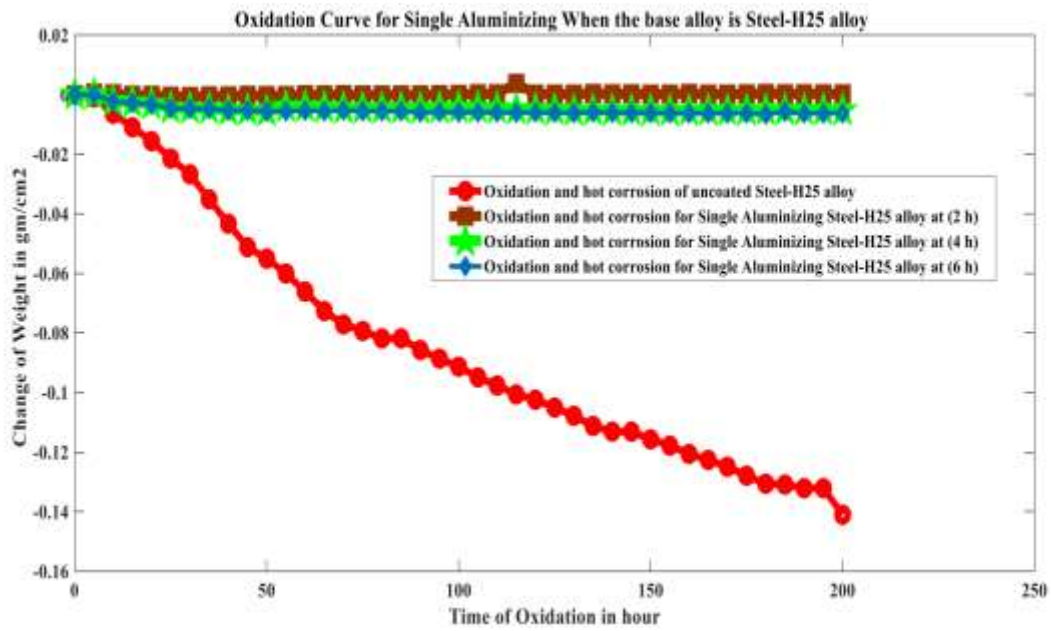


Fig. 8. The oxidation and hot corrosion curve of steel alloy (Steel-H25) aluminized for some time (2-4-6) hours at a temperature of (1000 °C) and exposed to a mixture of salt vapors (NaCl) and (Na<sub>2</sub>SO<sub>4</sub>) at a temperature of 900 °C and for 200 hr.

Table 5. Details of the weight and quality percentages using the scanning electronic device (SEM-EDS) for the steel alloy (Steel-H25) aluminized for (6 hours) at a temperature of (1000 °C).

SEM-EDS-Data for Single Aluminizing for Steel-H25 alloy				
Element	Line Type	Apparent Concentration	Wt. %	Atomic %
C	K series	1.29	25.38	47.01
N	K series	1.84	6.48	10.29
O	K series	1.35	5.37	7.47
Al	K series	7.00	23.52	19.40
Si	K series	0.09	0.33	0.26
P	K series	0.04	0.09	0.07
S	K series	0.10	0.33	0.23
Cr	K series	1.72	5.83	2.50



<b>SEM-EDS-Data for Single Aluminizing for Steel-H25 alloy</b>				
<b>Element</b>	<b>Line Type</b>	<b>Apparent Concentration</b>	<b>Wt. %</b>	<b>Atomic %</b>
Mn	K series	0.10	0.37	0.15
Fe	K series	7.50	27.32	10.89
Ni	K series	1.06	3.90	1.48
Mo	L series	0.23	1.05	0.24
Total:			100.00	100.00

### X. CONCLUSION

Figure (8) shows the mechanism of hot corrosion oxidation of the single aluminum coating (Steel-H25) for the periods (2-4-6 hours). From observing the weight change chart with time, we notice several things:

- The weight remains stable and does not decrease very clearly for most periods, as seen in the figure. However, the period (2hr) is the most stable, and then comes the periods (4hr, 6hr), respectively, compared to the uncoated alloy.
- Tests showed that the coating layer over the period, especially the period (2 hours), has good efficiency in resisting hot corrosion, and this, in turn, indicates that the protective oxide layer ( $\theta$  -Al<sub>2</sub>O<sub>3</sub>) does not fall off and that it has good adhesion.
- As for the other hours of coating, the weight decreased slightly with the thermal cycles and until the end of exposure compared to the uncoated alloy, which collapsed from the first hours of the thermal cycles.
- The reason for the slight decrease in weight is due to the formation of unstable oxide ( $\theta$  -Al<sub>2</sub>O<sub>3</sub>), which was formed during transition oxidation before the formation of the protective oxide shell ( $\alpha$  -Al<sub>2</sub>O<sub>3</sub>), which was formed during thermal cycles.

The reason for the collapse of the uncoated alloy is due to several things, including:

- The fact alloy contains a percentage (10wt.%) of the element chromium, which is added to the alloy to form the protective oxide crust (Cr<sub>2</sub>O<sub>3</sub>) during its oxidation. However, the examination with the (XRD) device did not show the presence of this protective oxide crust because it has the property of evaporating and converting to the oxide CrO<sub>3</sub> in degrees. Higher than 900, as researchers [13], indicated, indicating the formation of a protective oxide crust, Cr<sub>2</sub>O<sub>3</sub>. Which can react with volatile compounds such as (CrO<sub>2</sub>Cl<sub>2</sub>) in the presence of salt vapor NaCl, which leads to the formation of the volatile compound Na<sub>2</sub>CrO<sub>4</sub>, and volatile metal chlorides such as CrCl<sub>3</sub>, NiCl<sub>2</sub>, on the one hand, and the other hand, the presence of salt vapor (Na<sub>2</sub>SO<sub>4</sub>), which contributes to the formation of gases SO<sub>2</sub>, SO<sub>3</sub>., which in turn generates very strong steam pressure, which causes some cracks, which participate in the cracking and cracking of these protective shells.

In addition, corrosive media containing some sulfide salts or sulfide compounds of alloying elements such as FeS, Fe<sub>3</sub>C<sub>2</sub>, NiS, and NiCl<sub>2</sub> below the crust, all of which are characterized by their low melting point below 1050 degrees Celsius as well as their low plasticity, will contribute effectively to increasing the probability Falling off protective scales.

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