Effect of Pack Cementation Coating on Hot Corrosion Resistance of Low Alloy Steel

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Abstract

Two technique of coatings were performed by applying pack cementation technology, the first one was aluminizing diffusion coated low alloy steel substrate whereas the second one was single step simultaneous Co-deposition silicon- aluminum diffusion coated low alloy steel substrate. The pack mixture used for producing both coating systems in pack cementation process was composed of (30% Al, 2% NH₄Cl and the filler 68% Al_2O_3 , (30% Al, 5% Si, 2% NH4Cl and 63% Al_2O_3) respectively. The pack temperature was 1000°C and the diffusion time was 3 hr. XRD exhibits the phases formed on both coated systems are Fe₂Al₅, FeAl₃ and FeSi. Uncoated low alloy steel substrate and the both two coating systems were subjected to exposure environment deposit salts mixture of $(50\% \text{Na}_2\text{SO}_4 \text{ and } 50\% \text{NaCl})$ with weight $(2\text{mg}/\text{cm}^2)$, using cyclic oxidation method in the experiments at temperature of 750°C for 100hr each cycle of 5hr. It was found that the oxidation kinetic for uncoated low alloy steel will give evidence that the oxides formed on this alloy are non - protective. The XRD analysis exhibited the phases are Fe_2O_3 , Fe_3O_4 . It was found they followed linear rate law, and spalling was observed. After these hot corrosion tests were undertaken, It was found that the oxidation kinetics for both coated systems in environment consist of salt mixture (50% NaCl +50% Na₂SO₄) followed parabolic law (protective oxidation behavior). XRD exhibits the oxides formed on both coated systems are Al_2O_3 , SiO_2 .

Keywords: hot corrosion, pack cementation, low alloy steel, XRD

تأثير الطلاء بالسمتنة على مقاومة التأكل الحار لفولاذ منخفض السبائكية

الخلاصة

تم تنفيذ نظامين طلاء الأول طلأء الألمنة الأنتشاري والنظام الثاني طلأء الألمنة - السلكنة الأنتشلري في خطوة واحدة وهو عبارة عن ترسيب الألمنيوم والسيلكون على سطح الفولاذ في زمن واحد. وتم أستعمال خليط مسحوق السمنتة من المواد (Al, 30% Al, 20% Al, 20% Al, 20% Al, 20%) و (Al % منعمال خليط مسحوق السمنتة من المواد (Al, 30% Al, 30% Al, 20%) و (Al % منعمال خليط مسحوق السمنتة من المواد (Al, 30% Al, 30% Al, 20% Al,

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المطلية ذات سلوك كاينتيكي يتبع القانون الخطي ويحصل نقشر في الأكاسيد اما أختبارات التأكل الحار على العينات المطلية في كلا نظامي الطلاء فقد أظهرت النتائج ان السلوك الكاينتيكي العام يخضع الى قانون القطع المكافئ (سلوك الأكسدة الواقية) وأن الأكاسيد المتكونة هي Al₂O₃, SiO

Introduction

High temperatures are encountered in a number of industrial processes such as generation of electricity. A very large part of these heat engines, including much of the elevated temperature sections, are made of alloys, which have not been optimized at all for oxidation or high temperature corrosion resistance. All the plain carbon and low alloy steels are oxidizing at what one might call "natural" rate. their Without exception, they are oxidizing at temperature below that at which wustite, FeO, is stable for plain carbon steels this is 570°C, The oxide formed in air has an inner layer of magnetite, Fe₂O₃, Generally, the thickness of the magnetite layer is considerably larger than that of the hematite.[1].

Hot Corrosion / Sulfidation is sometimes defined as an accelerated corrosion because of the presence salt contaminants such as NaCl, Na₂SO₄ and V_2O_5 that combine to form molten deposits which damage the protective surface oxides [2]. Hot corrosion is commonly accompanied by the formation of a porous and non - protective oxide scale which has been attributed mostly to the condensation of Na₂SO₄ that attacks the protective oxide scale, besides; NaCl has been indicated as a corrodent in high temperature environments. The presence of NaCl ingested in the intake air together with Na₂SO₄ leads to more sever corrosion of materials. The mixtures with different compositions are all completely melted below $900^{\circ}C$, indicating that the Na_2SO_4 – fluxing mechanism. Therefore; the existence of a higher amount of metal chlorides

or sulphides would induce severe hot corrosion [3]. During the combustion of the fuel, vanadium reacts with oxygen to form an oxide V_2O_5 (melting point of vanadium $670^{\circ}C$ thus V_2O_5 is a liquid state at gas turbine operating temperature. These compounds known as ash deposit on the surface of material and induce accelerated oxidation (hot corrosion) in energy generation system. Corrosion occurs when these molten compounds dissolve the protective oxide layers, which naturally form on materials during gas turbine / boiler operation, further, Vanadate compounds oxidation are good catalysts and allow low oxygen and other gases in the combustion atmosphere to diffuse rapidly to the metal surface and cause further oxidation. As soon as the metal is oxidized, the cycle starts over again and high corrosion rate occurs further [4, 5].

Robert A. Rapp, P.I., 1995 [6] reported that the carbon steels or low allov steels have served as the material workhouse the in construction of utility boilers, heat exchangers and other power generation installations. However, these steels suffer from attacks in the of oxidation. sulfidation. forms resulting from the presence of sulfur in low grade fuel, shortens the service life. Appropriate coatings, applied to carbon or low alloys steels, have long been sought as a means to combat these problems. The best corrosion protection is provided by an enrichment at the surface of two or more beneficial alloying elements. So to modernize and improve the practice of halide - activated pack cementation coatings, a means was needed to codeposit two or more elements simultaneously in the proper concentration.

Shreir L.L, 2000 [7] report that the diffusion coatings are formed as a result of interaction of two distinct processes, the solute metal is brought into contact with the surface of the solvent, and this is followed by diffusion proper which consists in the gradual absorption of the solute into the lattice of the solvent. Whatever method is used to provide an adequate supply of diffusing metal, the diffusion mechanism remains identical for any given solute / solvent system, and it is appropriate to discuss the diffusion aspect in the first place. In this process, a halide of the solute metal is passed in vapor form over the surface of the metal to be coated, which is heated to a temperature at which diffusion can take place. Temperature of 500 – 1300°C or more can be used, depending on the particular system considered. Generally, filler atmospheres are provided to carry the halide vapor, these atmospheres usually reduce the gases such as hydrogen, ammonia or inert gases (helium, argon).

The aim of this study is to deposit Al and Si simultaneously on the surface of low alloy steel using a single step pack cementation. Study cyclic hot corrosion of low alloy steel aluminized diffusion coating with and without silicon in salt mixture (50% wt Na₂SO₄ + 50% wt NaCl) at temperature of 750°C.

Experimental Work

program plan The of this experimental work will determine the oxidation behavior of aluminizing diffusion coated and silicon modified aluminide diffusion coated of low subjected high alloy steel to temperature at environment contain mixture salt of (50% Na₂SO₄ +50% NaCl) which are approximately

similar to the environment of steam turbine system (boiler tube ,super heater tubes) and gas turbine system (blades ,burners) . As a material used by many industrial installation, the atmosphere resembling that encountered in those installations as flue gases containing is created in this experimental work using sold salts.

Metal of substrate:

The material had been supplied by Baghdad South Power Generation Plant as a tube with the diameter of 60 mm. The chemical composition analysis was done at Material Engineering Science Department– University of Technology. The results are shown in the **Table (1)**.

Pack cementation powder:

The pack mixture used for silicon – modified aluminide diffusion coating consist of ferroaluminum powder of an aluminum source, silicon, NH_4Cl as an activator and the rest was alumina powder of the inert filler. The pack was mixed for 3hr by mechanical mixer. The pack mixture used in the same as in that silicon modified aluminide diffusion coating pack, but that was of silicon was not used and replaced by pack alumina filler.

Specimens Preparation:

The samples were manufactured by milling machine into the shape of dimensions 15 * 17 * 3mm with a total surface area of 5890mm². All the surfaces of the specimen were wet ground with silicon carbide papers SiC, after that the polishing operation was done. These samples were cleaned with water and degreased with acetone, then ultrasonically cleaned for 60 minutes in liquid ethanol as a medium, after that samples were dried and stored in desiccators before being weighted in precise microbalance. the The ultrasonic operation can also act as a stress relieve. The dimensions of each sample were precisely measured using precise micrometer. The each sample was weighted by METTIER MICRO BALANCE

Pack cementation facilities:

The Figure (1) illustrate the pack cementation system and show the facilities were essential for carry out the process are clear in the figure and listed below:

1) Inner retort, 2) Outer retort, 3) Thermocouple type K (maximum temperature 1200°C) 4) Argon gas supply unit

5) Electrical cylindrical / tube furnace. **Hot corrosion facilities:**

1. Furnace of temperature more than 1100° C.

2. Simple tools for mixing and spraying the salt mixture

3. Thermocouples type K

Coating Technique:

The program of this study attempts to determine how high temperature and environment consist of salt mixture (50% Na₂SO₄+ 50% NaCl), affect the oxidation behavior of silicon modified aluminied diffusion coated low alloy steel, as well as tries to approach the optimum results of diffusion coating. Meanwhile one should take into consideration the pack powder can not be sufficiently achieved unless a suitable mixture ratio of activators should be added to the pack powder.

The low alloy steel samples with pack powder were placed inside the stainless steel cylinder of 5 cm in diameter and 8 cm height which can be considered as inner retort, this inner retort was placed in stainless cylinder steel which can be considered as outer retort, this combined system was placed in an electrical cylindrical/tube furnace and that was connected to argon gas supply unit, in order to prevent the process from changing to oxidation process, because oxidation may occur at elevated temperature. Then the

heating cycle can be started. Figure (1) illustrates the pack cementation system. The heating cycle for one step pack cementation process was started for diffusion when the argon gas was flowing to the retort group to keep argon atmosphere during the entire time of diffusion coating process. The time of the period of furnace cooling to room temperature clear in Figure (2). When reaching room temperature the group of retorts removed from furnace, the were samples were then removed from the pack mixture and ultrasonically cleaned for 60 min in ethanol media to remove the pack material and weighted. The weight of samples was recorded after coating since it was very important for identifying the element Al, Si to see if they diffused inside the surface to create coating. Each sample was mounted in cold setting epoxy. The samples were treated for preparation for the examination of the microstructures and coating morphology by (LOM) type (Carlziess Jena DDR) which was provided with digital camera.

Hot Corrosion

Cyclic oxidation tests at high temperatures and in environment(50%NaCl + 50%Na₂SO₄) were made to study the thermal shock oxidation resistance of low alloy steel with and without silicon - modified aluminide diffusion coatings. Thermocouple type k was inserted inside the tube furnace in the position located near the samples, the furnace temperature was controlled within the $\pm 3^{\circ}C$ during the cyclic oxidation. The thermocouple was calibrated by measuring a known melting point or boiling point of certain material such as aluminum or water.

Coated and uncoated samples were heated in the tube furnace at a test temperature in desired environment and then the samples were removed from the furnace in order to conduct the room temperature tests then ultrasonically cleaned in ethanol to release the spelled oxide and then they were weighed every 5 hr. The weight change per unit surface area was determined for the evaluation of oxidation resistance.

The hot corrosion experiments were performed on the coated samples of low alloy steel with coating systems Si-modified aluminide and aluminizing, as well as to the uncoated samples of low alloy steel. The procedure of this experimental work (Hot corrosion) was done according to Wang et al procedure [8]. The samples were measured and recorded its weight. saturated salt solution was prepared by adding the mixture of (50%NaCl +50%Na₂SO₄) to the distilled water until saturated solution was obtained, then the air gun spray was filled with the solution .The samples were weighted after that samples were heated the to temperature range of (100°C-120°C) for a short period to evaporate the water, the solution was sprayed on the heated samples. The coat of fine particles was formed on the samples surface because the water evaporated. This operation was repeated until the salt particles were deposited upon samples to approximately 2 mg/cm², the heat cycle was started at 750°C in static air for 100 hr, each cycle was for 5hr at programmable tube furnace, after oxidation was completed the samples were cleaned ultrasonically in ethanol and then weighted.

Results and Discussion Coating systems

The current study attempts to conduct and determine the effect of elevated temperatures and the environment (salt mixture) on the behavior of low alloy steel uncoated and coated relevant to that components service in a hot sections of power plant generation and petroleum refinery plant such as burners, combustors, discs since these components are exposed to failure by oxidation and hot corrosion. This study will review and discuss all the results, information, data, obtained from many tasks carried out by program plan of this investigation described in the previous papers covering oxidation experiments and hot corrosion by sulfites and chlorides (50%NaCl +50% Na_2SO_4) experiments. In addition to that will review the role of silicon when combined with aluminum in increasing the hot corrosion resistance. The uncoated samples of low alloy steel and Si-modified aluminide diffusion coated low alloy steel and aluminizing diffusion coated low alloy steel were studied in cyclic oxidized environments (50%NaCl + 50%Na₂SO₄) at $750^{\circ}C$, for 100hr.

This work comprises the coating system of aluminizing with and without silicon which simultaneously diffused for substrates of low alloy steel. The one step pack cementation was carried out in order to increase capabilities of low alloy steel at high temperature for components of power generation. **Figure (3)** shows the diffractograms from the surface of uncoated low alloy steels.

Microstructure of Coating Systems

The samples of low alloy steel were coated by simultaneous aluminizing and optimum siliconizing at condition of coating at temperature of $(1000^{\circ}C)$ for period (3hr), this agrees with condition applied by Akuezue and whittle [9]. The packs were cooled room to temperature under the same atmosphere, the samples were

removed from the pack and ultrasonically cleaned to remove any loosely entrapped pack material on the surface. Each sample was cut with a saw. All the samples were etched for approximately 30 sec in an etchant mixture of alcohol and nitric acid. The microstructure was observed after being etched. Samples were examined by (LOM) and (X R D) to characterize the coated layers of the coated samples.

Figure (4) shows (LOM) of Simodified aluminide diffusion coated low alloy substrates deposited in 3hr as packing period, coating layer consisted of the intermetallic compounds, FeAl, Fe₂Al₅, Fe₃Si located at the outer zone which was indicated by XRD analysis as shown in Figure (5). This characteristics attributed to the aluminum and silicon inward diffusion from pack powder mixture to the surface of substrate and outward diffusion of Fe from substrate to the coating layer.

Figure (6) shows (LOM) of aluminizing diffusion coated low alloy substrates deposited in 3 hr as a packing period. coating layer of consisted the intermetallic compounds FeAl, Fe₂Al₅ located at the outer zone which were indicated by XRD analysis as shown in Figure (7). Table (2) shows the intermetallic compounds for each coating system.

The phenomenon of hot corrosion or sulfidation can be observed ordinarily whenever the temperature rises to high level provided sulfur, vanadium, chlorine and sodium exist as oxide species in the environments which become a molten intermetallic because the temperature is high and the molten intermetallic will deposit on the surface of substrate leading to rapid attack for the substrate to cause the corrosion. It is known there are two types of hot corrosion [10]. The type one is HTHC (high temperature hot corrosion) which is observed in the temperature range ($820^{\circ}C - 920$ $^{\circ}C$), the type two is LTHC (low temp hot corrosion) observed in temperature range ($590^{\circ}C - 820^{\circ}C$) which is applied in this study.

In the current study the salts mixture created from 50% NaCl and $50\% Na_2 SO_4$ was used for low alloy steel oxidation in the laboratory air at 750°C, the attack was clearly severe on the surface of substrate. Figure(8) shows the oxidation kinetics of uncoated low alloy steel oxidized with (50%NaCl $+50\% Na_2 SO_4$) deposited at 750°C for 100hr at 5hr in cyclic plotted as weight change vs. time. The figure exhibits weight loss rate and the kinetic behavior does not follow the parabolic law but will follow the linear law. The values of (kL)(n) and are respectively.Figure(9) shows the (LOM) images for the cross section of uncoated low alloy steel subjected to cyclic oxidation salt (50%) in $NaCl+50\% Na_2SO_4$) deposited at temperature of $750^{\circ}C$ for 100hr each cycle 5hr. (LOM) images of cross section of samples exhibit voids and pores with high density in the scale. The results obtained by this study are in coincidence with these of the researcher Gurrappa I. [11].

Hot Corrosion of Coated Systems

The phenomenon of hot corrosion or sulfidation can be observed ordinarliy whenever the temperature rises to a high level provided sulphur, vandum, chlorine and sodium exist as oxide species in the environments which become a molten intermetallic compound because of the high compound will deposit on the surface of substrate leading to rapid attack for the substrate and cause the hot corrosion.

oxidation kinetics The of coating systems (Si-aluminide diffusion coated low alloy steel) and (aluminizing diffusion coated low alloy steel) diffusion coatings which are coated on low alloy steel after being exposed to cyclic oxidation in salt (50%) $NaCl+50\% Na_2SO_4$) deposits at $750^{\circ}C$ for 100hr each 5hr cycle are plotted as weight change vs. time as shown in Figure (10) & (11).The results $K_P = 6.46 \times 10^{-3} \text{ mg}^2 \text{ cm}^{-4} \text{ s}^{-3}$ ¹, n = 0.44 for Si-modified aluminide coated system and $K_P = 1.76*10^{-2}$ $mg^2.cm^{-4}.s^{-1}$ n = 0.44 for aluminide

mg².cm⁻⁴.s⁻¹ n = 0.44 for aluminide coated system show the oxidation kinetics of both those coated systems follows parabolic rate law, while The results K_L = 8.1* 10⁻⁵ mg².cm⁻⁴.s⁻¹, n=1 for uncoated low alloy steel show the oxidation kinetics follows a linear rate under the same test conditions.

On the other hand it is seen that Simodified aluminide coated system is one order of magnitude lower than that of aluminide coated system under the same test conditions after 100hr of the oxidation test at 750°C, both coated systems exhibit thick porous scale present at their outer surface. Underneath this scale the remainder of coating alloy has been affected by the hot corrosion attack. This is attributed to the nature of the sulfide scale which is non adherent, non protective and high porous whereas the oxide scale is compact and adherent to the substrate. with 2-Intermetallic compounds constitution of the coating systems

temperature molten intermetallic

increasing time and temperature, S ions penetrate the alumina and reach the alloy surface and react with Fe and form their sulfides Figure (12) & (13) shows the presence of intermetallic compounds (SiO₂, FeAlO₃, Al₂O₃) and (FeAlO₃, Al₂O₃) respectively after cyclic oxidation at 750°C for 100 hr.

Figure (14) & (15) shows images of (LOM) of cross section of coating systems for low alloy steel samples under cyclic oxidized in (50% NaCl+50%Na₂SO₄) deposits at $750^{\circ}C$ for 100hr, each of 5hr cycle, The images exhibit subsurface oxidation along grain boundaries and within grains. This is attributed to the increasing penetration rate with increasing temperature for oxygen concentration due to the pores and microcrackes in the scale .The presence of intermetalic compounds is shown in substrate and near the surface layers. This is due to occurrence of internal corrosion products, the cracks and pores caused by thermal stresses as a result of difference in thermal expansion coefficient between the substrate and oxide layer.

These are formed along grain boundaries and corrosion products increase as oxidation time increases. This result is agreement with results obtained by Pierre R. Roberge [12] Since the intermetallic compounds (SiO₂, Al₂O₃) and (Al₂O₃) formed as indicated in Figures (14) & (15) do not cover the whole matrix.

Conclusions

[1]-Alloys coated with both Si and Al have positive effects on the oxidation resistance.

(with and with out) Si - modified

aluminide were found to be FeAl, , FeAl₃, Fe₂Al₅ as indicated by XRD.

3- Both coated systems (Si – modified aluminide and aluminizing diffusion coating) show higher oxidation resistance than that of uncoated low alloy steel under the same conditions.

4- Oxidation rate constants (Kp) for both coated systems oxidized with $(50\% \text{ NaCl} + 50\% \text{ Na}_2\text{SO}_4)$ are higher than that of uncoated low alloy steel

5- Hot corrosion for both coated systems (Si – modified aluminide and aluminizing diffusion coating) exhibit thick porous scale at surface and severe attack underneath this scale.

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_	Table (1) chemical composition analyses of low alloys steel in wt%										
Г	Element	С	Mn	Si	Cr	Р	S	Cu	Δ1	Fe	
	Licinent	C	17111	51	CI	-	5	Cu	111	It	
Г	Wt%	0.051	1.412	0.639	0.36	0.03	0.022	0.39	0.04	Rem	
	VV C / O	0.051	1.712	0.007	0.50	0.05	0.022	0.57	0.04	Rem	

Table (1) chemical composition analyses of low alloys steel in wt%

Table (2) Intermetallics formed by coated systems

Tuble (2) Intermetantes formea by courea bystems				
Coated Systems	Intermetallics Formed			
Si-Aluminide Diffusion	FeAl, Fe ₂ Al ₅ , Fe ₃ Si			
Coated low Alloy Steel .				
Aluminizing Diffusion Coated	FeAl, Fe ₂ Al ₅			
low Alloy Steel .				



Figure(1) Pack cementation system



Figure (2) The heating cycle for one step pack cementation process



Figure (3) Diffractograms from surface of uncoated low alloy steel



Figure (4) LOM image for si-modified aluminide diffusion coating of low alloy steel substrate (cross section view).



Figure (5) diffractograms from surface of Si- aluminide coated low

alloy steel



Figure (6) LOM image for aluminizing diffusion coating low steel substrate (cross section view)



Figure (7) Diffractograms from surface of aluminzing diffusion coated low alloy



Figure (8) The relationship of weight change with time of uncoated low alloy steel in $(50\% \text{ NaCl} + 50\% \text{ Na}_2\text{SO}_4)$ at 750 °C for 100 hr each cycle of 5hr



Figure (9) LOM image for un-coated low alloy steel substrate cyclic oxidation in ($50\%Na_2SO_4 + 50\%NaCl$) at 750 °C for 100 hr each cycle of 5hr



Figure (10) Parabolic fitted of weight change vs time plot for both diffusion coating systems Si-modified aluminide coated low alloy steel and aluminizing coated low alloy steel, cyclic oxidized in(50% NaCl + 50% Na₂SO₄) at temperature of 750 °C for 100hr each cycle of 5hr



Figure (11) Linear fitted results of weight change vs square root time plot for both diffusion coating systems Si-modified aluminide coated low alloy steel and aluminizing coated low alloy steel, cyclic oxidized in(50% NaCl + 50% Na₂SO₄) at temperature of 750 °C for 100hr each cycle of 5hr



Figure(12) diffractograms from surface of Si- aluminide coated low alloy steel cyclic oxidized in salt (50% NaCl +50 % Na2SO4) at 750 °C, for 100hr each 5hr



Figure (13) Diffractograms from surface of Aluminzing diffusion coated low alloy steel cyclic oxidized in salt (50% NaCl +50% Na₂SO₄) at 750°C, for 100hr each cycle 5 hr



Figure (14) LOM image for Si-modified aluminide diffusion coating of low alloy steel substrate cyclic oxidation in(50%Na₂SO₄ +50% NaCl) at 750 °C for 100 hr each cycle of 5hr (cross section view).

Figure (15) LOM image for aluminizing diffusion coating of low alloy steel substrate cyclic oxidation in (50%Na₂SO₄+50% NaCl) at 750°C for 100 hr each cycle of 5hr (cross section view)