

## Effect of Activator Percent on Mechanical, Phases Properties of a Pack–Aluminized Low Alloy Steel.

Sanaa Abd alhadi Haffed

Department of Applied Science, University of Technology.

### Abstract

This study deals to diffusion coating treatment "Cementation" with aluminum in order to study phases and some mechanical properties including Micro hardness, Wear resistance, tensile strength and surface property indicating coating thickness layer. Low alloy steel was selected because it used widely in heat –exchangers and gas turbines so it facing a high mechanical pressures under oxidize and corrosive conditions. The specified specimens were treated with a different activator percent aluminizing pack (0,2,5%) of sodium chloride and under an inert atmosphere of argon gas flow ,to prevent the oxidation of treated samples ,results was illustrate successful of diffusion coating treatment to the all selected percent. That's a formation of a multi coating layer began from the surface layer ,transforming to interfacial layer and to a low matter diffusion layer , inspection show increment in micro hardness values in surface diffusion coating layer ,and dropping towards core ,this was ensure the diffusion coating mechanism successful. Microstructure images of treated systems ensure the formation of diffusion coating layer in different thickness with activator percent, also an improvement in wear resistance occurred and attributed to creation of aluminum phases with a high hardness.

Keywords : Cementation, aluminizing, Low Alloy Steel ,Activator Percent, mechanical properties.

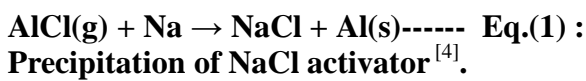
### Introduction

Low-alloy steels with moderate high-temperature strength, such as those used in petrochemical or electric utility plants, etc., often have poor oxidation, sulfidation and hot-corrosion resistance at (350 to 700°C). To take advantage of the low cost, good mechanical properties, and fabricability of these alloy steels, various types of CVD {The Chemical Vapor Deposition (CVD) processes are used in many fields of industrial activity to protect components from wear, corrosion or abrasion in engineering environments. These, industrial fields are diverse and range from gas turbines to gas cookers and from coinage to nuclear power plants. The CVD process relies firstly on the generation of a species that is produced by the reaction of the element that is to be deposited with another element that results in the substantial increase in the depositing elements vapor pressure. Secondly, this volatile species is then passed over or allowed to come into contact with the substrate being coated. This substrate is held at an elevated temperature, typically from 800 - 1150°C. Finally, the deposition reaction then usually occurs in the presence of a reducing

atmosphere, such as Hydrogen}. And PVD {physical Vapor Deposition} corrosion-resistant coatings are commonly deposited commercially. If high-temperature corrosion resistance can be realized for the surface, combined with the inherent strength, toughness, and fabricability advantages of the steel, a highly effective material will result. In particular, the melting, forming, and fabrication of the monolithic (Fe, Cr)<sub>3</sub>Al are difficult <sup>(1)</sup>. Recent papers, e.g., by DeVan *et al.*<sup>(2,3)</sup> show that the alloy compositions based on Fe<sub>3</sub>Al are very resistant to sulphdation and oxidation at high temperatures,<sup>[1,2,3]</sup>

The coating of alloys using a pack-cementation process is well known. Basically, pack cementation is a modified chemical-vapor-deposition process which consists of heating the parts to be coated in a closed or vented pack to an elevated temperature for a given time during which a diffusion coating of desired composition and thickness is produced on the metal. During the process, the contents of the pack are protected from oxidation by an inert or reducing atmosphere. In the cementation pack, the metal object or substrate to be coated is surrounded by the elements to

be deposited (usually in the form of metal or masteralloy powder), a halide-activator salt, and a filler powder. An inert gas, such as argon, or else its mixture with a small amount of hydrogen, is used to surround or permeate the pack. When the pack is heated in a retort to a sufficiently elevated temperature, the activator salt reacts with the elemental metal or masteralloy powder to form metal halide vapors, which diffuse to the substrate through the gas phase of the porous pack. By using alkaline or earth alkaline halide activators as for instance, NaCl, KCl or CaCl<sub>2</sub>, the precipitation of the activator salt has to be considered also <sup>[4]</sup>:



At the substrate surface, a reaction step results in deposition of the desired element(s) and the formation by solid-state diffusion of a very adherent coating with a composition gradient at the metal surface. <sup>[1,4,5]</sup>

In a normal pack cementation process, the substrates to be coated are placed in a sealed or semi-sealed container together with a well-mixed pack powder mixture containing the depositing elements (or their alloy), a halide salt activator and an inert filler (usually alumina). The substrates may be buried in (in-pack process) or suspended above (out-pack process) the pack powder mixture. The whole pack is then heated to and held for a required duration at a predetermined high temperature in the range of 700°C to 1150°C under a protective atmosphere of an inert gas. At high temperatures, the halide salt would react with the depositing elements. In a pack of specified composition, a series of halide vapour species containing the depositing elements would then be produced. In order to achieve the intended codeposition, the partial pressures of halide vapours of different depositing elements should be ideally controlled in a comparable range at any specified depositing temperatures. This may be accomplished by adjusting the content of the depositing elements in the pack and by carefully selecting a suitable halide salt as an activator <sup>[6]</sup>.

The Fe-aluminide intermetallic coatings can substantially improve the high temperature oxidation and corrosion resistance of alloy

steels through preferential oxidation of Al, which results in the formation of a stable Al<sub>2</sub>O<sub>3</sub> scale that acts as a protective barrier separating the underneath material from aggressive environment. Thermochemical surface treatment such as pack cementation or pack aluminising has proved to be an efficient technique for depositing a uniform layer of aluminide diffusion coatings on steels or on other types of metal alloys. Over the past two decades, there have been a few of detailed studies aimed at understanding the effects of pack variables and deposition conditions on the coating growth kinetics and hence to achieve a better control of the coating formation process. However, nearly all these studies were carried out at temperatures higher than 900 °C. Thermochemical treatment of alloy steels at these temperatures can severely degrade their mechanical properties such as high temperature strength and creep resistance. To prevent such degradation, the pack aluminising process is required to be conducted at temperatures below 700 °C. More recently, it has been demonstrated that alloy steels can be aluminised at temperatures as low as 525 °C. However, detailed processing data for pack aluminizing alloy steels at temperatures below 700 °C have been surprisingly sparse. This study is thus undertaken to systematically investigate the effects of pack compositions and deposition conditions on the Fe-aluminide coating formation process for low-alloy steels and subsequently to identify the parameters that determine the rate of coating growth at temperatures below 700 °C. It is anticipated that the results obtained can be applied to develop a diffusion coating for alloy steels with potential to substantially increase the long-term steam oxidation resistance of these steels at temperatures up to 700 °C, providing a critical scope to significantly increase the operating temperature and the efficiency of steam turbine power plants <sup>[3,6,7]</sup>.

## 2. Experimental Procedures

The substrate material used for the investigation was a low-alloy steel with a nominal composition {2.25 Cr - 1.0 Mo - 0.1C - Remainder Fe - (wt.%) } which is received from general company for mechanical industry. Specimens were cut to dimensions of

approximately  $\{(20 \times 10 \text{ mm})$  for wear,  $(4 \times 10 \text{ mm})$  for X-ray,  $(10 \times 10 \text{ mm})$  for microstructure and micro hardness} as in Fig.(1), and tensile test Specimens in conjunction with the relation  $(A=5D)$  as in Fig.(2) and then manually ground using SiC abrasive paper to a 600-grade finish. The specimens were then degreased before being placed in aluminising packs.

Aluminising powder mixtures were prepared by weighing out and mixing appropriate amount of powders of Al, tabular  $\text{Al}_2\text{O}_3$  which is received from Alcoa company and halide salt. The average particle sizes of Al is  $(850 \mu\text{m})$  and  $\text{Al}_2\text{O}_3$  powders were have the particle size distribution as illustrated in block diagram giving the powder identification in Fig. (3) .

Three percent of chloride salts (NaCl), were assessed as possible activator percents for the intended pack aluminising process at temperatures below  $700 \text{ }^\circ\text{C}$  {that's  $650 \text{ }^\circ\text{C}$ }. These salt percents were manually ground with a mortar and pestle, but not sieved, before being weighed out and added into aluminising powders. The in-pack process was used to pack aluminise the identified specimens. With this process, the substrates were buried in pack powders charged into a rectangular stainless steel retort ( $25 \text{ cm}$  length and  $15 \text{ mm}$  width) , which was then sealed with stainless steel lid and fire clay, Fig.(4) illustrated this container.

The fire clay seal was cured for at least 2 h at approximately  $110 \text{ }^\circ\text{C}$ . The pack was then loaded into an alumina muffle furnace (*nabertherm*), which was subsequently circulated with argon and the temperature was raised to and held at  $150 \text{ }^\circ\text{C}$  for 2 h to facilitate further cure of the cement. The furnace temperature was then raised to a final coating temperature, normally at  $650 \text{ }^\circ\text{C}$ , at a heating rate of  $7 \text{ }^\circ\text{C}$  per minute and was held at this temperature for a required duration. The furnace was then cooled to room temperature at its natural rate by switching off its power supply while maintaining the argon gas flow.

The coating times reported were the holding times at coating temperatures. The Vickers hardness values were measured using a micro –hardness testing device and a profile will given {Vickers hardness-testing technique (sometimes also called diamond pyramid). In

this test a very small diamond indenter having pyramidal geometry is forced into the surface of the specimen. Applied loads are much smaller than for Rockwell and Brinell testing techniques, ranging between 1 and 1000 g. The resulting impression is observed under a microscope and measured; this measurement is then converted into a hardness number or ((The modern microindentation hardness-testing equipment has been automated by coupling the indenter apparatus to an image analyzer that incorporates a computer and software package. The software controls important system functions to include indent location, indent spacing, computation of hardness values, and plotting of data. )). Careful specimen surface preparation (grinding and polishing) may be necessary to ensure a well-defined indentation that may be accurately measured. The Vickers hardness number is designated by HV, Vickers is referred to as microindentation-testing method on the basis of indenter size, its well suited for measuring the hardness of small, selected specimen regions. }, wear resistance values were measured by dry sliding wear test using pin on disc wear testing device .{ This testing device consist from a motor with a constant rotation velocity, arm to fixing of sample after connecting by the carrier and making it in a horizontal touching manner with aided of balancer (have babble) ,sample was touched the disc during rotation under the effect of vertical specified load. wear rates for the treated samples was calculated by the weighter methods which including the measuring of the sample weight after and before test by using sensitive apothecaries weighed from mettler (AE200) type with  $(0,0001) \text{ gm}$  sensitivity ,and then wear rates was calculated using the following equation }.

$$\text{Wear rate (W.R)} = \frac{\Delta W}{S.D} = \frac{W_0 - W_1}{\left(\frac{\pi D N}{1000}\right) \times t}$$

$\Delta W$ : Weight loss after test (gm).

S.D: Sliding distance (m).

W<sub>0</sub>: Initial weight before test (gm).

W<sub>1</sub>: Final weight after test (gm).

D: Diameter of sample trajectory (160mm).

N: Number of rotation per minute.(420 rpm).

T: Sliding time (10min.).

the tensile strength values were measured using computerized tensile testing device.

{by inspecting tensile specimen with a computerized tensile testing device through the application of a different vertical tensile forces until the fracture and determine the ultimate tensile strength for each treated sample }. Microstructure of the cross section of the coated specimens was analyzed using optical microscopy (OM) (Hitachi S-2400). X-Ray diffraction (XRD) was used to identify phases formed in the surface layer of coated specimens with a diffractometer who found in ministry of sciences and technology, and have the following measure conditions:-

-XRAY {(Target : Cu) ,(Wave:1.54060A), (Voltage:40.0KV) ,(Current:30.0(mA) }.  
 -Slit {(Divergence : 1.00000deg), (Scatter: 1.00000deg),(Receiving: 0.15000mm)}.  
 -**Measure** {(Axis:Theta-2Theta),(Scan Mode: Continuous Scan), (Range: 20.0000-60.0000 deg), (Step :0.0500deg), (Speed : 5.0000(deg/min) ,(Preset time:0.60 sec)}.

The thickness of the aluminised layer was very rough estimated of the depth of coat may be made from a fractured section of a test piece pack cementated along with the work but a more accurate measurement may be made metallographically. Across section of the test piece is grinded and polished with grinding papers (220,400,600,800,1000,1200) then with diamond paste, etched with a corrosive aching solution (nital) then washed with water and ethanol .dried and inspected under optical microscope , its image projected on to the screen of a camera of optical microscope at known magnification rule.

### Results and Discussion

The diffusion of Aluminum is one of the most promising methods to build superficial coatings for steel protection. Depending on the Activator Percent of the treatment, the studied properties can be varied. Obtained micro hardness profiles results for the aluminized coating treated systems were show an improvement and increment in surface values from (190 g/μm) in as received conditions to (354 g/μm) for pack alunminised with 5%NaCl, as in Fig.(6, 7) this was attributed to a substitutional solid solution formation and some precipitates of (Al) phases can be observed. The existence of these phases and their composition have been reported by X-ray diffraction patterns. {The possibility of

forming a natural composite, constituted by a hard phase of aluminides diffused in a substitutional solid solution, has an important consequence on the mechanical and protection properties of these coatings. Moreover, the diffusion of Al improves the adhesion of coatings}[8].

Diffusion coatings show a remarkable advantage over all other forms of coating, owing to the fact that the surface element moves from the coating to the substrate, gets into the crystal lattice and creates a band of "composite" material which makes surfaces resistant to hot oxidation ,aggressive environments and improve surface mechanical properties like hardness .The diffusion of Aluminum is one of the most promising methods for building surface coatings for steel protection <sup>[8]</sup> . This paper prove that the increment in activator percent provide an increment in hardness because that was in conjunction with the increment in master alloy (aluminum in this research) carrier ,and that increase the diffusion activities ,so hardness of a surface layers was increased with activator percent increment .{this in conjunction with the work of Brevaglieri et.al who studied the behavior of Aluminium coatings on AISI 304 stainless steel and evaluates the influence of intermetallic compounds obtained by suitable heat treatments <sup>[8]</sup>. A drop in microhardness values was recorded towards the core this was in conjunction with the deterioration of diffusion Phenomena throwing depth increment ,until the stability of hardness at 400μm depth, this was attributed to the little formation chances of hard intermetallic compounds,{this behavior was in conjunction with researcher A.fawzy mind when he Study the activator percentage influence of co-diffusion pack cementation coating on some phase, Mechanical and chemical Properties for steel alloys families <sup>[9]</sup>}.

Wear rates for pack aluminised systems shows a lower Values with activator percent increment, that's in conjunction with improvement in wear resistance ,this behavior is coincide with surface micro hardness improvement for higher activator percent(these two improvement behaviors are correlated to the formation of hard intermetallic compounds and a substitutional solid solution which give a

high atomic packing factor, with a high adhesion of coating, this is correlated with *brevaglieri* et al.<sup>[8]</sup>. The increment in diffusion coating layer thickness roughly approached to near 800  $\mu\text{m}$  for the highest activator percent, that's correlated with decrement in wear rate and improvement in wear resistance as in fig.(8). This concludes an empirical formula describes the activator percent effect on the surface Vickers hardness and wear rate for the aluminized coating treated systems. This improvement in microhardness and wear resistance was attributed to the created aluminum phases {which include some of intermetallic compounds} which investigated by X-ray diffraction including  $[(\text{Al}_3\text{Fe})]$ ,  $[(\text{FeAl}_2\text{O}_4)]$ ,  $(\text{FeAlO}_3)$ ,  $(\text{Al}_2\text{O}_3)$ , also this concludes inverse behavior between Vickers hardness and wear rate was give an empirical formula describes the hardening effect on the wear rate for the treated systems.

Coatings are designed in order to improve the surface properties of structural materials or to protect them against environmental degradation. As a result of different optimization priorities with respect to the specific tasks, coatings and structural materials differ from each other in chemical composition, microstructure and physical as well as mechanical properties<sup>[10]</sup>, according to the parameters of coating including activator percent, An improvement in ultimate tensile strength for the pack-aluminized systems were illustrated with activator percent increment as in Fig.(9) this behavior was analyzed in conjunction with Load is transferred from the structural material to the coating by shear stresses at the interface. If the bond strength at the interface is lower than the shear stress, delaminating will occur. When the interfacial strength is sufficient, the coating will follow the elongation of the structural material, this is in conjunction with the mind of K.Schneider and H.W.Grunling<sup>[11]</sup> and H.W. Grunling et.al<sup>[12]</sup> when they studied the TiC-Mo system This cracking will generally be multiple because of the load transfer to the coating by shear stresses. Cracks will occur within distances which are small enough that shear stresses within the remaining transfer

distance cannot build up as high as is necessary to create further cracks.

Residual stresses or strains in a coating should be added or subtracted depending on whether tensile or compressive conditions are found. Compression allows higher total strains before a coating cracks; tension reduces the tensile strain of the system that can be tolerated before the first crack formation in a coating. Cracking may occur with brittle coatings at total strains of about 0.1 %- 0.4% and even less when residual tensile strains have to be considered<sup>[11,12]</sup>. in a pack cementation process a formation of a compressive residual stresses chances is correlated with the activator percent increment and process duration. These simple estimations show that in most cases the mechanical properties of coatings themselves do not influence the properties of coated systems very much. A formation of a hard surface coating layer is in conjunction with the tensile strength improvement coating thickness in Fig.(10) were increased with activator percent increment this is attributed to the effect of halide salt which would react with the depositing elements. In a pack of higher activator percent, more series of halide vapor species containing the depositing element would then be produced and the efficiency of pack cementation process was increased this in conjunction with coating layer thickness, as illustrated in Fig.(11) which gives the microstructure and coating layers thickness of the treated samples in different activator weight percent, also formation of ferrite and pearlite phases was recorded in these images.

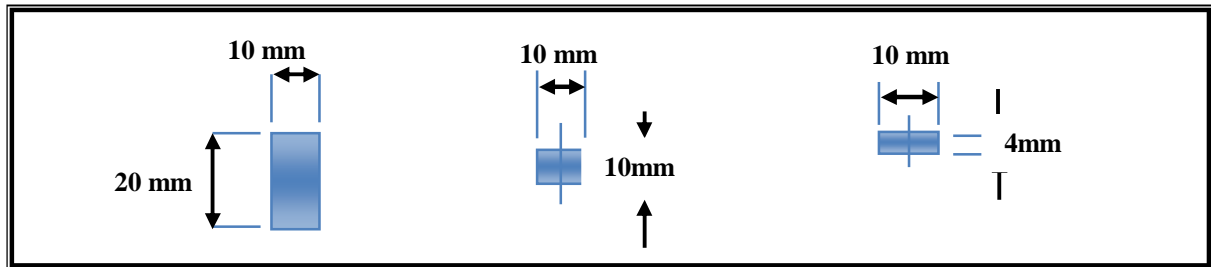
X-ray diffraction chart in Fig.(12) which show the formation of hard phases who recorded in Table (1).

From this table the formation of phase with activator percent increment was analyzed the cause of hardening with activator weight percent.

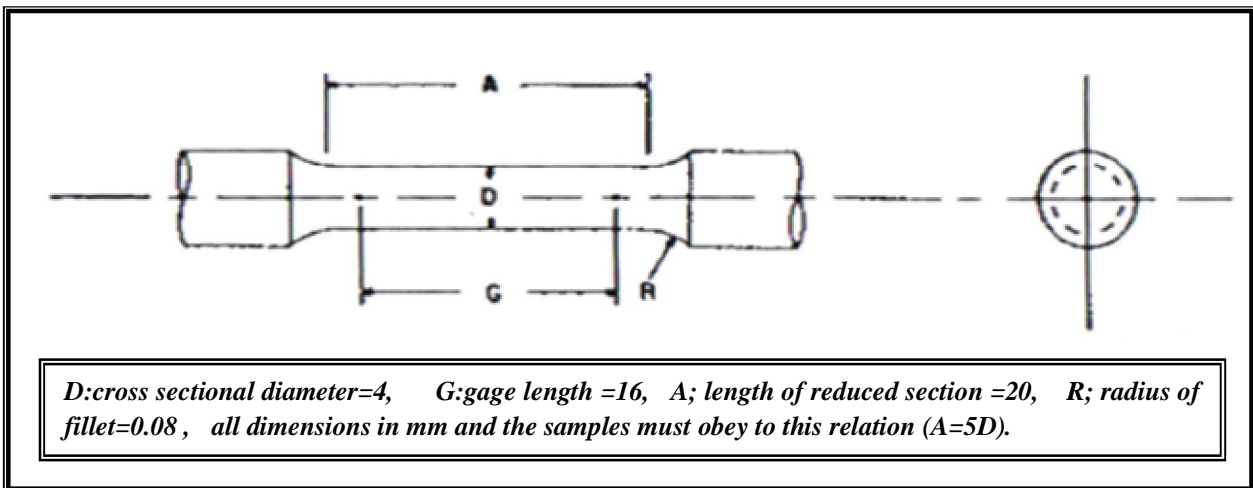
## Conclusions

1. Successful of diffusion coating.
2. Microhardness, wear resistance, tensile strength increase with activator weight percent of pack aluminized systems.
3. Coating thickness layer is increased with activator weight percent

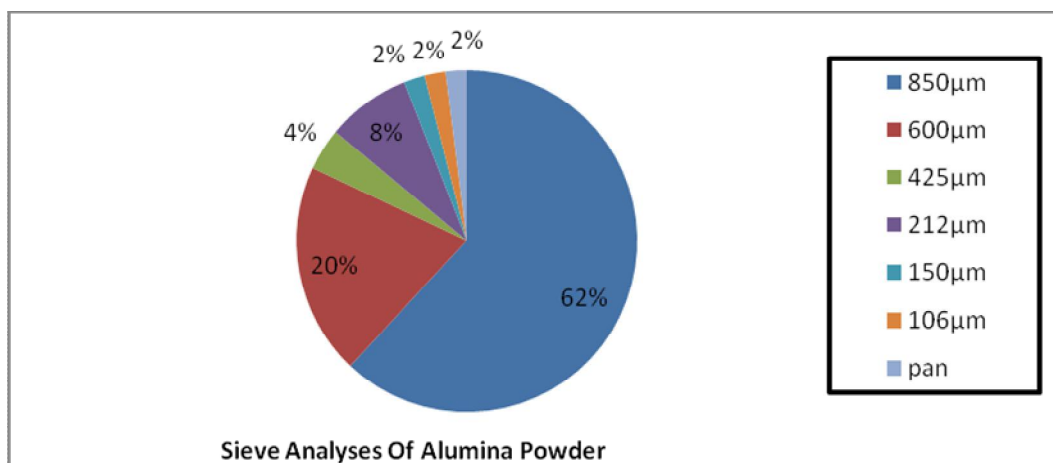
4. Formation of uniform coating layer for the pack aluminized systems.
5. The selected treatment has a high efficiency in varying of mechanical and surface properties.



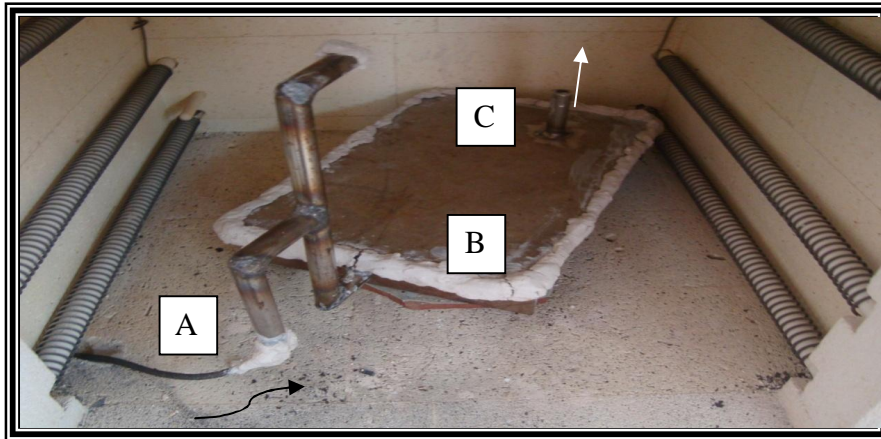
*Fig.(1) Shape and dimensions of Wear Sample test, Microstructure sample test, X-Ray sample test respectively.*



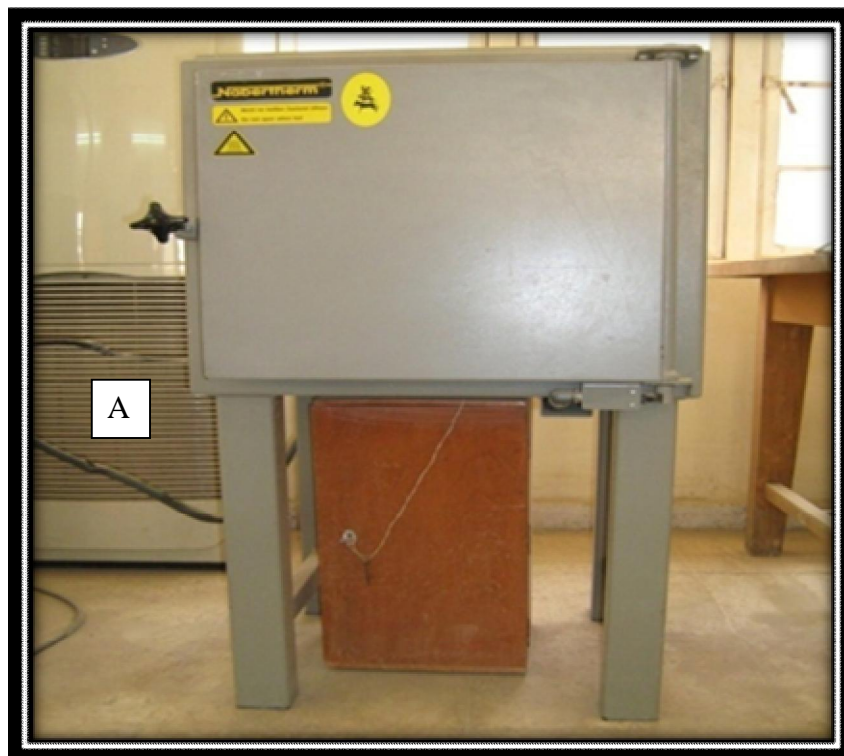
*Fig.(2) Show the shape &dimension of tensile Sample test.*



*Fig.(3) Block diagram giving the powder identification and the particle size distribution of alumina used as inert filler.*



*Fig.(4) The container A .inlet argon pipe B heat resisting steel container .  
C. outlet argon pipe.*



*Fig.( 5 ) The Furnace( nabertherm type) A .inlet argon pipe B. outlet argon pipe.*



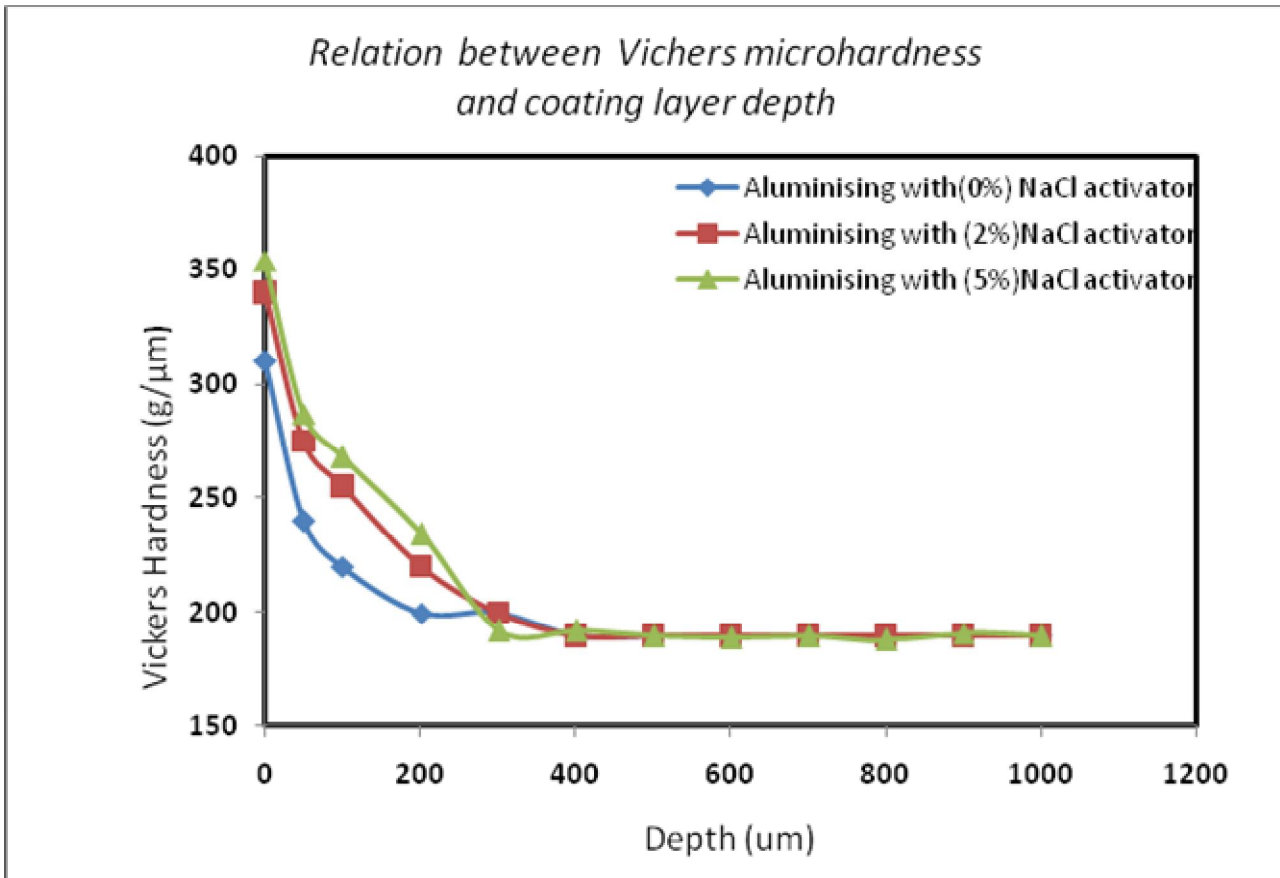


Fig.(6) Relation between Vickers micro hardness and coating thickness layer.

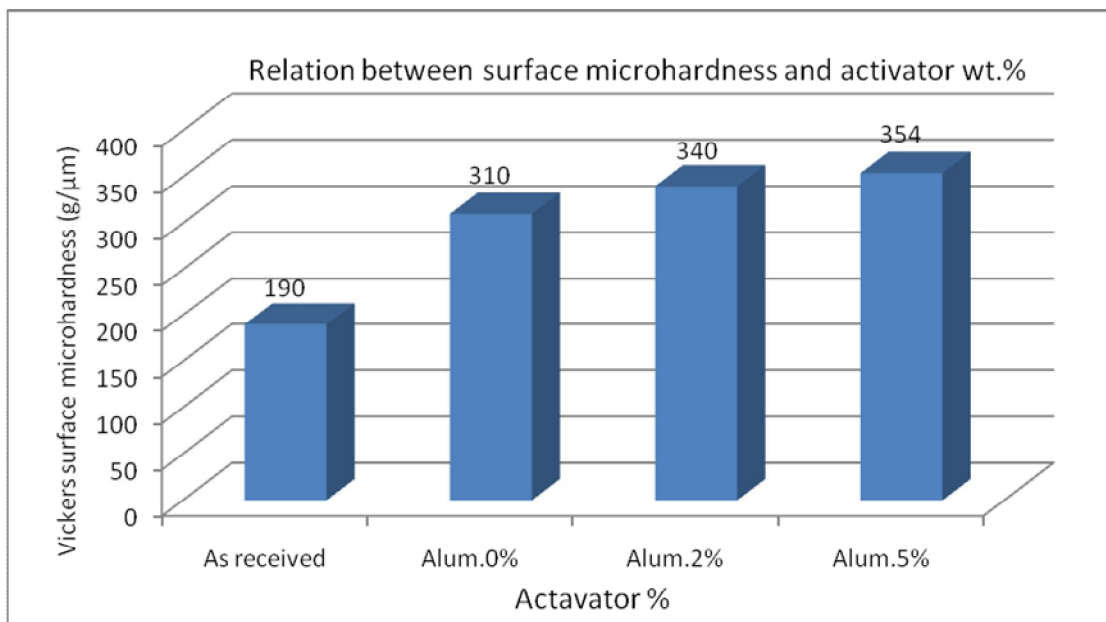


Fig.(7) Relation between Vickers surface micro hardness and aluminizing conditions.



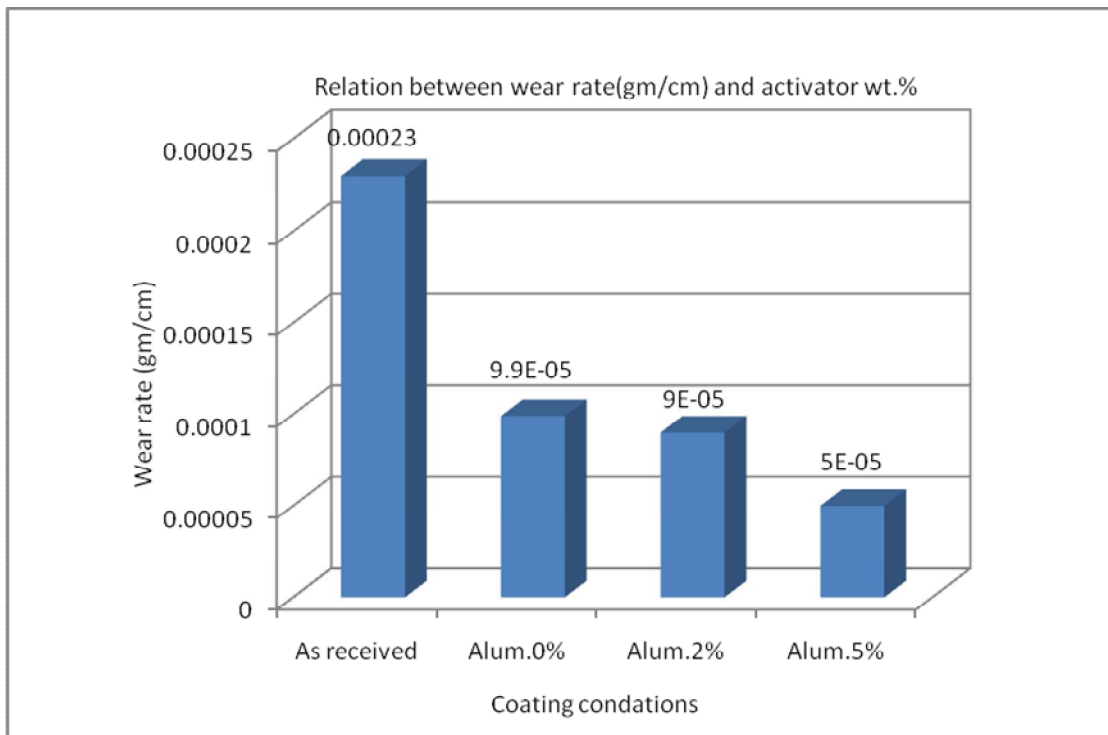


Fig.(8) (a) Relation between Wear rate (gm/cm) and Coating conditions.

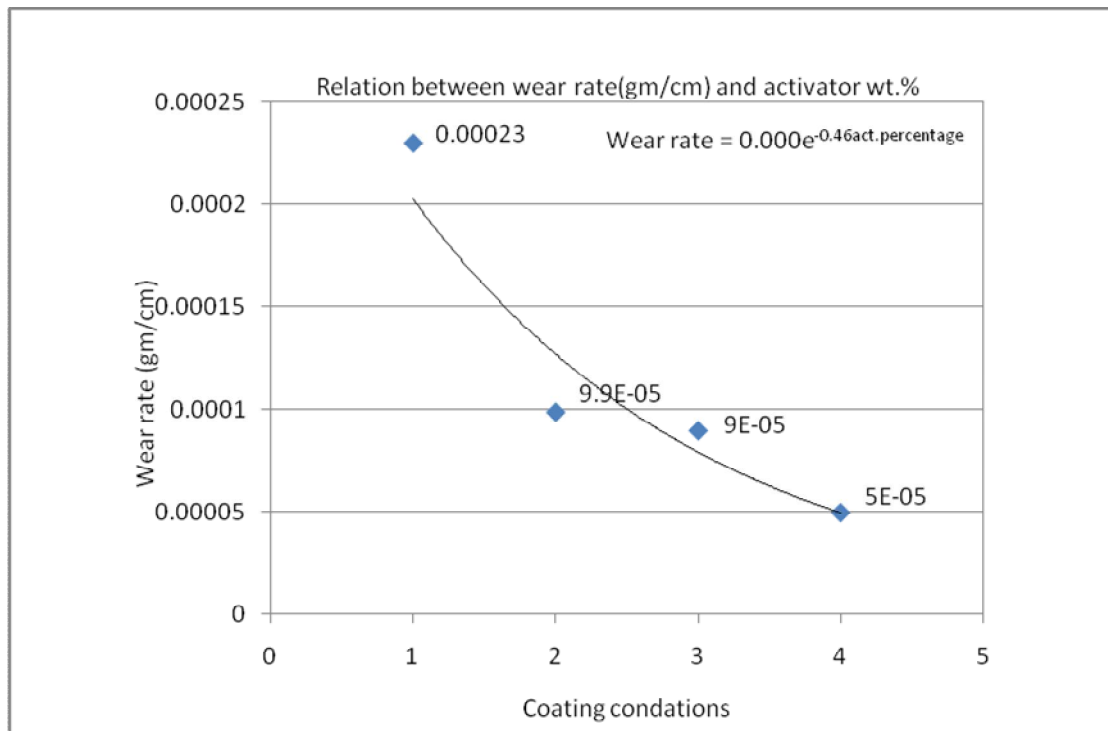
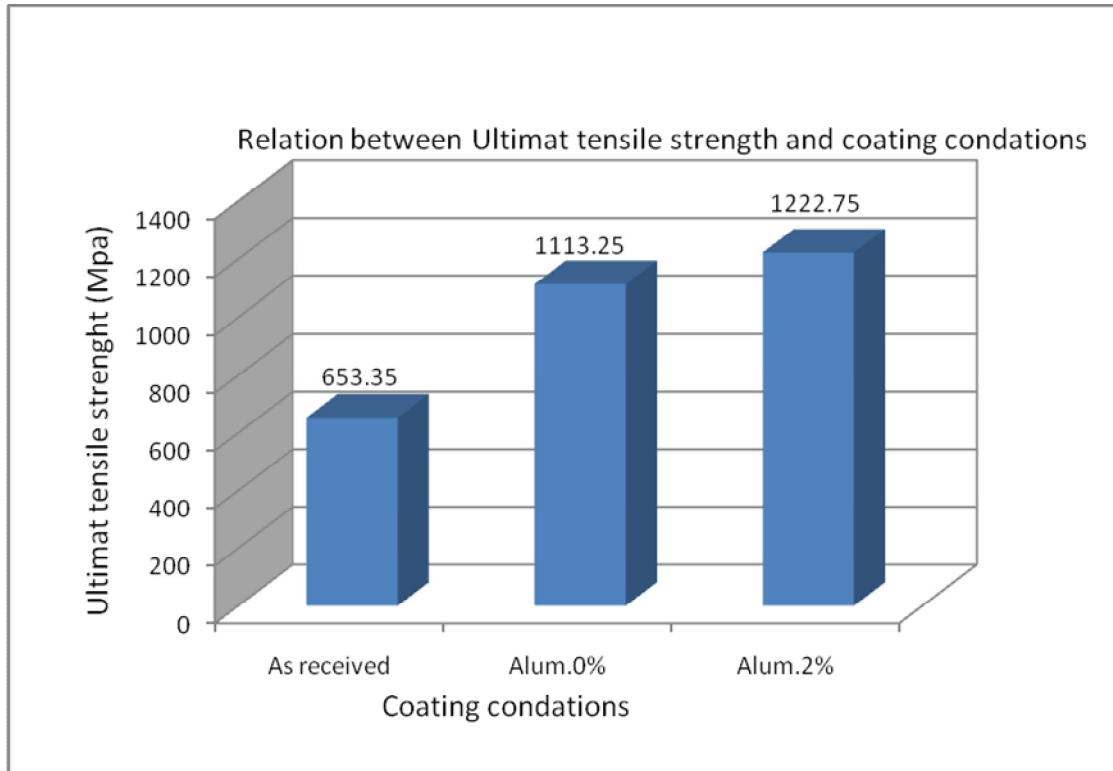
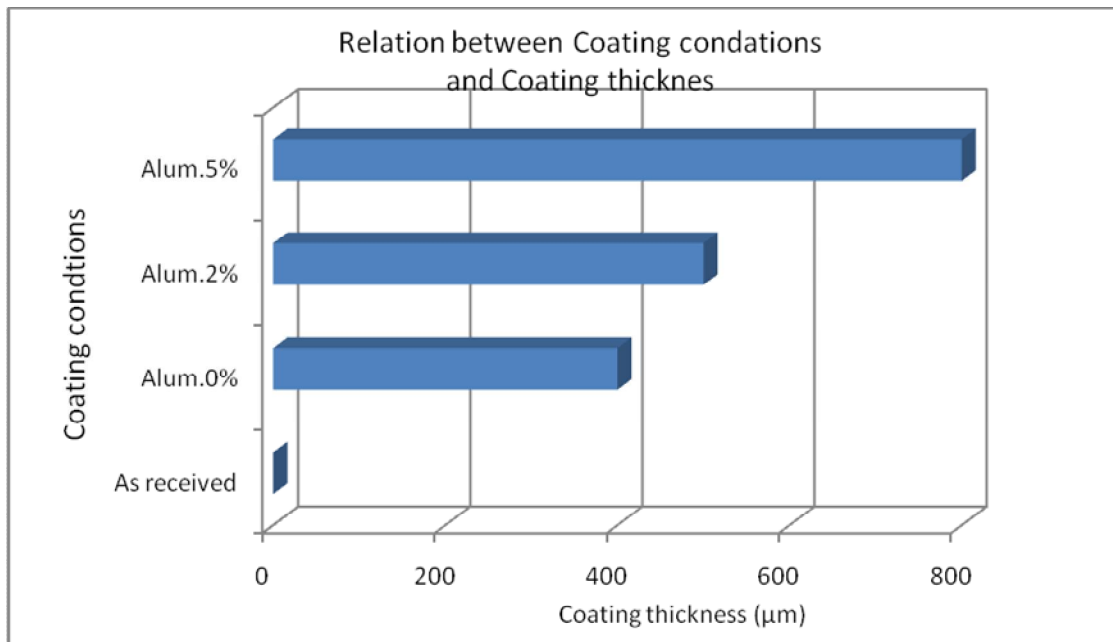


Fig.(8) (b) Relation between Wear rate (gm/cm) and Coating conditions (act%) empirical formula .



*Fig.(9) Relation between Ultimate tensile strength and Coating conditions.*



*Fig.(10) Relation between Coating conditions and rough estimated Coating thickness (µm).*



Image (1) core of as received condition X50 ,

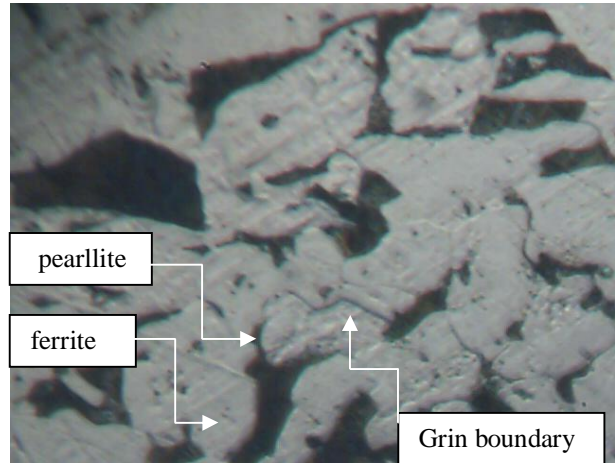


Image (2) as received condition X500

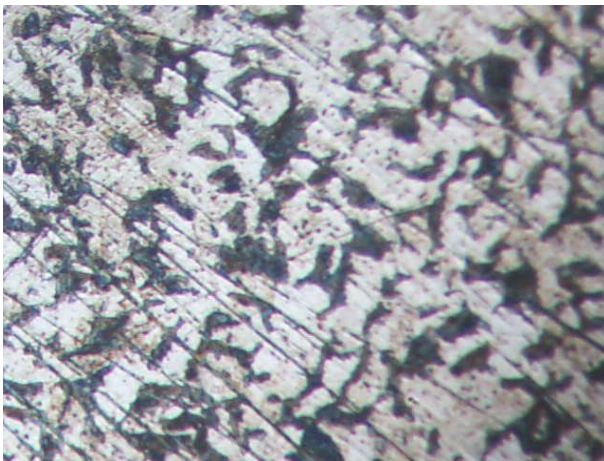


Image (3) core of Aluminising (0%act.)core X200

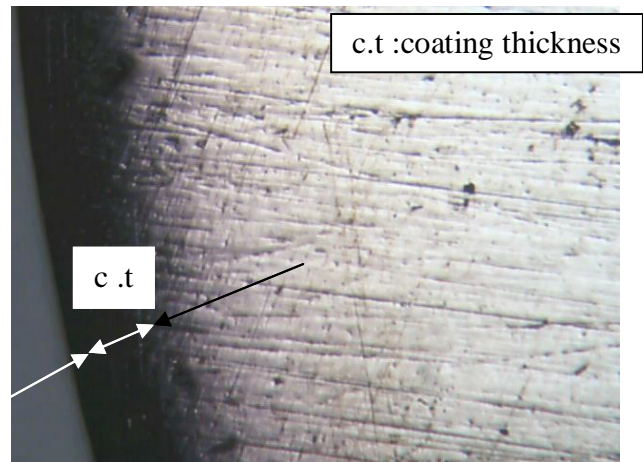


Image (3) X200 case.



Image (4) Aluminising(5%act.) core X200

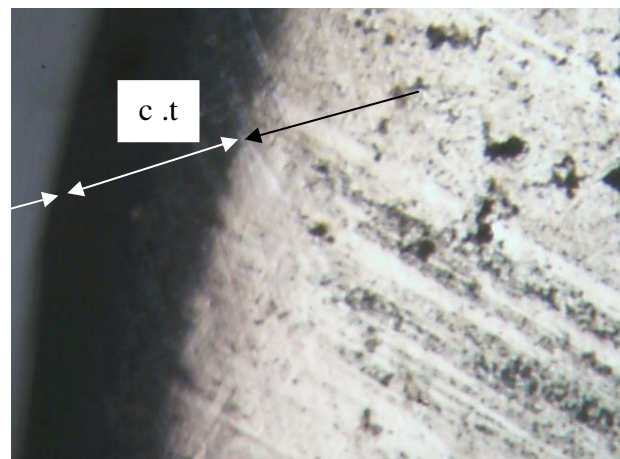
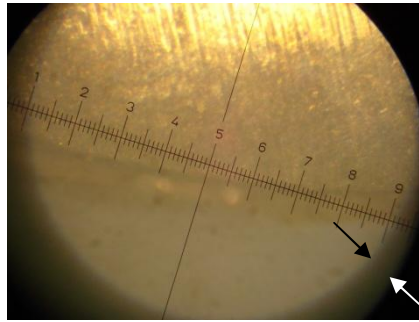


Image (5) case X200

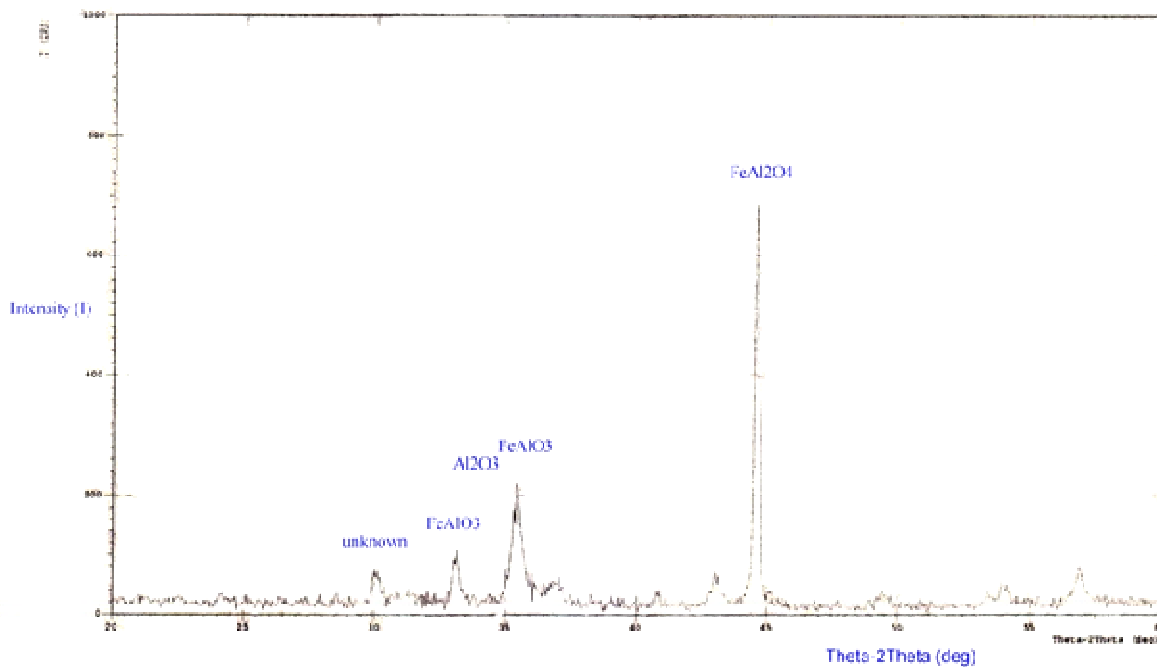
**Fig.(11) Microstructures images illustrate the created coating layers in aluminizing with different activator percent of sodium chloride.**



**Fig.(12) Surface images illustrate the created coating layers in aluminizing and mode of measurement.**

**Table (1)**  
**The created phases in aluminizing with(5%) of sodium chloride.**

| Seq. | 2θ      | d <sub>meas</sub> | d <sub>stan</sub> | Al <sub>3</sub> Fe | FeAl <sub>2</sub> O <sub>4</sub> | Al <sub>2</sub> O <sub>3</sub> | FeAlO <sub>3</sub> |
|------|---------|-------------------|-------------------|--------------------|----------------------------------|--------------------------------|--------------------|
| 1    | 34.384  | 2.60600           | 2.65              |                    |                                  |                                | ⊙                  |
| 2    | 35.487  | 2.52757           | 2.55              |                    |                                  | ⊙                              |                    |
| 3    | 43.0560 | 2.09916           | 2.09              | ⊙                  |                                  |                                |                    |
| 4    | 44.5805 | 2.03085           | 2.02              |                    | ⊙                                |                                |                    |



**Fig.(13) X-ray diffraction chart illustrate the created phases in aluminizing with (5%) of sodium chloride.**

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## الخلاصة

في هذا البحث تم إجراء معاملة طلاء انتشاري "سمنتة" بمادة الالمنيوم وذلك لغرض دراسة الخواص الطورية وبعض الخواص الميكانيكية متضمنة (مسقط الصلادة الدقيقة، مقاومة البليان ومقاومة الشد) وكذلك الخاصية السطحية التي تشير إلى سمك طبقة الطلاء. تم اختيار سبيكة الصلب السباتكي منخفض الكربون لاستخدامها في صناعة المبادلات الحرارية والتوربينات الغازية حيث تكون عرضة لضغوط ميكانيكية عالية تحت ظروف مؤكسدة وأكالة. عوملت العينات المحددة باستخدام نسب تنشيط (0,2,5)% من نوع كلوريد الصوديوم وتحت جو خامل وذلك من خلال ضخ غاز الأركون لضمان عدم تأكسد العينات المعاملة بهذه الطريقة وقد بينت النتائج ما يلي: نجحت عملية الطلاء الانتشاري عند كافة النسب المختارة حيث تكونت طبقة طلاء متعددة تبدأ من الطبقة السطحية وتتحول إلى الطبقة البينية ثم إلى طبقة يكون فيها تركيز المادة المنتشرة أقل ما يمكن مع الزيادة في قيم العمق وأظهرت نتائج الاختبارات والخواص الميكانيكية زيادة في قيم الصلادة الدقيقة في الطبقة الانتشارية السطحية وتدني هذه القيم باتجاه المركز ( قلب العينات) مما يؤكد نجاح آلية الطلاء الانتشاري . كما أظهرت نتائج مقاومة الشد القصوى ارتفاعا ملحوظا بزيادة نسبة مادة التنشيط المستخدمة . كما أتضح من خلال صور البنية الدقيقة للعينات المعاملة تكون طبقة الطلاء الانتشاري وبأسماك مختلفة حيث ارتبطت ارتباطا وثيقا بقيم نسب مواد التنشيط. كما أتضح تطور ملحوظ في مقاومة البليان وقد أوعز ذلك إلى دور الأطوار المتكونة ذات الصلادة العالية، التي أكدت كونها نتائج الفحص بالأشعة السينية.