Using of Pulsed Nd-Yag Laser in the Treatment of Thermal Spray by Aluminum and Diffusion Coating Phases of Low Alloy Steel

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Received on: 25/1/2010 & Accepted on: 7/4/2011

ABSTRACT

High power pulsed Neodymium doped Yttrium Aluminum Garnet-(Nd– YAG)- laser with 1J power was used to fuse coatings containing a mixture of FeAlO and Al. This mixture produced by aluminizing pack cementation treatment and thermal spray of low alloy steel, That is for Studying the mechanical , surface and metallurgical properties for each coating system , and to make a comparison before and after irradiation , and also the specified properties including the coating thickness . Aluminum Coated samples were made by two advanced techniques pack cementation and thermal spray methods in order to compare the resulted coating layer properties before and after irradiation, and also to determine the technique role from one hand ,also fusing effect and thermal residual stresses role which resulted from irradiation in the variation of studied properties on the other. Results showed that pack cementation coating layer has roughness, Micro hardness and wear resistance higher than thermal spray coating layer. Irradiation results was showed an improvement in studied properties, this was attributed to the fusing, thermal residual stresses and laser rays role in refining the grains which are in conjunction with finer microstructure . After irradiation a formation of a hard phase (martensite) was recorded, this was attributed to the sufficient percentage of carbon (0.4%) in the selected alloy, this was enshured by microstructure images taken by optical microscopy. X-ray diffraction showed, for laser surface irradiated coating layers, presence of the phase due to interaction between the base metal constituents. A modification of coating phases composition could be concluded for both coating techniques. Also the two techniques illustrate a formation of coating layer with a different thickness.

Keywords: laser surface irradiation, aluminizing, pack cementation, thermal spray, low alloy medium carbon steel.

استخدام ليزر ندميوم- ياك النبضي لمعالجة طبقات الطلاء الانتشاري والرش الحراري بالالمنيوم على سبيكة الصلب منخفض السبك متوسط الكربون

الخلاصة

ليزر نبضي عالي الطاقة من نوع نديميوم –ياك بطاقة 1جول قد أستخدم في البحث الحـــالي لصهر الطلاءات المحتوية على مزيج من FeAl و FeAlO والمنتجة من الطـــلاء بـــالألمنيوم

https://doi.org/10.30684/etj.30.2.4
2412-0758/University of Technology-Iraq, Baghdad, Iraq

بطريقتي السمنتة والرش الحراري لسبيكة من الصلب منخفض السبك متوسط الكاربون ، وذلك لغرض دراسة الخواص الميكانيكية والسطحية والطورية لكل نظام طلاء ومقارنة النتائج لكل معاملة بعد أجراء التشعيع بالحزم الليزرية. وتمت دراسة الخواص المحددة متضمنة سمك الطلاء. أنظمة الطلاء بالألمنيوم بتقنيتين مختلفتين هما السمنتة والرش الحراري قد استخدمت للمقارنة بين خواص طبقات الطلاء المنجزة قبل التشعيع مرة وبعده مرة أخرى. وذلك لتحديد دور تقنية الطلاء مرة والإجهادات المتبقية الحرارية الناتجة عن التشعيع بالحزم الليزرية في تحوير الخواص المدروسة مرة ثانية، وقد بينت نتائج الطلاء أن طبقة الطلاء بالسمنتة تمتلك خواص خشونة ، صلادة الدقيقة، و مقاومة البلى أعلى من طبقة الطلاء بالرش الحراري ،كما أوضحت نتائج التشعيع حدوث تطورا في الخواص المدروسة وقد أعزي ذلك إلى دور الصهر ، الاجهادات المتبقية الحرارية ودور الحزم الليزرية في تنعيم الحبيبات ، كما تم تسجيل ظهور لطور المارتنسايت بعد التشعيع وأعزي ذلك إلى وجود الكربون في السبيكة المختارة بنسبة كافية إضافة إلى الإخماد الذاتي مما يعزز من قابلية التصليد كما أكدت ذلك الصور الملتقطة بالمجهر الضوئي .وقد بينت نتائج الفحص بالأشعة السينية للطلاءات المشععة بالليزر تكون أطوار نتجت من التفاعل مع المادة الأساس ، وأن ذلك أكد على أحداث تحوير في أطوار كلتا التقنيتين المستخدمتين ،كما وأظهرت تقنيتي الطلاء المستخدمة تكون طبقات طلاء بأسماك مختلفة .

الكلمات المفتاحية: التشعيع بالحزم الليزرية ،الالمنة ،الطلاء بالسمنتة، الرش الحراري، الصلب منخفض السبك متوسط الكاربون.

INTRODUCTION

After 1968, the existing lasers were designed and fabricated with better reliability and durability. By mid 1970s more reliable lasers were made available for truly practical applications in the industrial applications such as cutting, welding, drilling and marking. During the 1980s and early 1990s the lasers were explored for surface related applications such as heat treatment, cladding, and alloying, glazing and thin film deposition. During the 2000s surface related applications which enabled ‘rebuilding’ of the previously created coatings and to improve wear hardness, of coated or uncoated materials become interesting [1-7].

It was observed that improved quality coatings could be obtained using this process. Iron aluminides possess several attractive properties motivating significant research and development efforts over the past years [8]. Fe_3Al and FeAl show excellent surface properties and resistance to oxidation and sulfidation in aggressive environments[8-12]. Due to their excellent properties and cost considerations, appropriate compositions of iron aluminides could find applications as coatings on more traditional higher-strength materials with inferior corrosion resistant properties at higher temperatures and/or wear resistance at ordinary temperatures. Previous studies on the corrosion resistance of weld overlays of iron aluminides on stainless steels have shown that weld overlays with at least 30 at. % Al provided adequate corrosion resistance in oxidizing/sulfidizing atmospheres [11]. Other experiments with bulk iron aluminides have shown that these aluminides have superior wetting characteristics with hard particles such as TiB_2 [13]. Thus, iron aluminide coatings treated with laser irradiation, could potentially possess a combination of superior oxidation/sulfidation properties along with excellent wear-resistance.

Pack cementation is a surface modification technique primarily used for metallic components to increase oxidation/corrosion resistance, wear resistance, and/or surface hardness. Pack cementation is a diffusion-based technique in which the desired species diffuses into the material through the surface. The powder pack consists of three general components: - the desired diffusion source material, a halide activator (such as NaCl or NH_4Cl), and inert filler (typically alumina). At elevated temperatures, the source material reacts with the halide to form a gas, which permeates the porous pack. Upon reaching the surface of the sample, another reaction occurs, depositing the source material on the substrate. Due to the high processing temperatures, the deposited element typically diffuses into the surface, leading to the desired surface treatment. The filler material is present simply to prevent sintering due to its simplicity and ability to coat even on complicated parts, pack cementation continues to be a popular surface treatment method.. Usually relatively expensive aluminum or binary alloys grade reagent is used during the pack process with aluminum as a source[14].

Thermal Spray techniques are processes which involve spraying melted materials onto a surface. The energy to heat the feedstock (coating precursor) is supplied by electrical (plasma or arc) or chemical means (combustion flame). Coating thicknesses range between approximately 20 micrometers (µm) and several millimeters (mm) depending on the process and feedstock. The materials to be deposited as the coating are typically fed into the spray gun in powder or wire form before being accelerated towards the material to be coated. "As the sprayed particles impinge upon the surface, they cool and build up, splat by splat, into a laminar structure forming the thermal spray coating. Generally, the coating quality increases with increasing particle velocities. Some common reasons for spray coating are, protection ,increase conductivity ,increase surface resistance and to reduce wear ,high temperature protection (thermal barrier coatings) ,and medical implants.

2-THE PURPOSE oF THIS STUDY

The purpose of this study was to explore the use of high-powered Nd-YAG lasers to synthesize iron aluminide coatings on low alloy medium carbon steel. The approach used in this study was to coat substrates made of steel with iron aluminide-based precursor coatings and subsequently fuse this coating based layers with the substrate using lasers.

3-EXPERIMENTAL

The substrate material used for the investigation was a low-alloy steel with a nominal composition as in table (1). Round Specimens were cut to dimensions of approximately (20L×10D)mm for wear specimens , (10 L ×10 D) mm for microhardness, microstructure ,coating thickness specimens and (4 L ×10 D) mm for XRD specimens .The specimens were manually ground using SiC abrasive paper to a 600-grade finish. The specimens were then degreased before being coated in aluminizing pack cementation, but for thermal spray technique, samples were not ground.

Aluminizing powder mixtures were prepared by weighing out and mixing appropriate amount of powders of Al_2O_3, halide salt and master alloy (for deposition of Al). The average particle sizes of master alloy and Al_2O_3 powders were 600 µm and 850µm, respectively. 5% percent of halide salts (NaCl), were assessed as possible activator percents for the intended pack cementation process at temperatures below 700 °C. The salt was manually ground with a mortar and pestle for 15 minute, but not sieved, before being weighed out and added in to cementation powders.

The in-pack process was used to pack the specimens. With this process, the substrates were buried in pack powders charged into a rectangular stainless steel retort (25 cm length ,15 cm width and 8 cm), which was then sealed with stainless steel lid and fire clay. The fire clay seal was cured for 1 h at 110 °C. The pack was then loaded into an alumina muffle furnace (Neber Theorm type), which was subsequently circulated with argon and the temperature was raised to and held at 150 °C for 2 h to facilitate further cure of the cement.

The furnace temperature was then raised to a final coating temperature, normally at 1000 °C, at a heating rate of 7 °C per minute and was held at this temperature for a required duration (2h). 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 resulted pack cemented specimens represent the first two sets of the coated specimens.

Another two sets of the specimens were coated by thermal spray with pure aluminum powder at 2000 °C for 15Sec followed by diffusion annealing under argon at 400 °C, four sets of the coated specimens were divided to four groups , first two groups from the coated specimens by two techniques were inspected immediately, the second two groups were surface treated by pulsed Nd-Yag Laser with 1.0 J and a pulse duration for1Sec and 10 pulses were then inspected.

The hardness values were measured using a Vickers microhardness testing device(100 gm normal load) and a profile will be given.

wear resistance was tested (dry sliding wear) against a(20 cm diameter) hardened steel pin. Weight loss measurements were made after 30 min intervals, with a normal force of 50N.Using pin on disc wear testing device and the wear rates was calculated using the following equation ;

$$\textbf{Wear rate (W.R)} = \frac{\Delta W}{S.D} = \frac{WO - W1}{\{\left(\frac{\pi DN}{1000}\right) \times t\}}$$

ΔW: mass loss after test (gm) with (0,0001 gm sensitivity).
S.D: Sliding distance (m).
Wo: Initial mass before test (gm), W1 : Final mass after test (gm).
D: Diameter of sample trajectory (160mm).
N: Number of rotation per minute.(420 rpm), t: Sliding time (10min.).

Microstructure of the cross section of the coated specimens was analyzed using optical microscopy. X-Ray diffraction (XRD) measurements were carried out to identify phases present in the samples of as-coated specimens with a diffractometer found in ministry of sciences and technology ,and have the following measure conditions:-

-Monochromatic XRAY { Cu Kα, 1.54 °A , 40.0KV,30.0(mA) }.

-Slit {(Divergence: 1. deg), (Scatter: 1. deg),(Receiving: 0.15mm)}.
- Measure {(Axis:θ-2θ),(Scan Mode: Continuous Scan), (Range: 20.-60.deg),(Step :0.05deg),(Speed :5.(deg/min) ,(Preset time:0.60 sec)}.

The thickness of the aluminised layer was measured metallographically. Across sectionof the test piece is ground and polished with grinding then with diamond paste, etched with a corrosive aching solution (Nital) and inspected under optical microscope , its image projected on to the screen of a camera of optical microscope at known magnification rule.

4- RESULTS AND DISCUSSIONS

Microstructure of essential or primary samples appeared ferrite and pearllite,also in their cores due to presence of medium carbon percentage Fig. 1 Image (a_1) . For diffusion (pack cementation) coating and thermal spray coating, a formation of an obvious dark coating layer began from the surface towards the core. The darkness was changed and decreased towards the centre of the samples Fig. 1 Image (b_1),(c_1). Also examination of non etched surface of alloy was considered in Fig. 1 Image (d_1) . During the process of laser surface treatment, Structural investigations of remelting path reveal significant refinement of structure Fig. 1 Image (a_2) the created molten pool was conducive to intensive mixing of materials of coating and base material and caused creation of so called 'outflow' on the edge of the solidifying pool. Also examination of etched surface of alloy was considered in Fig. 1 Image (b_2),(c_2) for the samples cores of diffusion (pack cementation) coating and thermal spray coating . Image (d_2). Presents an example of a remelted layer surface with the outflow on its side for the used irradiation rate.

Presence of morphology of solidification front was directly connected with temperature gradient and rate of solidification throughout the mass of the material.

High speed of heating and cooling accompanying remelting processes considerably increase the risk of loss of coherency and appearance of cracks. The investigations have not revealed such defects, which could have influenced deterioration of functional properties. The investigations involved determination of phase composition of the pack cementated, thermal sprayed coating surface layer. The investigations have been carried out by means of X-ray diffractometer, The obtained diffraction patterns are presented in (Fig. 2{charts (a),(b),(c),(d) }).

The investigations of phase composition of the aluminising pack cementated layer Fig. 2{charts (a)} revealed presence of three polymorphic phases of AlO : **alpha alumina** Al_2O_3**, Hercynite** $FeAl_2O_4$ {Iron (II)Aluminum oxide} with cubic cell (400) and $FeAlO_3$ **{Iron (III)Aluminate}** with orthorhombic cell (221). **Aluminum Iron** AlFe also will be distinguished in one phase Al_3Fe with orthorhombic cell (100).

But the investigations of phase composition of the thermal sprayed coating layer Fig. 2{chart (b)} reveals the presence of two polymorphic phases of AlO : **Hercynite** $FeAl_2O_4$ {Iron (II)Aluminum oxide} with cubic cell (422) and $FeAlO_3$ **{Iron (III)Aluminate}** with orthorhombic cell (222,420). which proves the fact of alpha alumina absence. The presence of three polymorphic phases of **Aluminum Iron** AlFe also distinguishes : Al_3Fe with orthorhombic cell (333,662), $(Al_5Fe_2)_a$ with orthorhombic cell (310) and **Iron Aluminide** Fe_3 Al with cubic cell

(311,222). Also presence of two polymorphic phases of aluminum chromium : {$Al_2Cr_{8?}$ and Al_9Cr_4 }with orthorhombic cell are detected.

For laser surface irradiated coating layers of the pack cementation the investigations of phase composition Fig. 2{chart (c)}reveals the presence of one phase of AlO: **Hercynite** $FeAl_2O_4$ {Iron (II)Aluminum oxide} with cubic cell (400) Moreover, presence of two polymorphic phases of **Aluminum Iron** AlFe also distinguishes : Al_3Fe with orthorhombic cell (21.1.1,14.6.0), $(Al_5Fe_2)_a$ with orthorhombic cell (130) and formation of aluminum chromium phase : $Al_2Cr_{8?}$ with orthorhombic cell(10),which revealed presence of the phase coming from the base material. That's indicates a modification of coating phases composition obtained.

But for laser surface irradiated coating layers of the thermal sprayed coating the investigations of phase composition Fig. 2{chart (d)} reveals presence of one phase of AlO: **Hercynite** $FeAl_2O_4$ {Iron (II)Aluminum oxide} with cubic cell (220,311,400) Moreover, presence of three polymorphic phases of **Aluminum Iron** AlFe also distinguishes: Al_3Fe with orthorhombic cell (10.2.0,440,333), $(Al_5Fe_2)_a$ with orthorhombic cell (310) and **Iron Aluminum** Fe_3 Al with cubic cell (220). Presence of three polymorphic phases of aluminum chromium phase AlCr: $AlCr_2$ with tetragonal cell,(103), Al_2Cr_3 with tetragonal cell, $Al_2Cr_{8?}$ with tetragonal cell which ensured presence of the phase coming from the base material. As in the irradiation of pack cementation That's indicates a modification of coating phases composition obtained in both coating techniques.

Measurements of Vickers microhardness on the cross sections were performed. On the basis of the test it can be assumed that remelting of the surface leads to occurrence of metastable structures which are characterized by higher strength properties. Microhardness of the alloyed zone amounted little improvement, Figs. 3 (a) show the variation in Vickers hardness values as a function of position within the coating- the heat-affected zone - (for laser treated surfaces) -, and the substrate . Indentations were made normal to the surface. Note that the hardness values within the coating are much larger (Vickers hardness values >300) than that obtained for the base material (Vickers hardness values < 255). It is intriguing to note that the coating prepared with pack cementation in the Fig. 3 (a) exhibits an average of larger hardness values than in the surface layers of thermal spray, and the two lower than that obtained with the laser surface treatment. Furthermore, higher hardness values are also observed for the overall profile as shown in Fig. 3 (b).

From the results of Vickers micro hardness, observation of an increment in hardness values for diffusion coating systems (aluminizing,) , formation of hardness profile which indicate increase in hardness at the sample edge this will be attributed to role of created hard phases ,this mind is in conjunction with Sanna A.Hafeed when she studied the effect of activator percents on mechanical, phases properties of a pack –aluminized low alloy steel and will be attributed the improvement in microhardness to the creation aluminum phases including [$(Al_5Fe_2)_a$,(Al_3Fe)],[$(FeAl_2O_4)$,(Fe_3Al),(α –Al_2O_3)] [15] .also these results in conjunction with Z.D. Xiang and P.K. Datta when they studied Pack aluminization of low alloy steels at temperatures below 700 ^{o}C whom obtained a similar observation [16].

For thermal spray coated systems which treated by diffusion annealing under argon also an increment in hardness was illustrated , and for the laser treated surface coated systems also an increment in microhardness profile as a result of hardening effect ,this mind is in conjunction with G. Muralidharan et al. when they studied Laser-Assisted Surface Modification of 4340 Steel with Iron-Aluminum Alloys, and Note that the hardness values within the coating were much larger (Knoop hardness values >400) than that obtained for the base material (Knoop hardness values < 300) [17].

From the pin-on-disk wear tests results showed in the Fig. (4) . The cumulative weight loss obtained over a period of 10 minutes has been normalized with that obtained for the base material. A study of the wear rates shows that both laser surface processing conditions and the composition of the initial coating layer have a significant effect on the wear properties. The effect of the laser can be observed by comparing the properties of the samples with the group that had the same initial composition of the sprayed or pack coating. Note that the wear property is better for the laser surface processing.

The relationship between the wear properties shown in Fig. (4,a) and the average Vickers microhardness values within the region 200 μm from the surface shown in Fig. (4, b), follows some interesting trends. The most prominent feature is that the wear in the coatings prepared by the thermal spray precursor layer is inferior to that prepared by pack cementation precursor layer. This is consistent with the trends in the average hardness values, as shown in Fig. 4 (b). Note that for the laser surface processing, the average hardness in the samples prepared with the thermal spray precursor is higher with corresponding lower wear rates. The trends in the wear properties can be related to the microstructure of the coatings. As could be observed from Fig. {2chart(d) } and as argued earlier, there is an evidence for the formation of **iron aluminides** on the surface only when the samples were processed with laser. In the pack cementation samples processed with the laser, dilution results in the formation of Fe-Al solid solution rather the iron aluminides Fig. 2(c).

The effect of this microstructural change is dramatically reflected in the improvement of the wear properties. These results, which show a significant influence of the phase constituents of the coating on the wear behavior, are consistent with previous measurements conducted on the wear properties of bulk aluminides and Fe-Al solid solutions, which showed that the wear properties of the aluminides were much better than that of the solid solution ,this in conjunction with G. Muralidharan et,al , J. A. Hawk et,al, D. E. Alman et,al and D. E. Alman et,al. their observations showed that the wear properties of the coatings containing an iron aluminide are comparable to that of 4340 steel [17-20].

Previous work on bulk materials showed that the wear properties of the iron aluminides are a strong function of the composition. For example, Fe_3Al with a composition of $Fe_{75}Al_{25}$ was found to be worse than 4340 in its wear properties, while Fe_3Al with a nominal composition of $Fe_{66}Al_{34}$ was found to better than 4340 with respect to its wear properties [19] . On the other hand FeAl with a nominal composition of $Fe_{64}Al_{36}$, showed wear properties comparable to that of 4340 steel. Alloying additions of Cr and Ti to FeAl was shown to improve the wear properties. The interplay between the composition, the phase structure of the iron aluminide ($FeAl/Fe_3Al$), effect of addition of TiB_2 and the wear properties of the coating is the subject of on-going experimental work.

Stress –strain curve for the selected alloy was illustrated in the fig. (6) and the inspected mechanical properties was given in table (2) in order to determine the identification of the studied alloy in this research.

5-CONCLUSIONS

1-During the experiment, laser treatment of Al was carried out in order to execute a process of alloying i.e. mixing of the coating and steel base material.

2-Application of remelting of metallic coating eliminates problem of adhesion to steel surface of thermal sprayed coating, which results cohesion occurrence forces

3- The layers did not reveal cracks and, despite higher speed of heating and cooling typical of laser surface treatment, i.e they are coherent.

4-Investigations of phase and chemical composition in the alloyed surface layer revealed
high concentration of the introduced elements, such as Al.

5-Presence of Fe in remelted region proves effectiveness of the applied laser treatment for thermal sprayed coating.

6-Microhardness measurements for the remelted region definitely prove increase of hardness parameters in the alloyed layer through introduction of alloying elements.

7- Pack cementation diffusion coating (aluminizing) have better functional properties and laser improved these properties.

8- Formation of diffusion coating layer have a gradient character from the surface to the core and their surface only was affected by laser treatment.

9- Laser surface treatment lead to Increment in surface hardness values as a result of hard new phases formation.

10- Wear resistances of the coatings increase in the presence of an iron aluminide in the
coating.

11- Laser surface treatment lead to Observable improvement in wear resistance for all selected treatment.

12-Even though the presence of the Fe-Al coating with an aluminide layer processed under
the present conditions did not significantly improve the wear properties over low alloy steel it
has anticipated potential advantages in improving the oxidation resistance.

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Table (1) Chemical composition of selected low alloy medium carbon steel alloy.

Ingredient	C	P	Si	Ni	Mn	Cr	Mo	V	Cu	W	Ti	Mg	Fe
Percentage	0.4	0.0158	0.2	0.101	0.311	1.101	0.09	-	-	-	-	-	98.1

Table (2) Illustrate the mechanical properties Values to selected low alloy medium carbon steel.

UTS MPa	Yield strength MPa	Elongation %	Hardness Vickers 100 g
755	502	20	250

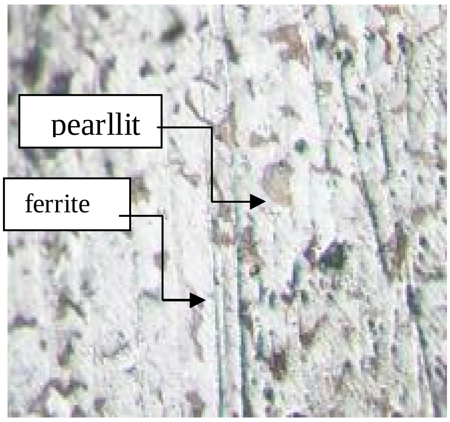

Figure (1) Image (a_1) microstructure for as received condition X200

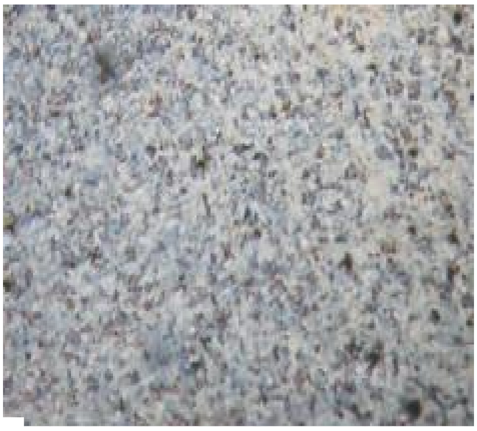

Figure (1) Image (a_2) for laser treated condition (treated with which amount of power energy or energy density in 1J/cm)

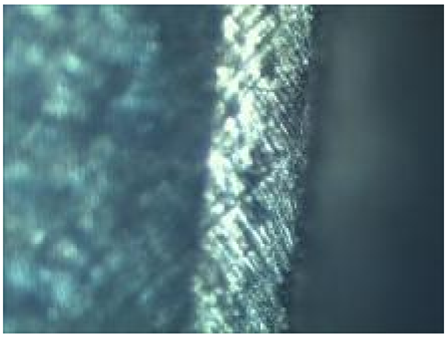

Figure (1) Image (b_1) surface micro structure Structure X200

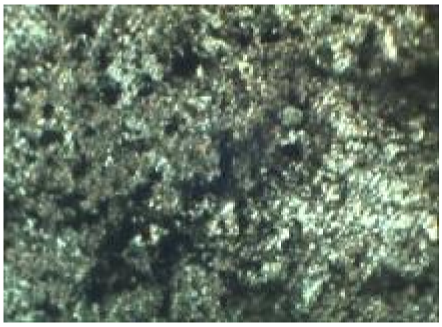

Figure (1) Image (b_2) core micro structure X 200

For pack cementation coating systems (Aluminizing)

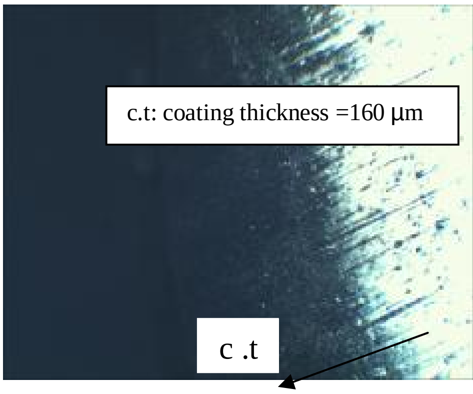

Figure (1) Image (c_1) surface micro structure X 200

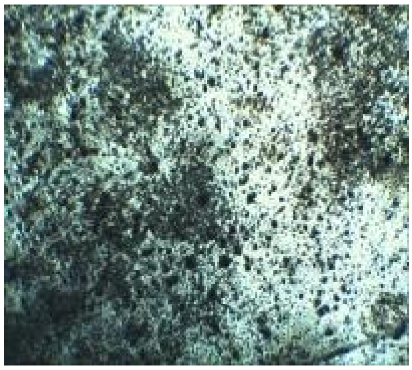

Figure (1) Image (c_2) core of diffusion coated systems X 200

For thermal spray coating systems. Treated by diffusion annealing at 550 °C.

Figure (1) Image (d_1) surface of as Received condition X100

Figure (1) Image (d_2) laser of as received condition X100

Figure (1) Show the microstructure, and morphological investigation for each selected treatment to low alloy medium carbon steel.

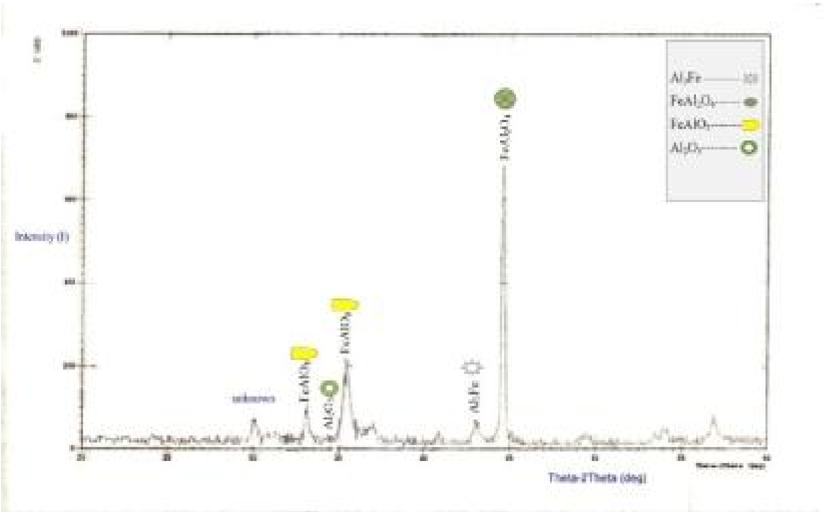

Chart (a) {XRD chart for samples treated by Aluminsing}

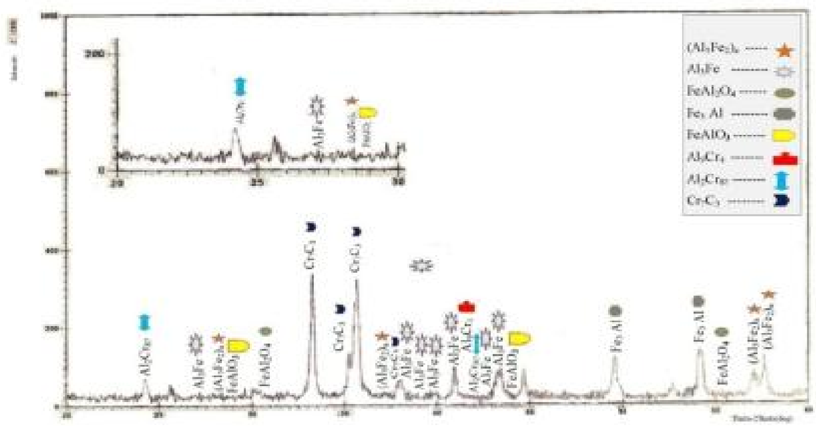

chart (b) {XRD chart for samples treated by Thermal Spray }

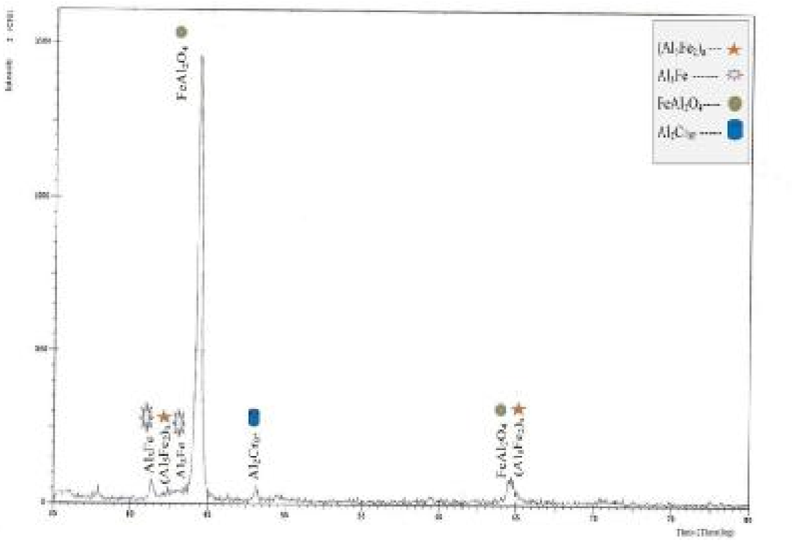

chart (c) {XRD chart for samples treated by Aluminsing +Laser }.

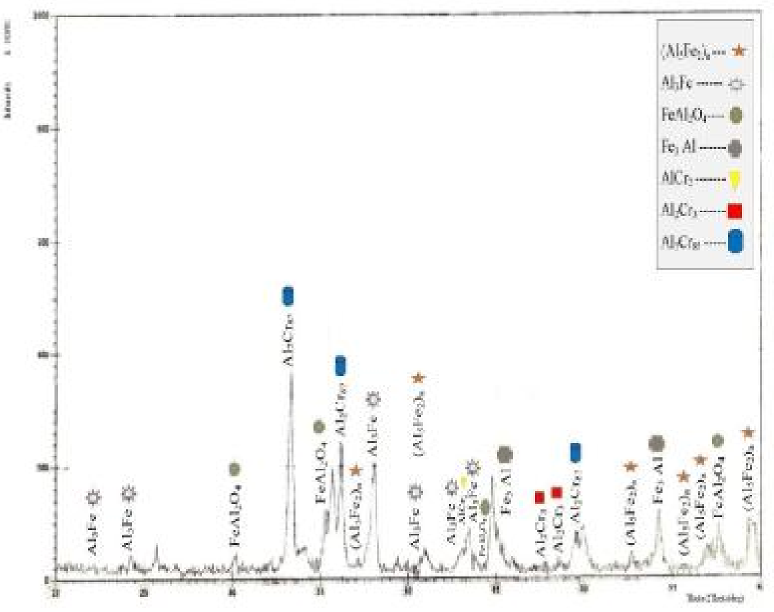

Chart (d) {XRD chart for samples treated by Thermal Spray +Laser}
(Figure (2) {XRD charts (a), (b), (c), (d)}).

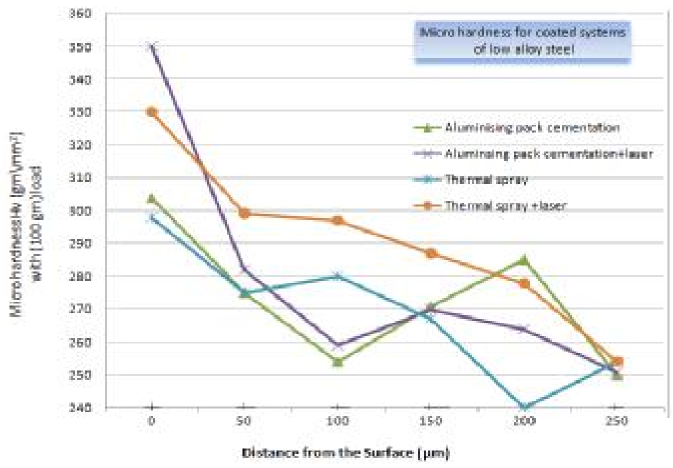

Figure (3) Micro hardness profiles curves of, (diffusion coated (aluminizing), thermal spray coated systems and for the laser treated surface coated systems.

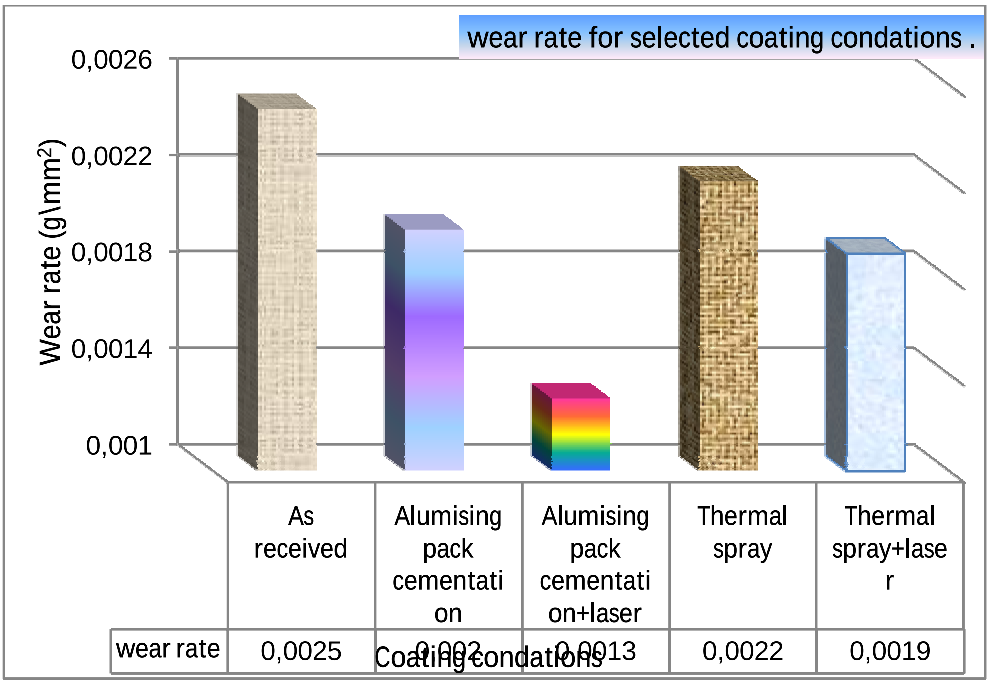

Figure (4a) A histogram diagram illustrating surface treatment influence on a wear rate of low alloy steel treated with surface treatment and laser surface treatment.

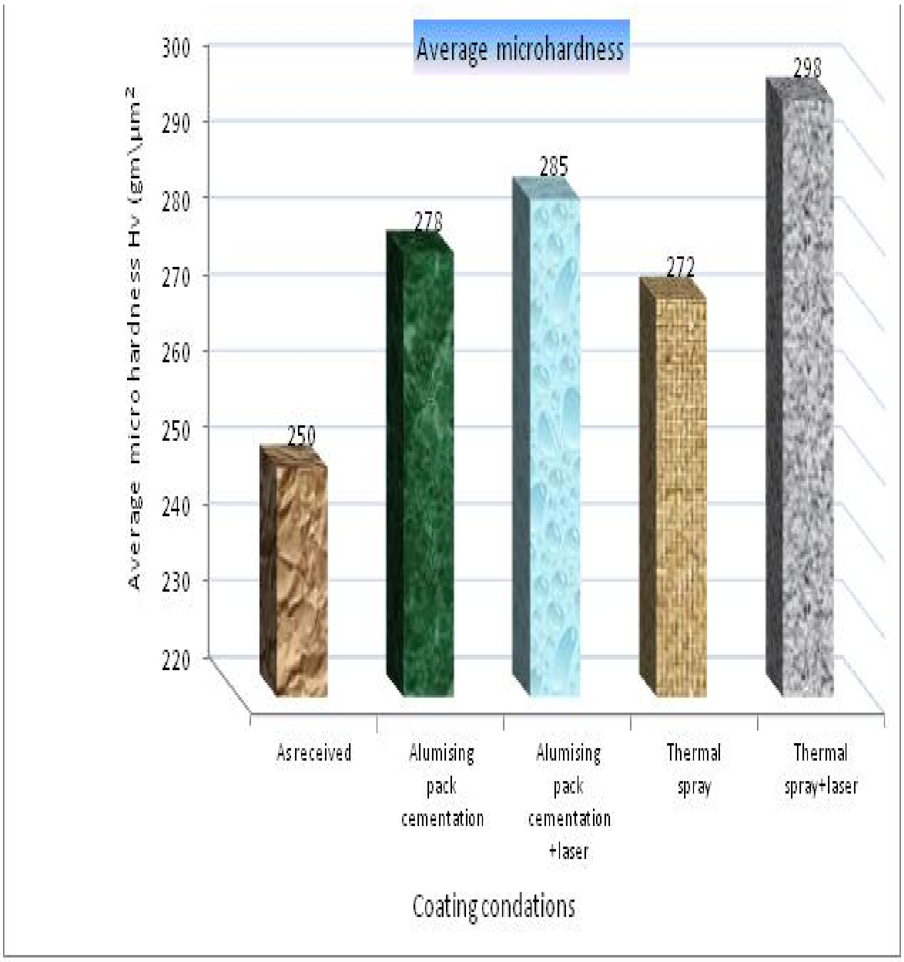

Figure (4b) A histogram showing the behavior of average micro hardness for (200mµ)to all the selected treatments and for the laser treated surface coated systems.

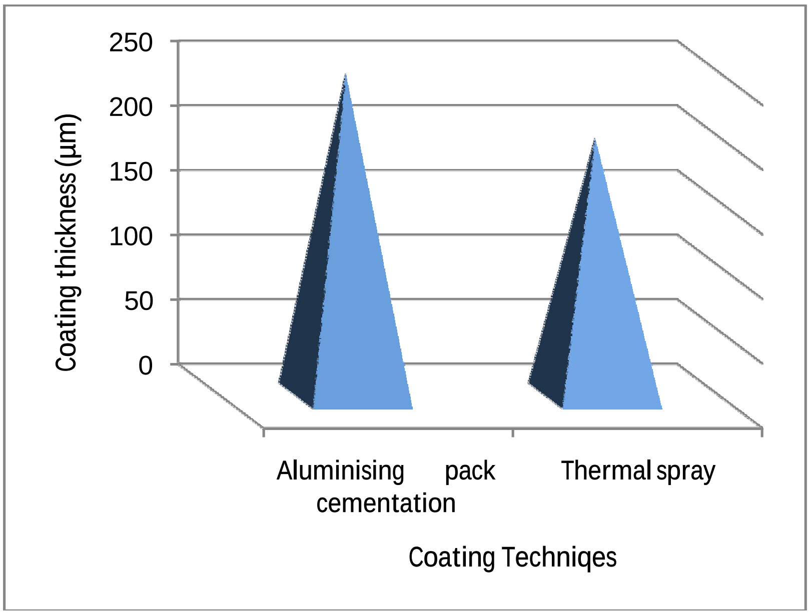

Figure (5) Rough estimated Coating thicknesses (µm) for Coating techniques.

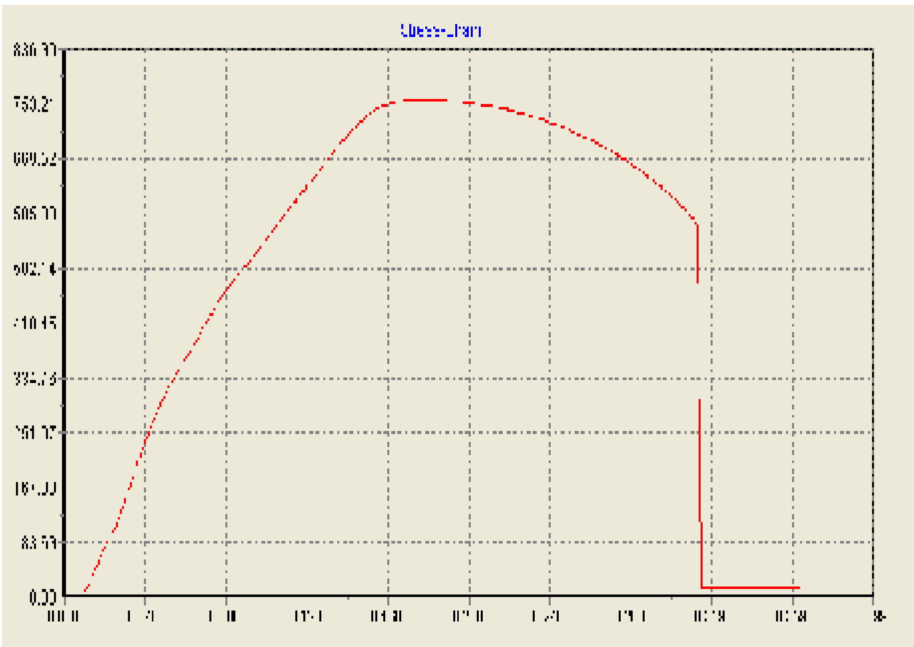

Figure (6) Stress strain curve for the alloy in as received conditions.