Taguchi Approach to Optimize Pack Aluminization Parameters in Carbon Steel Using MINITAB13

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Abstract

Pack aluminization has been rapidly developed and widely used in many fields due to its superior properties of coating. Surface is the only part of the component which has to coexist with external environment. Majority of engineering failures originate from the surfaces and components degrade in service leading to failures such as fatigue, wear, corrosion and oxidation. The present study deals with the surface modification of steel base through diffusion of aluminium by aluminium pack cementation for improving wear and corrosion resistance. The material chosen for study is medium carbon steel. Effect of varying weight percentage (wt.%) of halide activator (NH_4Cl) at different diffusion temperatures and times on the microstructure and microhardness of aluminized specimens was studied. Taguchi robust design technique using MINITAB13 was used to rank several factors that may affect the microhardness and microstructure in order to formulate the optimum conditions. The Taguchi orthogonal array L9 (3^3) was used for experimental design with three level of consideration for each factor. The respone (Microhardness) was analyzed based on the Taguchi's signal-to-noise ratio. The use of 4%wt. of (NH_4Cl) at 5hr and diffusion temperature of 700^{o}C seems to be the optimum condition, where the surface hardness could be increased to 1000Hv when aluminized. X-Ray diffraction studies have been confirmed the presence of aluminides in the surface layer, which could be instrumental in the significant increase in the surface hardness.

Keywords: Pack cementation, Diffusion, Taguchi method, Taguchi orthogonal array.

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استخدام طريقة تاكوجي لتحديد العوامل المثلى في عملية الطلاء بالألمنة في الفولاذ الكاربوني بواسطة MINITAB13

الخلاصة

ان طريقة الطلاء بالألمنة قد تطورت بشكل سريع و استخدمت بصورة واسعة في العديد من المجالات نتيجة خواص الطلاء الفائقة. و يعتبر السطح الجزء الوحيد من المنتج الذي يتعرّض بشكل مباشر الى الوسط الخارجي. ان أغلب حالات الفشل الهندسي تنشأ من السطح و تؤدي الى فشل الجزء الهندسي في التطبيق مثل الفشل بالكلال ، البلى، التآكل و الأكسدة. يتضمن هذا البحث، تحوير سطح الفولاذ من خلال انتشار الألمنيوم بواسطة التغليف بالسمنتة للألمنيوم و ذلك لتحسين مقاومة البلى و التآكل. ان المادة التي تم اختيارها في هذه الدراسة هي الفولاذ المتوسط الكاربون. وقد تم في هذا البحث دراسة تأثير النسب المئوية الوزنية المختلفة لهاليد المادة الفعّالة (NH_4Cl) عند درجات حرارية و أوقات مختلفة على التركيب المجهري و الصلادة المجهرية للعينات الخاضعة لعملية الألمنة. تم استخدام طريقة التصميم لتاكوجي بواسطة MINITAB13 لتحديد العوامل التي يمكن أن تؤثر على الصلادة المجهرية و التركيب المجهري و من ثم تحديد الظروف المثلى. مصفوفة تاكوجي من نوع L9(3^3) قد تم اعتمادها في تصميم التجارب نظرا لاستخدام ثلاث مستويات لكل عامل تحت الدراسة. كما تم تحليل الاستجابة (الصلادة المجهرية) من خلال الاعتماد على نسبة الاشارة/الاستجابة لتاكوجي. أظهرت النتائج أن استخدام 4%wt. من NH_4Cl عند زمن مقداره 5 ساعة و درجة حرارة مقدارها 700^oC يؤدي الى الحصول على الحالة المثلى ضمن مجموعة الظروف التي أستخدمت في هذا البحث حيث أن الصلادة السطحسية يمكن أن تزداد الى 1000Hv عندما يخضع الفولاذ الى الألمنة. أظهرت دراسات حيود الأشعة السينية وجود مركبات Aluminides في السطح و التي يمكن أن تلعب دورا فعّالا في الزيادة الكبيرة في الصلادة السطحية.

Introduction

The Fe-aluminide intermetallic coatings can substantially improve the high temperature oxidation, corrosion resistance and wear resistance of steels [1]. Pack aluminizing is a process that has been used for Ni-based superalloys and has been proven to be a very effective treatment for the oxidation protection up to 1100^{o}C [2]. Pack cementation is a diffusion coating formation process traditionally used to deposit Al on nickel base superalloys to form nickel aluminides coatings resistant to oxidation. More recently, some attempts have also been made to use this process to deposit Al on steel surfaces to form diffusion coatings resistant to corrosion and oxidation [3]. Many investigations in the corrosion and oxidation community have suggested that alumina scales form primarily by inward diffusion of oxygen [4]. Nature and properties of the initially-grown oxide scale gradually change and transform inmore protective scale (Alumina scale) [5].

In this study, an attempt has been made to improve the surface hardness of medium carbon steel by surface alloying with aluminium through diffusion. Aluminium coating also provides steel with excellent oxidation and corrosion resistance at elevated temperature.

Design of Experiment

The design of experiments (DOEs) is an experimental technique that helps to investigate the best combinations of process parameters, changing quantities, levels and combinations in order to obtain reliable results [6]. Design of experiments provide a powerful means to achieve breakthrough improvements in product quality and process efficiency [7]. Conventional statistical experiment design can determine the optimum condition on the basis of the measured values of the characteristic properties, while Taguchi's experimental design (also known as robust parameter design) does this on the basis of the variability of characteristic properties. In other words, the Taguchi method can determine the experiment condition having the least variability as the optimum condition [8]. Orthogonal arrays were designed by Taguchi as a basis for experimental design [9]. Taguchi used the signal-to-noise (S/N) ratio as the quality characteristic of choice [10]. The signal-to-noise is calculated according to the following equation [11]:

$$S/N = -10\log_{10}\left(\sum_{i=1}^{n} y_i^2\right) \qquad (1$$

Where n is the sampling size and y_i is the response at each sampling point. The units of signal and noise are in decibel (db) [12].

In this work , an approach based on Taguchi method is used to determine the optimum pack aluminization parameters more efficiently. The larger-the better approach of Taguchi method has been used for analysis of experimental results. A model based on L9(3^3) orthogonal array of Taguchi design was created to optimize the process parameters.

With L9(3^3) the signal to noise ratio can be calculated and is used in analyzing the effect of various factors [13]. Improvement in computer technology,made it easier to use Taguchi approach in applications.The most common analyze technique by computer is MINITAB13. All tables and figures in this work was processed using MINITAB13.

Experimental Procedure

The substrate material used in this work was medium plain carbon steel. The samples were cut into squares shapes with dimensions (20mm×20mm×5mm). The chemical composition of the material was analyzed using a direct reading (carried out at Materials Engineering Department /University of Technology) , and is given in Table 1. All surfaces, including the edges were wet ground using 320,600,800, and 1200 grit silicon carbides papers. These sample were then cleaned with water, degreased with acetone and then ultrasonically cleaned for 30 minutes using ethanol as a medium. After drying the samples were stored in polyethylene zip-lock hags. The pack mixture used in each run is shown in Table 2. It was decided to employ three different diffusion temperatures and times as listed in Table 3. The orthogonal array $L9(3^3)$ was used to study the influence of three factors which were (NH_4Cl (%wt.), Time and Temperature of diffusion coating) against microhardness. Each factor was considered at three levels. The factors involved and their levels were shown in Table 3. By using Taguchi orthogonal array $L9(3^3)$ for experimental design, the number of trial runs was reduced to 9 simple and effective experiments. It could save experimental cost and time. Table 4 illustrated the orthogonal array $L9(3^3)$ as indicated in [9], since there were three of three levels factors, these factors were assigned to all three columns in the $L9(3^3)$ array.

Optical Micrography

For metallographic examination, the specimens were sectioned and mounted. To observe the microstructure of the surface coating layer formed by diffusion-treatment, the cross-sections were mechanically polished using emery papers of grade 220-600 and final polishing was carried out using diamond paste. The polished specimens were etched using 2% nital at room temperature for 3s.

Microhardness on the cross section

The microhardness of the surface layers of as-aluminzed specimens were measured using microhardness tester (carried out at Department of Materials

Engineering /University of Technology). The hardness tests, including hardness traverse were carried out from the surface to the core (Applied Force=0.01 N).

Results and Discussion

The results of nine trial conditions, with six run per trial condition are shown in Table 5. In this study, the higher values of microhardness is desirable. Thus, it was categorised in the "Bigger is Better" quality characteristic. All of the results were transformed into signal to noise (S/N) in last column of table of Table 5. A Taguchi design of experiments has the advantage of allowing the effect of each process variable (called "Main Effect") to be statistically evaluated. In this case, a popular statistical technique called MINITAB13 has been used. Using quality engineering we compare the response for each factor using a response curve such as the one shown in Figure 1. The horizontal axis correspond to control factors. The points in the upper part of the graph (high S/N ratio) indicate superior conditions. Response curve can be used to find optimum levels. Figure 1 shows the main effects plot for the S/N ratio. The main effect plot shown in this figure indicates that the highest point is the optimum parameter for each factor. This means that the 4%wt.of $NHCl_4$, 5hr and 700^{o}C, are at the optimum level. The details of average effect of S/N ratio is given in Table 6. Table 7 gives the optimum control parameters obtained from S/N ratio in each level.

Figure 2 illustrates typical microstructures of aluminized diffused steel specimens with varying process parameters. At higher temperatures 800^{o}C, the coating appears to become more porous in nature possibly due to oxidation. The lower amounts of NH_4Cl also yields thin coatings which are delaminated even in the low diffusion temperatures. The higher percentage of NH_4Cl with low diffusion temperatures showed a less porous diffusion layers. Figure3 shows the hardness distribution from surface to steel substrate core at various level. In the case of as it is received (without aluminization) the surface hardness was found to be low around 55-70 Hv. After diffusion process, the surface hardness of the steel increased to a maximum of 1000Hv. This is due to the pick up of aluminium by steel during aluminizing and formation of iron-aluminium complex layer during diffusion. The lower the weight percentage of NH_4Cl of specimens yielded the increased hardness at the near surface where as the high percentage of NH_4Cl extended its increased hardness towards the core material. The specimen diffused at 700°C and 5hr as a diffusion time , considering a less porous surface and has the increased microhardness and case depth.

X-Ray Diffraction Studies

X-ray diffraction (XRD) were performed on the outer layer of aluminized specimens. The XRD characteristics of aluminized specimens using Cu-K are shown in Figure 4. The diffraction patterns of these specimens show that FeAl,Fe_3Al and Fe_2Al_5 intermetallic phases are present in theses coatings. Thus, the increased hardness in aluminized samples would be due to the presence of iron aluminides in the layer.

Conclusions

In this work the Taguchi method was used to find the important parameters affecting the pack aluminzation of medium carbn steel with hope the optimum parameters can be controlled to obtain the best microhardness. Based on this study the following conclusions can be arrived at:

1. Based on the Taguchi approach, considering a low porous surface and the increased case depth, the use of 4%wt. of NH_4Cl at diffusion temperature of 700°C and diffusion time of 5hr seems to be the optimum condition.
2. The hardness of medium carbon steel could be increased to a maximum of 1000Hv when aluminized at (4%wt. of NH_4Cl at diffusion temperature of 700°C and diffusion time of 5hr).
3. X-Ray diffraction studies have confirmed the presence of alumindes in the alloy layer, which could be instrumental in the significant increase of surface hardness in medium

carbon steel when aluminized.

Hence, Aluminizing could improve the surface characteristics of medium carbon steel components.

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Table 1 Substrate Material Composition

Element	C	Si	Mn	P	S	Mo	Fe
%	0.40	0.111	0.643	0.022	0.021	0.001	Balance

Table 2 Pack Mixtures

Source	%wt.	Halide Activator	%wt.	Inert Filler	%wt.
Al	80	NH_4Cl	2	Al_2O_3	18
Al	80	NH_4Cl	3	Al_2O_3	17
Al	80	NH_4Cl	4	Al_2O_3	16

Table 3 Design factors and their levels for orthogonal experiment.

		Level Number		
Column	Factors	1	2	3
1	NH4Cl(wt.%)	2	3	4
2	Temperature(oC)	600	700	800
3	time(hr)	5	6	7

Table 4 L9 Orthogonal array.

L9 Orthogonal Array

Ex.No	NH4Cl(wt.%)	Temperature(oC)	time(hr)
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

Table 5 Experimental results and their S/N Ratio.

Experimental Results and Their S/N Ratio

Ex.No	R1	R2	R3	R4	R5	R6	S/N Ratio
1	600	700	800	900	200	215	50.4376
2	800	700	1000	200	215	216	49.1680
3	400	800	1000	200	200	205	48.6113
4	200	250	400	600	650	205	48.9625
5	600	630	640	600	400	200	51.5732
6	600	800	1000	650	1000	210	52.8146
7	800	800	200	198	199	199	47.6055
8	1000	1000	950	800	700	280	54.8886
9	900	800	900	900	800	250	54.1967

Table 6 Variation of S/N ratio with different parameters.

Response Table for Signal to Noise Ratios

Larger is better

Level	NH4Cl(Wt.%)	Temperature(oC)	Time(hr)
1	49.4056	49.0019	52.7136
2	51.1168	51.8766	50.7757
3	52.2303	51.8742	49.2633

Table 7 Optimum parameters details.

Optimum Parameters Details

Parameters	Optimum Level	Value
NH4Cl (Wt.%)	3	52.2303
Temperature (oC)	2	51.8766
Time (hr)	1	52.7136

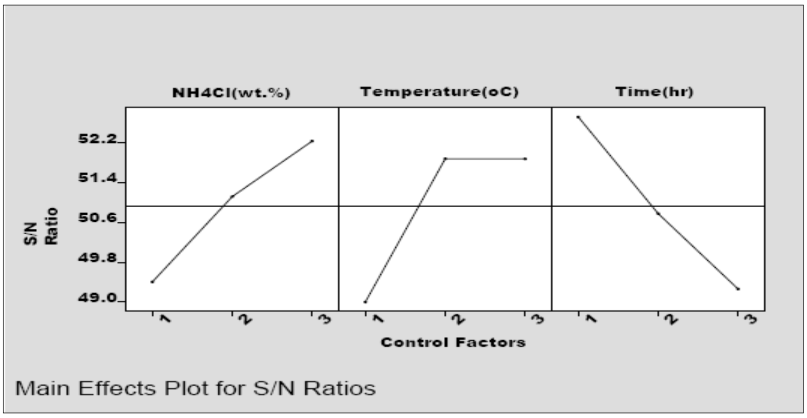

Figure 1 Graph of factor response of the microhardness.

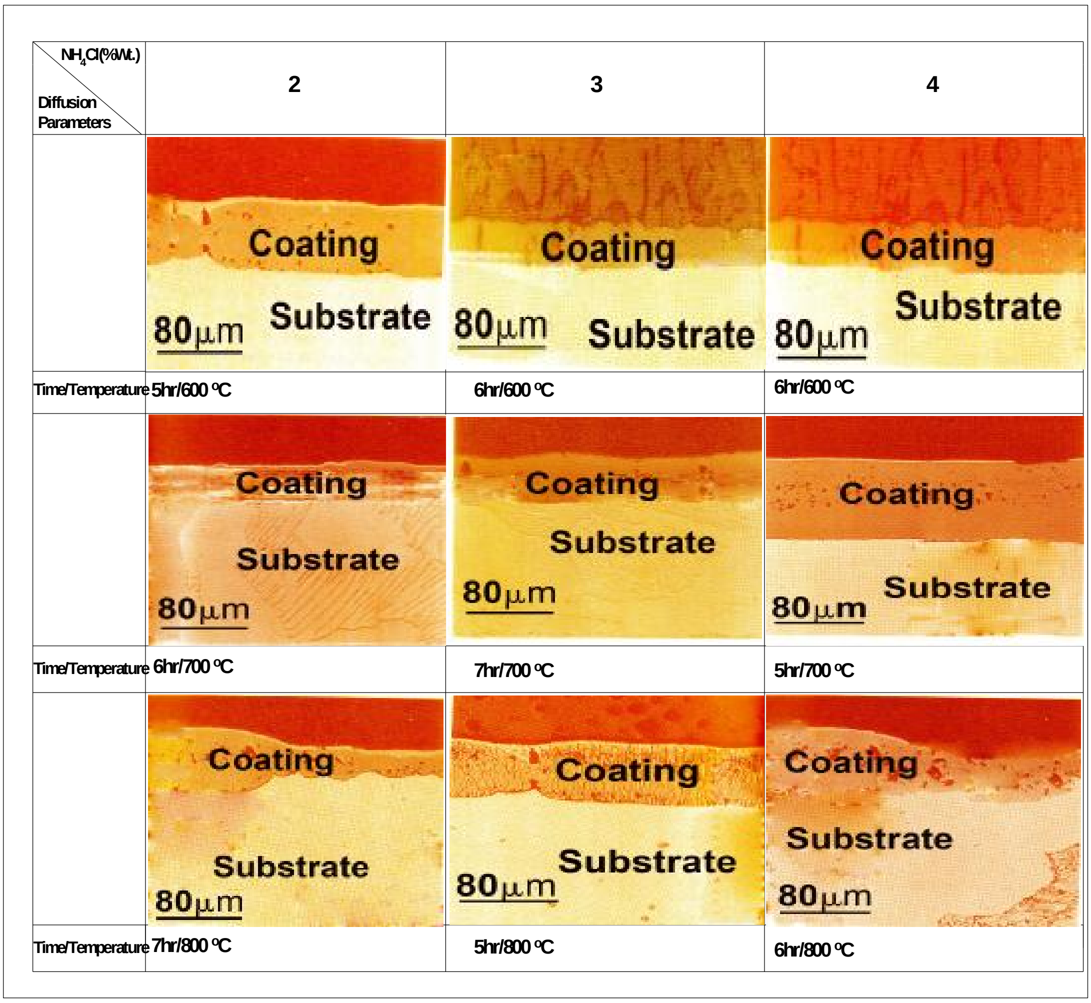

Figure 2 Microstrucures of aluminized specimens with varying process

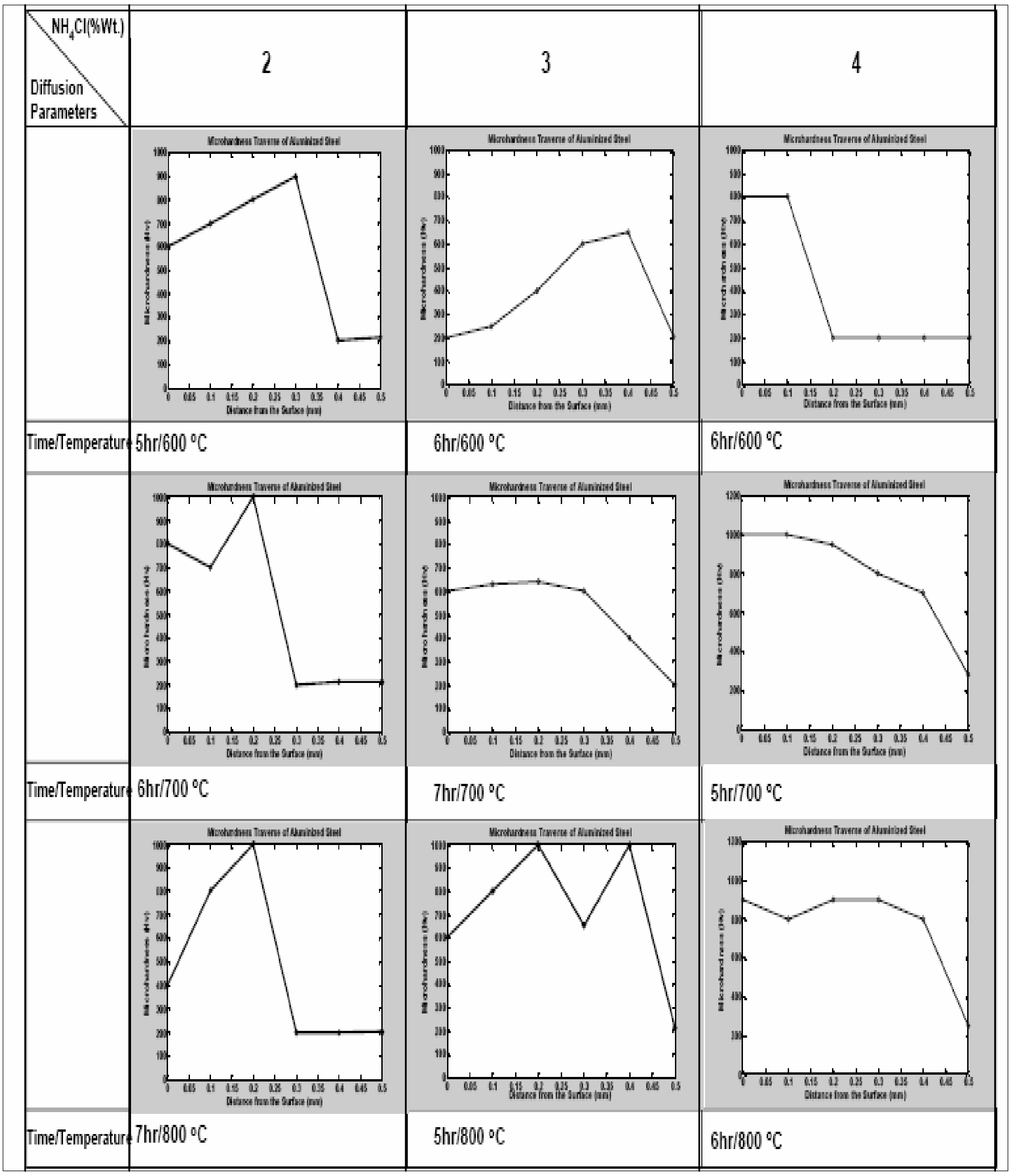

Figure 3 Microhardness traverses of aluminized specimens with varying process parameters

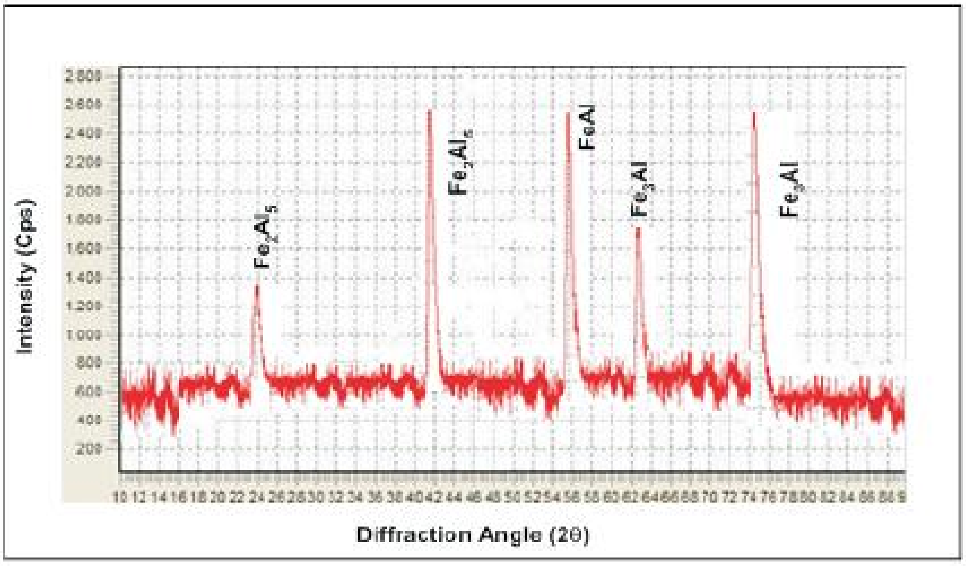

Figure 4 XRD characteristics of aluminized specimen using CuKα radiation.