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# Optimization of Cyclic Oxidation Parameters in Steel-T21 for Aluminization Coating Using Taguchi-ANOVA analysis by MINITAB13

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## Abstract

The increasing demands for high quality coatings has made it inevitable that the surface coating industry would put more effort into precisely controlling the coating process relative to media for which is subjected. Statistical design of experiments is an effective method for finding the optimum cyclic oxidation parameters for aluminization coating. In the present investigation, an attempt is made to produce high-quality aluminization coating by optimizing the cyclic oxidation parameters following a ($L9\text{-}3^3$) Taguchi-design approach. ($L9\text{-}3^3$) Taguchi orthogonal array has been used to determine the signal to noise ratio (S/N). The oxidation parameters that were varied include the Temperature (600,700,800$^o$C), Time (15,20,25hr at 5hr cycle) and Media (Air,$CO_2$,$H_2O$). The coating characteristics were qualitified with respect to parabolic oxidation rate constant ($K_P$). The performance of the coating was qualitatively evaluated using cyclic oxidation testing. Analysis of the experiments using Taguchi method indicated that 800$^o$C,25hr and CO2 media are to be the optimum cyclic oxidation conditions for pack aluminization. The contribution of each of these parameters to the parabolic oxidation rate constant ($K_P$) was determined employing an analysis of variance (ANOVA) and the effect of the level of each parameter was determined using Taguchi analysis. ANOVA results show that temperature and media are the parameters that most significantly affect the parabolic oxidation rate constant ($K_P$) compared to time.


**Keywords**:Taguchi Method,ANOVA, Cyclic Oxidation, Pack Cementation.

## تحديد عوامل الأكسدة الدورية المثلى لطلاء الألمنة في الفـــولاذ نـــوع -T21 باستخدام تحليل Taguchi-ANOVA بواسطة MINITAB13


**الخلاصة**

ان الطلب المتزايد حول الطلاء ذي النوعية العالية قد جعل صناعة الطلاء السطحي تبذل الجهـــود من أجل السيطرة على الطلاء بشكل دقيق نسبة الى الوسط الـــذي تتعـــرّض لـــه. يعتبـــر التـــصميم الاحصائي للتجارب طريقة فعّالة لايجاد عوامل الأكسدة الدورية المثلى لطلاء الألمنة. في هذا البحث، تم الحصول على طلاء الألمنة ذات النوعية العالية من خلال تحديد عوامل الأكسدة الدوريـــة المثلـــى طبقا لطريقة تصميم Taguchi ($L9\text{-}3^3$) . ان مصفوفة Taguchi ($L9\text{-}3^3$) قد تمّ استخدامها لتحديـــد نسبة الاشارة/الضوضاء (S/N) . ان معاملات الأكسد التي خضعت الى التغييـــر تتـــضمن، درجـــة


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الحرارة ($600,700,800^oC$) ، الزمن($15,20,25hr$ at $5hr$ cycle) و الوسـط($Air,CO_2,H_2O$) . ان خصائص نوعية الطلاء قد تم تحديدها نسبة الى ثابت معدل أكسدة القطع المكافئ ($K_P$) . و تم تقيـيم أداء الطلاء من خلال اختبارات الأكسدة الدورية. أظهرت نتائج تحليل التجـارب باسـتخدام طريقـة Taguchi ، أن عوامل الأكسدة الدورية المثلى لطـلاء الألمنـة هـي: ($800^oC,25hr$ , CO2) . ان مساهمة تأثير كل عامل على ثابت معدل أكسدة القطع المكافئ ($K_P$) قد تم تحديده باسـتخدام تحليـل المتغير (ANOVA) و تأثير كل مستوي من هذه العوامل قد تم تحديده باستخدام تحليـل Taguchi . أظهرت نتائج(ANOVA) بأن كل من درجة الحرارة و الوسط كان لها الأثر الأكبر على قيمة ثابـت معدل أكسدة القطع المكافئ ($K_P$) مقارنة مع الزمن.

## Introduction

Metal diffusion processes like pack-cementation are widely used for nickel-based and iron-based alloys to improve the oxidation resistance [1] . One of the major problems connected with the application of these alloys is their insufficient oxidation resistance, which can be improved by the usage of aluminide protective coatings [2]. Aluminized coatings have the best isothermal and cyclic oxidation resistance at high temperatures and were still protective under hot corrosion conditions [3]. Pack aluminizing is a process that has been used for iron and nickel-based and has been proven to be a very effective treatment for oxidation protection at high temperatures. The Fe-aluminides intermetallic coatings can substantially improve the high temperature oxidation and corrosion resistance of alloy steels through preferential oxidation of Al, which result in the formation of a stable $Al_2O_3$ scale that act as a protective barrier separating the underneath material from aggressive environment [4,5]. The nature and properties of the initially growth oxide scale gradually change and transform into a more protective scale, as is likely the case for the transformation of transient alumina into protective -alumina [6]. Many investigations in the oxidation community have suggested that -alumina scales form primarily by inward diffusion of oxygen whereas other oxides such as chromia scales, form primarily by outward diffusion of the metal [7].

In this study, the applicability of the aluminum pack cementation to enhance the oxidation resistance was examined in steel-T21 alloy over a wide range of temperatures according to Taguchi-orthogonal array ($L9\text{-}3^3$) under various testing conditions. Furthermore, the analysis of variance (ANOVA) is employed to investigate the most significant control factors.

## Purpose of Coating: Historical Development

The first public descriptions of pack cementation aluminizing were by Van Aller in a US patent field in 1911 and in a 1914 paper by Allison and Hawkins, all of the General Electric Research Laboratory(G.E.R.L.). Later, Gilson, also at G.E.R.L, patented the use of alumina as inert filler. Ruder summarized uses of aluminizing in 1915, including coating steel furnace fixtures and nickel combustion chambers. In about 1942 Anselm Franz in Germany used aluminized low alloy steels for combustion chambers, and possibly blades. In the post-war period, additional development and uses of the process up to the first practical use of pack cementation aluminizing of cobalt-base gas turbine vane airfoils in 1957 are not well documented [8].

The first aluminizing of nickel-base turbine blades may have been by hot dip processes at Allison and Curtiss Wright in about 1952. Pratt and Whitney introduced nickel-base blades aluminized by slurry-fusion process in about 1963. Since about 1970 most vane and blade coatings have been applied by pack cementation and more recently developed chemical vapor deposition processes [8].

Kelley described his invention of pack cementation chromizing of steels in 1923. One of the first uses was for the protection of steam turbine buckets. Samuel and Lockington published a comprehensive review of chromize coatings on steel in 1951-1952, and Drewett another in 1951. In 1953 Gibson patented aluminizing of chromized steel with aluminum paints and heat treatment to improve high-temperature oxidation resistance. Time of first use of chromizing of gas turbine airfoils is obscure it may have been in Europe for protection of industrial gas turbine airfoils in the early 1960s. From the 1970s on, developments in the field of diffusion coatings include modification of aluminide coatings with chromium, silicon, and platinum. In the 1990s, aluminide coatings have been recognized as useful bond coats for some types of thermal barrier coatings. Incorporation of so-called active elements in diffusion aluminide coatings to enhance adherence of protective alumina appears to have been successful until 1995. Recently, Liu and his colleagues at Oak Ridge National Laboratory (O.R.N.L.) have examined Cr-$Cr_2Nb$ leaves phase alloys as a possible advanced high temperature structural material [8,9] .

**Coating Classification**

Materials for high temperature applications are generally selected for specific properties such as strength, creep, mechanical and/or thermal fatigue. During their use at high temperatures these properties may degrade as a result of interactions with corrosive environments. One solution to avoid this alteration is to protect the component with a coating but coating must provide a significant and cost effective increase in substrate lifetime. Most high temperature coatings rely on the formation of a protective oxide scale by interaction with the environment. The role of this scale is to isolate the coating from the aggressive environment and thereby limit the degradations due to high temperatures corrosion. Obviously, this scale must fulfill several conditions: be stable, slow-growing, dense (pores cause a decrease in cyclic-oxidation), and adherent (often in cyclic oxidation). In practical applications only three oxides correspond to these criteria: alumina ($Al_2O_3$), chromia ($Cr_2O_3$), and silica ($SiO_2$), without these protective means, the selective dissolution of materials would be destroy the containment structure rapidly. There are essentially three types of high temperature coatings. The first type is an overlay coating in which material is deposited at the surface of the substrate. The second type is a thermal barrier coatings (TBCs) in which the substrate is insulated from the heat of the gas path because the TBCs system consist of a top ceramic coat and a metallic bond coat that is located between the substrate and the top coat. The third type is a diffusion (or conversion) coating in which the deposited mass is diffused and/or reacted with the substrate to form a somewhat continuous gradation in composition [10,11].

**Overlay Coating**

Pratt and Whitney initiated a program in the late 1960s to develop coatings with compositions nominally independent of substrates, and with capabilities for tailoring to the wide range of requirements of gas turbine applications. In 1964 an alloy with composition Fe-25%Cr-4%Al-1%Y emerged from nuclear programs. Pratt and Whitney research confirm that the alloy exhibited exceptional alumina adherence in cyclic oxidation. A model composition FeCrAlY, with 10-15 aluminum, was applied as a coating to nickel-base superalloys by electron beam physical vapor deposition (EBPVD)-thickness about 125µm. Tien and Petti came to similar conclusions from oxidation of FeCrAl alloys doped with yttrium and scandium. Latter work by Giggins and Pettit continued to show that the active elements minimize void formation at the scale metal interface. In 1986, Ashary et al. postulated that the active elements may assist in the formation of strong chemical bonds between oxides scales and the metal substrate. MCrAlY (M=Fe,Co,Ni) overlay coatings have been developed to protect hot section components in gas turbine engines against hot corrosion induced by salt deposit[8].

**Thermal Barrier Coatings (TBCs)**

G.W.Goward's first experience with thermal barrier coatings was with a system for protection of a TD-Nickel transition duct in a military engine in 1970. Some research has been performed on possible effects of hot corrosion on the ceramics used as thermal barriers, first by Barkalow and Petti and also by Jones et al.[8].

Hot Dipping Aluminzing

By the mid-1950s high-temperature coatings were being applied to hot section gas turbine aerofoils . Frem these beginnings has emerged a major worldwide industry in which fully automatic machines apply coatings to complex air-cooled, high-work, controlled structure aerofoils , flanges, marine turbines and stationary power generators.

**Diffusion Coatings**

In 1971, research by Goward and Boone provided a qualitative description of diffusion mechanisms for the formation of aluminide coatings on the on nickel-base superalloys. Diffusion coatings are formed as a result of interaction of two distinct process; the solute metal is brought into contact with the surface of the solvent, and this is followed diffusion proper which consists in the gradual absorption of the solute into the lattice of the solvent. The coating obtained is never a pure metal but a solid solution or a compound formed with the substrate metal [7, 8, 12]. The pack cementation process is commonly used to form diffusion coatings which improve the oxidation and erosion resistance of various heat resistance alloys at elevated temperatures. The addition of Cr, Si, or Al to the substrate surface changes the composition locally and improve the alloy resistance to corrosive environments. Chromizing, siliconizing, and aluminizing are the best document pack process, but other element such as titanium, zirconium, etc., can be transferred from a pack onto substrate.

Case Study: Coating of Tubes or large parts (substrate):

In this application gas-aluminizing is usually used . the driving force for the transport of the coating element is the difference in activity between the source and the substrate surface, where the activator react at high temperature with aluminum source to form aluminum halides . As the aluminum activity of the source is adjusted to be higher than that of the tube surface the aluminum halides are transport towards the tube surface, which become coated. Once on the surface these halides react with tube-alloy, releasing aluminum. A diffusion reaction occurs, with the subsequent formation of an aluminide activity, thereby lowering the aluminum activity on the tube surface, and the process continues [11].

The main advantages of pack cementation process as compared to other methods are as follows:

1) The process is simple to reproduce. Using the same set of easily controlled parameters (Temperature-Time schedule, Pack composition, etc.) the same coating can be produced on a given substrate. Coupons placed at predetermined locations in the retort provide an easy means of composition and thickness control.
2) The entire external surface of a component of complex shape can be uniformly coated. Cavities or holes ( e.g. cooling passages) can also be coated by filling the holes with reactive powders or by using processes with improved reactant gas transport.
3) This process is relatively inexpensive to run: several hundred parts can be coated at the same time, labour and capital investments are low compared with other processes [8].

**Taguchi-Analysis**

Taguchi methods have been performed recently. The statistical experimental design can determine the effect of the factors on the characteristic properties and the optimal levels of them. It uses the tables of the orthogonal arrays and analysis of variance (ANOVA) which can estimate the effect of a factor on the characteristic property [13]. 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 [14]. The numbers of all possible combinations corresponds to the number of needed experiments. Here, orthogonal arrays make it possible to carry out fractional factorial experiments in order to avoid numerous experimental works as well as to provide shortcuts for optimizing factors [15]. Orthogonal arrays were designed by Taguchi as a basis for experimental design. In this work, A model based on (L9-$3^3$) orthogonal array of Taguchi design was create to optimize the cyclic oxidation parameters.

Taguchi used the signal-to- noise ratio (S/N) as the quality characteristic of choice [16]. It is also, used the S/N ratio approach to convert the experimental results into a value for the evaluation characteristic in the optimum parameter analysis. The S/N ratio is quoted in decibel (db) as shown in the following equation [17]:

$$MSD = \frac{1}{N}\left(\sum_{i=1}^{N} Y_i^{\,2}\right)$$

Where MSD is the mean-square deviation for the quality characteristic. The smallest-the-better quality characteristic of Taguchi method has been used for analysis of experimental results. The MSD for the smallest-the-better is as shown in equation below [17]:

$$(S/N)Ratio = -10\log(MSD)$$

Where N is the sampling size and $Y_i$ is the response at each sampling point.

## ANOVA-Analysis

Analysis of variance (ANOVA) method has been applied to find out the significance of main factors and interaction factors [18]. ANOVA is performed to see statistically significant process parameters and percent contribution of these parameters on the characteristic properties. Larger F-value indicates that the variation of the process parameter make a big change on the performance characteristics [19].

All the statistical analysis of the data as well as the drawing of curves were made using MINITAB13.

## Experimental Procedure

The substrate material used in this work was steel-T21. 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 Material Department Engineering/University of Technology), and is given in Tables 1. The nominal chemical composition is given in Table 2. All surfaces, including the edges were wet ground using 320,600,800 and 120 grit silicon carbides papers. These samples 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. Pack powders were made using particle sizes below 60 m and are comprised of a powder mixture of aluminum of (20%wt.), halide activator ($NH_4Cl$) (2%wt.) and inert filler ($Al_2O_3$) (Balance). The pack-aluminum process was conducted at 650°C for 7hr under a flowing Ar environment according to Z.D.Xiang, P.K.Datta procedure[5]. It was decided to employ three different levels of three oxidation parameters (Temperature(°C),Time(hr) and Media) as listed in Table 3. Cyclic oxidation at high temperatures(600,700 and 800°C),different environments(Air,$H_2O$ and $CO_2$) and at different diffusion times (15,20 and 25hr at 5hr cycle) were conducted in order to study the oxidation resistance of steel-T21 with aluminide diffusion coating. The orthogonal array (L9-$3^3$) was used to study the influence of oxidation parameters against parabolic oxidation rate constant $K_P$ ($mg^2/cm^4$)/sec. The parameters involved and their 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 [14], since there were three of three levels factors, these factors were assigned to three columns in the (L9-$3^3$) array. During cyclic oxidation the furnace temperature was controlled within ±5°C by using Ni-Chrome thermocouple type-K. The evaluation of the oxidation resistance of the coating has been carried out by heating the samples in a furnace at test temperature and weighing them every 5hr, the samples were removed from the furnace, allowed to cool, ultrasonically cleaned in ethanol to detach the spalled oxide and then weight change per unit surface area are determined.

**Pack Cementation Process**

Substrate steel was placed in a sealed stainless steel cylindrical retort of 50mm in a diameter and of 80mm in a height in contact with the pack mixture. The retort was then put in another stainless steel cylindrical retort of 80mm in a diameter and 140mm in a height. The outer retort has a side tube through which argon gas passes and second in the top cover for argon gas outlet. Type-k calibrated thermocouple was inserted through the cover of the outer retort for recording real temperature near inner retort. This combined system then put in an electrical holding furnace under an argon atmosphere with a flow rate of 1.5 L/min. to avoid the oxidation of the underlying materials during the process. The argon atmosphere was maintained during all the diffusion coating process as well as cooling. The samples were then removed from the pack and ultrasonically cleaned for 30 minutes in ethanol to remove any loosely entrapped pack material on the surface, and weighed in order to determine the Al pickup. No further heat treatment was done to the samples after coating.

**Cyclic Oxidation**

Cyclic oxidation at high temperature and at different environments, of air, carbon dioxide ($Co_2$ gas), and water vapor ($H_2O$) were conducted in order to study the oxidation resistance of substrate. The evaluation of the oxidation resistance of the coatings has been carried out by heating the samples in a furnace at test temperatures and weighing them every 5hr. The samples were removed from the furnace, allowed to cool, ultrasonically cleaned in ethanol to detach the spalled oxide and the weight change per unit surface area was determined according to procedure indicated in [20].

**Oxidation in Dry Air**

Cyclic oxidation tests were carried out in a tube programmable furnace in the range 600-800$^oC$ in air at 1 atmosphere pressure. Each heating cycles includes heating in furnace for 5hr at the test temperature and cooling in still air. Samples of weight changing before and after each oxidation cycle were measured. Normally; at least 3 weight measurements were taken.

**Oxidation in $CO_2$ Gas**

Diffusion coating samples were oxidized in high purity $Co_2$ gas at 1 atmospheric pressure at a flow rate of 2 L/min. These samples were oxidized in the temperature of (600-800$^oC$) $\pm 3^oC$, for 100hr at 5hr cycles. Samples were then hanged by nickel-chrome wire through perforated stainless steel stand, and this stand was fixed inside stainless steel retort which has a side tube through which the $CO_2$ gas passes and the second hole in the cover of retort for the $CO_2$ gas outlet. Type K thermocouple was inserted through the cover of the retort for recording the temperature near the suspend samples.

**Oxidation in Water Vapor ($H_2O$)**

The experimental setup consists of carbolite tube furnace has a water vapor inlet, which permits a preheated vapor at a test temperature before it makes contact with the samples. The reaction chamber was first heated to 200$^oC$ in air. Water vapor generated in an evaporator was introduce thereafter with flow rate 0.7L/min. After purging the chamber with water vapor, the chamber was heated up to the desired temperature. After each oxidation cycle, the sample was allowed to cool to room temperature for interrupted weight measurements after each cycle.

**Optical Micrography**

For metallographic examination, the specimens were sectioned and mounted. To observe the microstructure of the surface oxide layer formed by cyclic oxidation tests. The cross-sections were mechanically polished using emery papers of grade 220-600 and final polishing was carried out using diamond paste. These samples were then cleaned and the microstructures of the oxide-layers were observed. Cross sections of oxide layer morphology were examined using light optical microscope (LOM) type CARLZEISS JENA, DDR).

**X-Ray Diffraction Inspection**

A Riga Ku X-ray generator with Cu-K radiation at 40KV and 20mA was used. The X-ray was generated by electric diffractometer, type philps (PW 1840), operating at scanning speed of 6$^o$ (2 ) per minute. The detector was moved through an angle of 2 =10 to 88 degrees. The XRD analysis was carried out at S.C. of Geological Survey and Mining.

**Results and Discussions**

For the analysis of data acquired through DoE, Taguchi method is applied for gathering required data by using an orthogonal array and investigating the S/N ratio ( Signal-to-Noise ratio) derived from data. In these experiments , “Smaller the Better Characteristics” were taken into account in order to determined optimum cyclic oxidation conditions for aluminide coating. The factors are assigned and the experiments are carried out. The results of nine trial conditions, with one run per trial condition are shown in Table 5. In this study, the lower values of parabolic oxidation rate constant $K_P$ $(mg^2/cm^4)$/sec. is desirable. Thus, it was categorized in the “ Smaller is Better” quality characteristic. All of the results were transformed into signal-to-noise ratio in the 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 corresponds to control factors. The points in the lower part of the graph (low S/N ratio) indicate superior conditions. Response curve can be used to find optimum level. Figure 1 shows the main effects plot for the S/N ratio, the main effect plot shown in this figure indicates that the lowest point is the optimum parameter for each factor. In short, (Temperature($^o$C), Time(hr),Media)=(800,25,$CO_2$) are at the optimum level. The details of average effect of S/N ratio is given in Table 6. optimum control parameters obtained from S/N ratio in each level are given in Table 7.

Different factors affect the oxidation resistance to different degrees. The relative effect of the different factors can be obtained by the decomposition of total variation into its appropriate components, which is commonly called analysis of variance (ANOVA). ANOVA is also needed for estimating the error variance. The results of ANOVA were shown in Table 8. The review of the “Percent” column in Table 8, showed that temperature-factor contributed the highest percentage (78.7634%) to the factor effects, followed by media-factor (13.6927%) and time-factor (7.5439%). Since the contribution of time was the smallest, it was considered insignificant compared to that of temperature and media. Thus, this factor was pooled with error-term. The new ANOVA after pooling was showing in Table 9. It was observed that as small factor affect [Time] was pooled, the percentage contributions of the remaining factors decreased slightly. But the ranking of factors effects still remained the same. In estimating the performance at optimum condition, all the factors were taken into account. As shown in Table 10, the contribution from these factors are (9.3313333),(2.761) and (3.94333333) for temperature, time and media respectively. Thus, the (Time) had little significance compared to the other factors.

Figure 2 illustrates typical microstructures of aluminized diffused steel specimen that form due to the pick up of aluminium by steel during aluminizing. Studies of oxidation kinetics provide valuable information about the oxidation mechanism. The kinetics of oxidation can be described examing the growth rate time constant or n value, which is found as the exponent of rate equation[21]:

Where ΔW is the weight change (mg), A is the sample surface area ($cm^2$), K is the rate constant, n is the growth rate time constant, and t is the time of oxidation (hr). For the parabolic kinetics, the rate equation takes the form:

Where k now refers to the parabolic rate constant. A plot of specific weight change *vs.* square root of time gives a linear-relationship as shown in Figure 3. The slope is the parabolic rate constant in units $(mg/cm^2)/hr^{1/2}$. The K value is then squared to give $K_P$ in units of $(mg^2/Cm^4)/hr$. as in the following expression:

$$(\Delta w / A)^2 = K_P t$$

The kinetics behavior of cyclic oxidation of coated system of Steel-T21 substrate at Taguchi-design oxidation conditions follow the parabolic behavior as shown in Figure 3. The point to be noted , is the $K_P$-value increases with increasing temperatures, these are in good agreement with Arrhenius-type relationship [21].

Effects of oxidation behaviors as well as the microstructural stability of high temperature oxidation is determined by combining the results of kinetics studies with extensive analytical investigation using light optical microscopy and X-ray diffraction (XRD). Figure 4 shows the oxidized surface features of aluminide coated system that subjected to cyclic oxidation tests according to Taguchi orthogonal array. A large number of voids exist at the oxidation conditions: ($600^oC$,15hr,Air), ($600^oC$,20hr,$CO_2$) and (600,25hr,$H_2O$). These voids may reduce the oxidation resistance of coated system. Little voids are observed near the alloy/scale interface at the oxidation conditions: ($800^oC$,20hr,Air), ($800^oC$,15hr, $H_2O$) and (800,25hr, $CO_2$). From the surface features of the samples ($700^oC$,15hr, $CO_2$), ($700^oC$,20hr, $H_2O$) and (700,25hr,Air) , the spalled areas are obviously, larger than that observed in above samples. The phase constitution of the oxidized aluminide coated system was determined using XRD. The major phase was found to be $Al_2O_3$. This is evident from Figure 5.

**Conclusions**

In this paper, a scheme based on the Taguchi-ANOVA method has been proposed to quickly identify the strategy important parameters affecting the oxidation resistance of pack aluminization of steel-T21 with hope the optimum parameters can be controlled to obtain the best oxidation resistance. Based on this study the following conclusions can be arrived at:

1. Based on the Taguchi approach, the cyclic oxidation of steel-T21 aluminide at cyclic oxidation conditions (800,25hr, $CO_2$), seems to be the optimum.
2. Through ANOVA analysis it is possible to recognize that the sequence of the most influential factors for oxidation resistance of aluminide steel-T21 corresponds to temperature,media and time.
3. Analysis of S/N ratio using ANOVA method has been finding out that the contribution where the percent contribution of Temperature ($^oC$) was (78.7634%), of media (13.6927%) and of time (7.5439%).
4. According to the contribution of each of optimum factor, indicated in Figure 9, it could be inferred that both Temperature(800$^oC$) and media($CO_2$) are the most predominant factor.
5. The XRD observations suggested that distribution of $Al_2O_3$ after the oxidation process dominantly affects the microstructure and oxidation resistance of the oxidized aluminide steel-T21.

**Reference**

[1]-Michihisa Fukumoto,"Coatings of Nb -based Alloy by Cr and /or Al Pack Cementations and Its Oxidation Behavior in Air at 1273-1473K", Materials Transaction,Vol.44,No.4,(2003),pp.73 1-735.

[2]-M.Goral, G.Moskal,Liswadzba, "The Influence of Si on Structure of Aluminide Coatings Deposited on TiAl alloy", Journal of Achievements in Materials and Manufacturing Engineering", Vol.18,issue 1-2,(2006), pp.463-466.

[3]-Ching-Yuang Bai,et.,Datta,., "Improvement of High Temperature Oxidation and Corrosion Resistance of Supperalloy IN-738LC by Pack Cementation", Surface and Coatings Technology 183(2004),pp.74-88.

[4]-R.Sakidja, F.Rioult, J.Werner and J.H. Perepezko, "Aluminum Pack Cementation of Mo-Si-B Alloys", Scripta Materialia,55(2006),pp.903-906.

[5]-Z.D.Xiang, P.K.Datta, "Pack Aluminization of Low Alloy Steels at Temperatures Below 700$^oC$" , Surface and Coatings Technology 184(2004),pp.108-115.

[6]-W.J.Q.uadda kkers,et. D.Waumenko,E.Wessed,V.Kouchy, L.Singheises. ,"Comments on Growth Rates of Alumina Scales on Fe-Cr-Al Alloys", Oxidation of Metals, Vol.64.Nos.5/6, (2005),pp.397-403.

[7]-J.A.Nychka and D.R.Clarke, "Quantification of Aluminum Outward Diffusion During Oxidation of FeCrAl Alloys", Oxidation of Metals,Vol.63,Nos.5/6,(2005),pp.325-352.

[8]- G.W.Goward, "Progress in Coatings for Gas Turbine Airfoil",Surface and Coating Technology, Vol.108-109,1998,pp.73-79.

[9]-Robert A.Rapp et al. "Oxidation-Resistant Ge-doped Silicide Coating on Cr-$Cr_2$Nb Alloy by Pack Cementation", Materials Science and Engineering, A222, 1997,pp.109-117.

[10]-M.Dust, P.Deb and D.H.Boone, "Structure and 700$^{o}$C Hot Corrosion Behavior of Chromium Modified Platinum-Aluminide Coatings", J.Vac.Sci.Technol.A4(6), Nov/Dec., 1986,pp.2571-2576.

[11]-National Research Council, "Coatings for High-Temperature Structure Materials: Trends and Opportunities", Committee on Coatings for High Temperatures Structural Materials, Washington, D.C.2000.

[12]-Donald R.Askeland and Pradeep P.Phule, "The Science and Engineering of Materials", Thomson, Brooks/Cole, 2003,p.85-178.

[13]-Mohsen, Jahanshahi,, "Applying The Taguchi Method for Optimizing Fabrication of Bovine Serum Albumin (BSA) Nanoparticles as Drug Delivery Vehicles", African Journal of Biotechnology, Vol.7(4),19 February,(2008), pp.362-367.

[14]-G.O.Verran, Chapman., "DoE Applied to Optimization of Aluminum Alloy Die Castings", Journal of Materials Progressing Technology 200, (2008),pp.120-125.

[15]-Dong-Woo Kim ,and A.Davide., "Application of Design of Experiment Method for Thrust Force Minimization in Step-Feed Microdrilling", Sensors,8,(2008),pp.211-221.

[16]-A.Jayant, Tanyildiz., "Perdition of Surface Roughness in CNC Turning Operation Using Taguchi Design of Experiments", Vol.88, March,(2008),pp.19-25.

[17]-Khairur Rijal Jamaludin, Coskun., "Analysis of Variance on the Metal Injection Molding Parameters Using a Bimodel Particle Size Distribution FeedStock", International Conference on Mechanical and Manufacturing Engineering (ICME2008), 21-23 May 2008,Johor Bahra, Malaysia.

[18]-S.Kumar, A.P. Statistical Analysis on Erosion Wear Behavior of A356 Alloy Reinforced with *in situ* Formed $TiB_2$ Particles", Materials Science and Engineering, A476,(2008), pp.333-340.

[19]-Zhizhong Sun, Henry HU, Xiang, Qigui, and Wenying Yang , " Gating System Design for Magnesium Alloy Casting", J.Mater.Sci.Technol., Vol.24, No.1,(2008), 93-95.

[20]- Ali.H.H., "Improvement of Inconel600 Oxidation Resistance by Aluminizing-Chromizing", Ph.D.Thesis, University of Technologh, Iraq, Jan, 2006.

[21]-A.A.Moosa, " Oxidation Properties of SteelT22 Alloy Coated by Simultaneous Ge-Doped Chromizing-Siliconizing Process", Eng.& Tech.,Vol.26,No.6,(2008),pp.669-709.

**Table(1) Spectrochemical analysis of low alloy steel T21**

| Element | Fe | C | Mn | P | $S_m$ | Si | Cr | Mo | V | Ti |
|---|---|---|---|---|---|---|---|---|---|---|
| Wt.% | Rem. | 0.04 | 0.34 | 0.014 | 0.012 | 0.35 | 2.96 | 0.10 | 0.002 | 0.01 |

**Table(2) Nominal composition of low alloy steel-T21ASTM(A200-94)**

| Element | Fe | C | Mn | $P_{max}$ | $S_{max}$ | Si | Cr | Mo |
|---|---|---|---|---|---|---|---|---|
| Wt.% | Rem. | 0.05-0.15 | 0.3-0.6 | 0.025 | 0.025 | 0.5max | 2.65-3.35 | 0.87-1.13 |

**Table 3 Design factors and their levels for orthogonal experiment.**

| Design Factors and Their Level for Orthogonal Experiment | | | | |
|---|---|---|---|---|
| Column | Design Factors for Orth. Exper. | Level for Orthogonal Experiment | | |
| | | 1 | 2 | 3 |
| 1 | Temperature (oC) | 600 | 700 | 800 |
| 2 | Time (hr) | 15 | 20 | 25 |
| 3 | Media | Air | CO2 | H2O |

**Table 4 L9 Orthogonal array**.

| L9-Orthogonal Array | | | |
|---|---|---|---|
| Run | Temperature(oC) | Time(hr) | Media |
| 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 | | |
|---|---|---|
| Run | Response(mg2/Cm4)/sec. | S/N Ratio(db) |
| 1 | 0.0000037 | 108.589 |
| 2 | 0.0000039 | 108.09 |
| 3 | 0.0000038 | 108.404 |
| 4 | 0.0000071 | 102.999 |
| 5 | 0.000005 | 106.09 |
| 6 | 0.0000049 | 106.232 |
| 7 | 0.0000115 | 98.786 |
| 8 | 0.0000156 | 96.138 |
| 9 | 0.0000685 | 83.286 |

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

**Response Table for Signal to Noise Ratios**

**Smaller is better**

| Level | Temperature(oC) | Time(hr) | Media |
|---|---|---|---|
| 1 | 108.361 | 103.458 | 103.653 |
| 2 | 105.107 | 103.439 | 98.125 |
| 3 | 92.737 | 99.307 | 104.427 |

**Table 7 Optimum parameters details.**

| Optimum Pararmeters Details | | |
|---|---|---|
| Parameters | Optimum Level | Value |
| Temperatur(oC) | 3 | 92.737 |
| Time(hr) | 3 | 99.307 |
| Media | 2 | 98.125 |

**Table 8 ANOVA-Table of control parameters.**

| ANOVA-Table | | | | | | |
|---|---|---|---|---|---|---|
| Column | Factors | DOF | Sum of Squares(SS) | Variance | F | Percent |
| 1 | Temperature(oC) | 2 | 407.677 | 203.8385 | — | 78.7634 |
| 2 | Time(hr) | 2 | 39.0468 | 19.5234 | — | 7.5439 |
| 3 | Media | 2 | 70.8731 | 35.4365 | — | 13.6927 |
| 4 | Error | 0 | — | — | — | — |
| 5 | Total | 6 | 517.5969 | 258.7984 | — | 100 |

**Table 9 Pooled-ANOVA Table of control parameters .**

| Pooled ANOVA-Table | | | | | | |
|---|---|---|---|---|---|---|
| Column | Factors | DOF | Sum of Squares(SS) | Variance | F | Percent |
| 1 | Temperature(oC) | 2 | 407.677 | 203.8385 | 10.44073 | 71.2195 |
| 2 | Time(hr) | {2} | {39.0468} | — | — | — |
| 3 | Media | 2 | 70.8731 | 35.4365 | 1.815078 | 6.1488 |
| 4 | Error | 0 | 39.0468 | 19.5234 | — | 22.6317 |
| 5 | Total | 6 | 478.5501 | 258.7984 | 12.25581 | 100 |

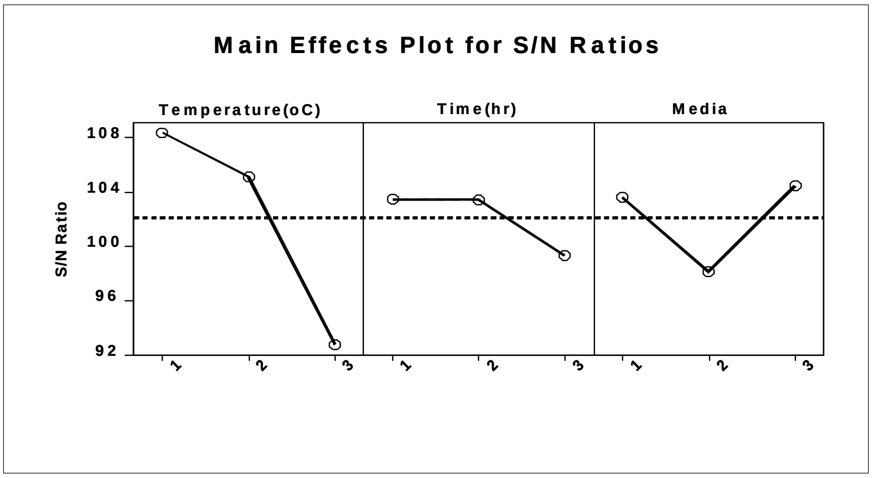


**Figure 1 Main effect plot of S/N ratio.**

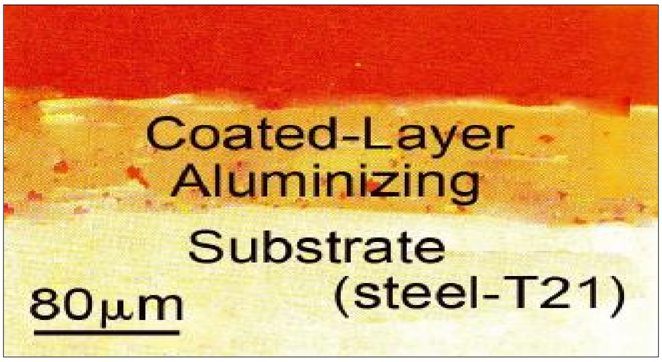


**Figure 2 Aluminized Steel-T21.**

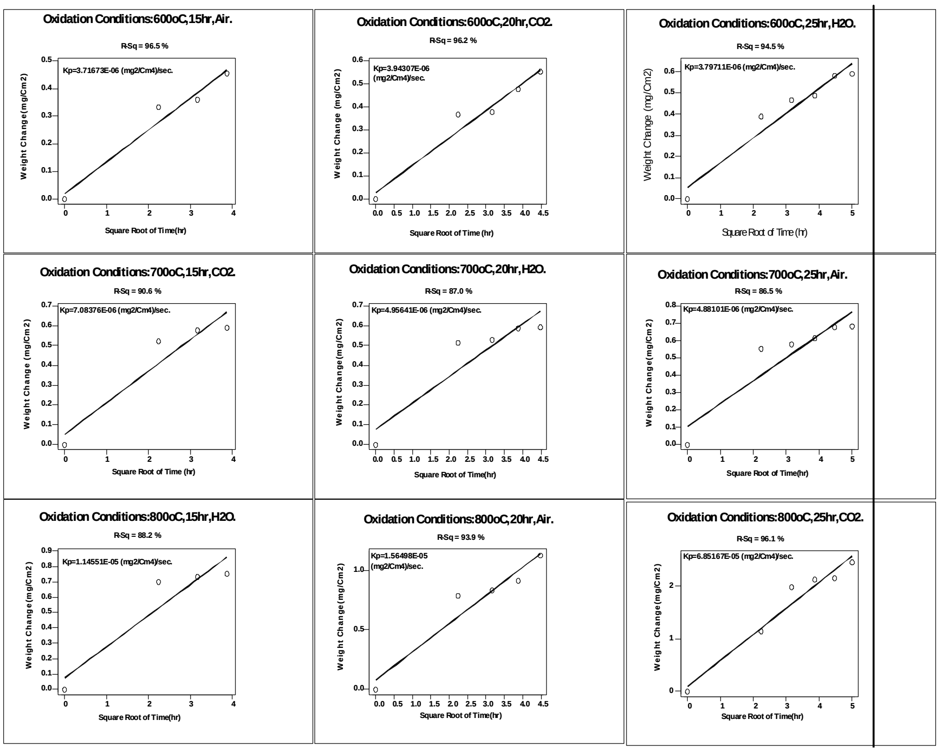


**Figure 3 Oxidation kinetics curves of aluminide steel-T21 according to Taguchi-Design.**

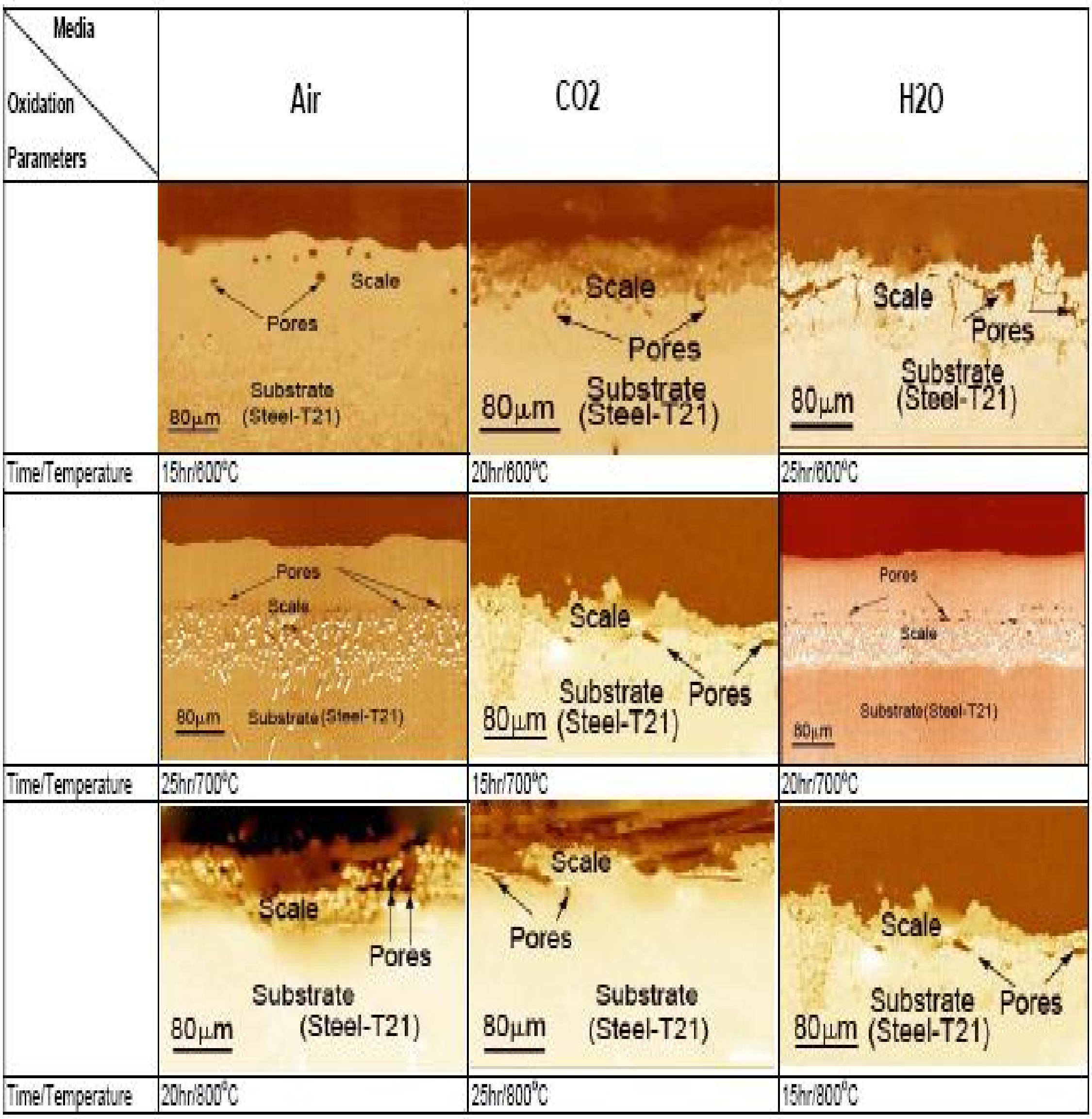


**Figure 4 Microstrucures of oxidized aluminized specimens with varying process parameters**.

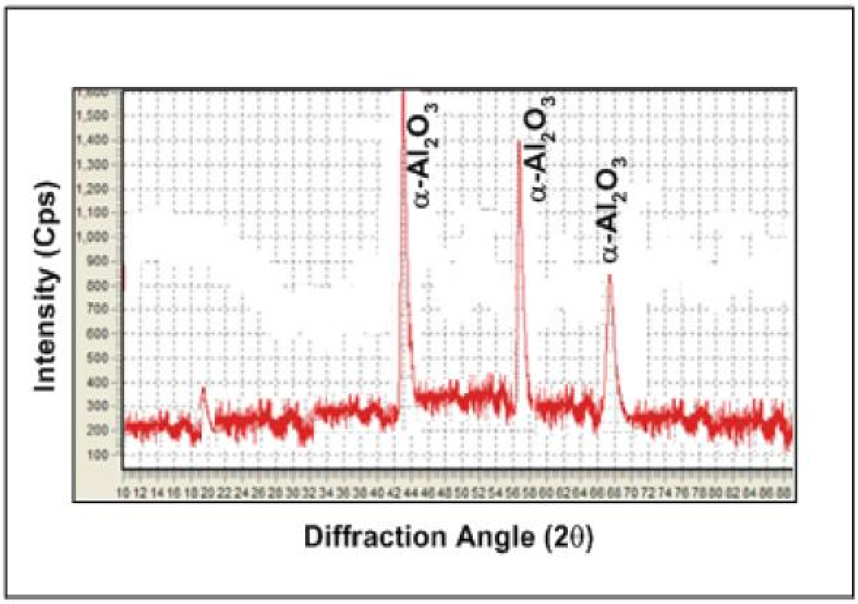


**Figure 5 XRD of oxidized-aluminized steel-T21**