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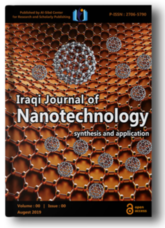

# Effect of Nano-coating on Molten Salts for Turbine Blades

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**Keywords:**
Grey Relational Analysis;
K417G;
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**Abstract**

The purpose of this study is to optimize hot corroded pack coated Ni-based superalloy K417G using grey relational analysis. Optimization of the pack cementation parameters was performed using quality characteristics of diffusion coatings for pack cementation process, i.e., salt activator, Nano-powders master alloy powder, and wt.% $Y_2O_3$. Analysis of variance (ANOVA) was used for observing the most influencing pack cementation parameters on the quality characteristics, i.e., $Na_2So_4$-6% wt. $V_2O_5$ (kp1), 100 wt% $NaSO_4$ (kp2), and 75 wt. % $NaSO_4$-25 wt % NaCl (kp3). The optimal process parameters were calculated using a grey relation grade and a confirmation test was performed. Based on the analysis of variance results, the wt.% $Y_2O_3$ is the most significant controllable diffusion coating factor for the hot corroded pack coated K417G at optimum setting conditions (A2, B3, C3) i.e., activator (NaCl), master alloy (94Cr-6Al), and wt.%$Y_2O_3$ (4%). according to the quality characteristics. Grey relational analysis was successfully applied to optimization of hot corroded pack coated K417G using multi-performance characteristics.

## Introduction

High-temperature coatings are used for protecting the high-temperature components from environmental attack due to oxidation and hot corrosion. These coatings have developed from simple aluminide coatings to complex overlay and duplex coatings. Aluminide coating was prepared using a pack cementation method. Pack cementation is one of the widely used surface coating technologies to economically improve high-temperature oxidation and corrosion resistance of components [1] . Recently, the surface operating temperature of the K417 G Ni-base superalloy used in turbine blades has increased to 1200 C° and even more. High temperature has a big potential to degrade the surface of the components resulting in oxidation or corrosion which can shorten the lifetime of the components [2]. Many efforts have been undertaken to overcome this severe problem, one of them is by applying the aluminizing or chromizing coating to prevent the oxidation process on the surface of the components [3].



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Aluminizing – chromizing diffusion coatings are widely used for high-temperature oxidation and hot corrosion protection of turbine blades used in engine hot sections [2]. The pack cementation method is used for the position of protective coatings on the protection against oxidation, corrosion, and damage [4] . At high temperatures, Al and Cr in the coating are oxidized and forms a thin Al2O3 and Cr2O3 scale, which works as the diffusion barrier and reduces the oxidizing speed of the base material. The coated elements are placed in the closed or half-closed containers and covered with mixture powder, which consists of metals used for deposition (Al and Cr), the halide activating agent, and inactive filler. The coating is fabricated through the reduction of metal-halide vapors on the surface of base material followed by diffusion in the solid-state between the introduced metal and the substance [5].

The good repeatability of the manufacturing process and low costs are the main advantages of the pack cementation method [6]. Generally, the pack cementation process can be classified into two types depending on its process temperature and the activity of deposition metal available in the pack. They are high-temperature low-activity and low-temperature high-activity[7]. Recently novel pack cementation coating with improved hot corrosion and oxidation resistance due to "reactive element effect"(REE)" produced using pack cementation method at low temperature on the substrate was developed [8]

In the present, the multi-objective methodology based-on the Taguchi approach and Grey Relational Analysis (GRA) has been used for the optimization of multiple parameters of coating for hot corroded of K417G alloy. The outcome of this study shall be used to explore the possible use of the developed coating for high-temperature components.

## Experimental procedure

The experimental work was performed by using a sample of Ni-based super alloy K417G. the Spectro-chemical analysis substrate material is reported in Table 1.

Table 1. Spectro-chemical analysis of K417G Alloy

| Ele. | Cr | Co | Mo | Si | Y | Ti | Al | Ni |
|---|---|---|---|---|---|---|---|---|
| Wt.% | 8.8 | 10.1 | 2.9 | - | - | 3.93 | 5.7 | Bal. |

Specimens with dimensions of approximately 20×20×5 mm were prepared. A hole of 2mm in diameter was drilled in each sample in order to hang the sample in the thermos-balance by means of a platinum wire. All surfaces, including the edges, were wet ground using 3201800 and 1200 grit silicon carbide papers.

These samples were then cleaned with water, degreased with acetone and then ultrasonically cleaned for 20 min using ethanol as a medium. After drying, the samples were stored in polyethylene Zip lock bags.

The pack powder mixture consisted of a Cr-Al master-alloy, halide salt activator (NH4Cl, NaCl and NaF) and aluminum powder as a filter. In selected packs, some alumina was replaced by Y2O3 Nano-powder (90 nm) which acted as a source of reactive yttrium (Y).

The sample was placed in a sealed stainless steel cylindrical retort of 50 mm in diameter and 80 mm in a height in contact with the packed mixture. The crucible was then put in another stainless-steel crucible of 80 mm in diameter and 140 mm



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in a height. The pack cementation process was conducted at 1150 C° for 7 hours in the pure Ar atmosphere. A schematic illustration of the coating process is shown in Figure 1.

Following the diffusion coating process, the samples were ultrasonically cleaned and cut perpendicular to the interface. Finally, the surfaces and cross-sections were examined by scanning electron microscope (SEM) and X-ray diffraction (XRD). To characterize the surface and cross-sectional morphology of the coatings at optimum setting conditions.

Cyclic hot corrosion studies were performed in molten salts (Na2So4, NaCl, and V2O5) for 20 cycles, each cycle consisted of 5 hours heating at 700 C° in a programmable tube furnace specimens were deposited with each of these salts until a total coating weight of 5 mg/cm3. Was reaching salt coated specimens were then kept in the oven for 4 hours at 100 C°. Then they were weighed again on digital balance before exposing to hot corrosion tests in a tube furnace.

The studies were conducted for all coated specimens. After testing the specimens were cleaned in an ultrasonic bath, first in distilled water, and then in ethanol. Then they were weighed on a digital balance to determine the weight change. The parabolic rate constants. (Kp) of hot corrosion are calculated by the linear least square algorithm of the following equation [9]:

$$(W/A)^2 = K_P t \quad (1)$$

Where (W/A) = weight gain per unit area (mg/cm$^2$), t=time of exposure (hour) and kp=parabolic rate constant (hot corrosion rate) (mg$^2$ cm$^{-4}$ S$^{-1}$). Many efforts were made to formulate the kinetics of hot corrosion- SEM and XRD techniques were used to analyze the hot corrosion products at optimum setting conditions.

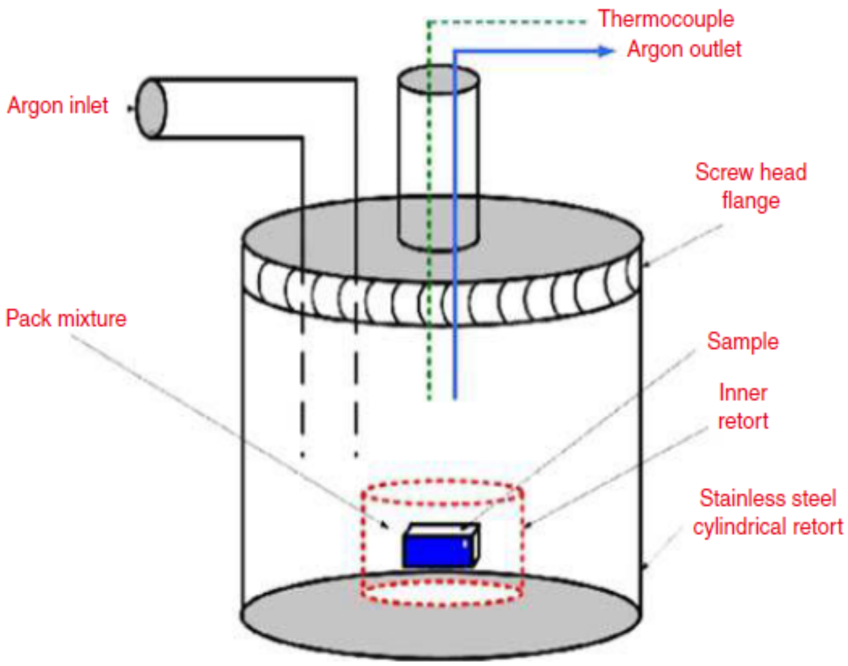


Figure 1. Pack Cementation Set-up



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Experiments of hot corrosion were conducted based on the design of experiments (DOE) using the Taguchi method of three levels, each level with three factors (L9-$3^3$) as shown in Table 2.

Table 2. Taguchi-Array L9

| RUN | Control factor and levels | | |
|---|---|---|---|
| | A | B | C |
| 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 |

Taguchi orthogonal array- the values are taken by a factor that is termed to be leveled. The Taguchi approach is a more effective method than the traditional design of experiment methods such as factorial design, which is resource and time-consuming. It is correct to point out also the limitations of the Taguchi method. The most critical drawback of the Taguchi method is that it does not account for higher-order interactions between design parameters. Only main effects and two-factor interactions are considered. Taguchi methods, developed by Dr.Genichi Taguchi, are based on the following two ideas[10]

1- Quality should be measured by the deviation from the specified target value rather than by conformance to present tolerance limits.
2- Quality cannot be ensured through inspection and rework but must be built-in through the appropriate design of the process and product

In the Taguchi method, two factors such as the control factor and the noise factor are considered to study the influence of output parameters. The controlling factors are used to select the best conditions for a process, whereas the noise factors denote all factors that cause variation.

The following three parameters were chosen for this study: halide salt activator (A), Nano-powders (70 nm) of the master alloy (B), and weight percent of yttria ($Y_2O_3$). The values or levels for the pack cementation parameters were determined according to the thermodynamics mechanism and kinetics of the position of elements in diffusion coatings by the pack cementation method [7]. The parameters and their levels used in the experiments are shown in Table 3.

Table 3. Parameters and their levels used in the experiments

| Symbol | Control Factor | Levels | | |
|---|---|---|---|---|
| | | 1 | 2 | 3 |
| A | Activator | NH4Cl | NaCl | NaF |
| B | Master Alloy | 90Cr-10Al | 92Cr-8Al | 94Cr-6Al |
| C | Wt.% $Y_2O_3$ | 1 | 2 | 4 |



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To evaluate the performance of the pack cementation process, the following output characteristics were selected: parabolic rate constants (Kp1) for hot corrosion coated Ni-based superalloy K417G in the molten salt environment, Na2So4-6% wt. V2O5 (kp1), 100 wt% NaSO4 (kp2), and 75 wt. % NaSO4-25 wt % NaCl (kp3). A simplified multi-characteristics methodology based on Taguchi's approach and grey relational analysis (GRA) has been used to optimize the performance of pack cementation coating.

## Results and discussion

Hot corrosion kinetics can be monitored using weigh change plots. The weight gain square (mg2/cm4) vs time (number of cycles) plots were plotted to establish the rate law for the hot corrosion. These graphs have been plotted to know the hot corrosion kinetics of specimens subjected to cyclic hot corrosion in the molten salt environment, Na2SO4, V2O5 and NaCl at 700 ºC. it is observed that the coating follows a nearly parabolic rate low. The parabolic rate (hot corrosion rate) constant kp was calculated by a linear least square algorithm to function in the form (W/A)2 = kpt. The values of parabolic rate constant kp are reported in Table 4. The lower the value of kp, the higher will be the hot corrosion resistance and vice versa.

Table 4. Parabolic rate constant (hot corrosion rate) kp ($10^{-10}$ $mg^2$ $cm^{-4}$ $s^{-1}$)

| Exp.No | $K_{P1}$ | $K_{P2}$ | $K_{P3}$ |
|---|---|---|---|
| 1 | 1.33 | 1.10 | 1.01 |
| 2 | 1.24 | 1.21 | 1.91 |
| 3 | 1.43 | 0.91 | 0.79 |
| 4 | 1.35 | 1.13 | 0.80 |
| 5 | 1.11 | 1.05 | 0.75 |
| 6 | 1.02 | 1.14 | 0.97 |
| 7 | 1.27 | 1.07 | 0.73 |
| 8 | 1.92 | 0.81 | 0.88 |
| 9 | 1.30 | 0.85 | 0.76 |

Grey relational analysis (GRA) is a decision-making technique based on grey. The theory which is originally developed by Deng Julong. According to grey theory, there are two kinds of data that can exist namely "known" and "unknown" in the experimental investigation. These data are called in the "Grey" theory as black and white data respectively.

The black and white represent unknown information, whereas white data represents known information. Between the white and black data, there is incomplete information exists in all experiments. This incomplete information is known as the Grey system [11] .

The range of each input and output factors and their respective units are different. Therefore, data must be normalized. The data processing transforms the original sequence into a comparable sequence. Hence, each response is normalized between 0 and 1. [12].

Some performance characteristics may need larger-the-better and some performance characteristics may need smaller-the-better. These two requirements use the following equations (2) and (3) respectively [13]:

$$X_i\ (k)= X_i(k)\text{-min } X_i(k) \ /\ \max X_i(k) - \min X_i(k) \qquad (2)$$

$$X_i\ (k)= \max X_i(k)\text{-}X_i(k) \ /\ \max X_i(k) - \min X_i(k) \qquad (3)$$



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Where, $X_i(k)$ and $X_i(k)$ are the sequences after the data processing and comparability sequence respectively, min $X_i(k)$ is the smallest value of $X_i(k)$ for the $k^{th}$ response and max $X_i(k)$ is the largest value of $X_i(k)$ for the $k^t$ response, k=1, 2, 3,… n.

$$\Delta_{oi}(k) = |X_o^*(k) - Xi^*(k) \quad (4)$$

$\Delta_{min}$ is the minimum deviation

$\Delta_{max}$ is the maximum deviation

In data processing, it is necessary to establish a relation between ideal and actual normalized experimental valves. It is done by calculating the grey relational coefficient which is obtained from the relation given below:

$$\xi_i(k) = \Delta min + \xi\, \Delta max / \Delta_{oi}(k) + \xi\, \Delta max \quad (5)$$

Where:

$\xi$ (k) = Grey relational coefficient

$\xi$ is distinguishing coefficient fall

The parameters are given equal preference is taken as 0.5.

After obtaining the grey relational Coefficient; the grey relational grade is computed by averaging the grey relational coefficient corresponding to each performance characteristic. The overall evaluation of the multiple performance characteristics is based on the grey relational grade [14]:

$$\gamma_i = 1/n\, \Sigma^n\, \xi_i(k) \quad (6)$$

where $\gamma_i$ is the grey relational grade for the $i^{th}$ experiment and n is the number of performance characteristics.

Based on the data sequence characteristics, data pre-processing have different methodologies pre started for the GRA [13].

Response or output can be converted into the comparative series according to Eq . (3) "smaller-the-better" characteristics.

Every one of the sequences of each performance characteristic after data processing utilizing Eq. (3) as shown in Table 5 normalization of performance characteristics.

Table 5. normalization of performance characteristics

| Ex.no | Kp1 | Kp2 | Kp3 |
|---|---|---|---|
| Reference sequence | 1.000 | 1.000 | 1.000 |
| 1 | 0.66 | 0.28 | 0.00 |
| 2 | 0.76 | 0.00 | 0.36 |
| 3 | 0.54 | 0.75 | 0.79 |
| 4 | 0.63 | 0.20 | 0.75 |
| 5 | 0.90 | 0.40 | 0.93 |
| 6 | 1.00 | 0.18 | 0.14 |
| 7 | 0.72 | 0.35 | 1.00 |
| 8 | 0.00 | 1.00 | 0.46 |
| 9 | 0.69 | 0.90 | 0.89 |



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Using Eq. (4) computed the deviation sequence, and the outcomes are clear in Table 6.

Table 6 deviation sequence and the outcomes of Kp values

| | Δoi (1) | Δoi (2) | Δoi (3) |
|---|---|---|---|
| | 1.000 | 7.000 | 7.000 |
| Exp.no | | | |
| 1 | 0.34 | 0.73 | 1.00 |
| 2 | 0.24 | 1.00 | 0.64 |
| 3 | 0.46 | 0.25 | 0.21 |
| 4 | 0.37 | 0.80 | 0.25 |
| 5 | 0.10 | 0.60 | 0.07 |
| 6 | 0.00 | 0.83 | 0.86 |
| 7 | 0.28 | 0.65 | 0.00 |
| 8 | 1.00 | 0.00 | 0.54 |
| 9 | 0.31 | 0.10 | 0.11 |

After data pre-processing is performed, GRC is determined from the normalized data to establish a relation between the preferred and real data. The distinguishing Coeff. (ξ) has been calculated using Eq. (5) as shown in Table 7:

Table 7. grey relation grade and its order in optimization

| Exp.no | ξ1 | ξ2 | ξ3 | GRG | Rank |
|---|---|---|---|---|---|
| 1 | 0.59 | 0.41 | 0.33 | 0.44 | 8 |
| 2 | 0.67 | 0.33 | 0.38 | 0.46 | 7 |
| 3 | 0.52 | 0.55 | 0.67 | 0.58 | 4 |
| 4 | 0.58 | 0.34 | 0.63 | 0.52 | 6 |
| 5 | 0.83 | 0.41 | 0.86 | 0.70 | 1 |
| 6 | 1.00 | 0.33 | 0.33 | 0.56 | 5 |
| 7 | 1.00 | 0.31 | 0.63 | 0.64 | 3 |
| 8 | 0.54 | 0.15 | 0.47 | 0.39 | 9 |



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| | | | | | |
|---|---|---|---|---|---|
| 9 | 0.58 | 1.00 | 0.43 | 0.67 | 2 |

Table 7 indicates that experiment 5 has the highest GRG. It is the mal among nine experiments optimal settings of pack cementation parameters for optimization based on corrosive salts is given in Table 8.

able 8. Response table for the Grey Relational Grade

| Factor | Halide salt activator | Powder of master alloy | Wt-% Yttria |
|---|---|---|---|
| Level | | | |
| 1 | 0.4948 | 0.5348 | 0.4624 |
| 2 | 0.5904 | 0.5155 | 0.5481 |
| 3 | 0.5662 | 0.6012 | 0.6410 |
| Rank | 2 | 3 | 3 |

So ($A_2B_3C_3$) as presented in Table 8 is the optimum parameter. The level with the maximum GRG is an optimal level of the process parameter. Analysis of variance (ANOVA) results for raw data of Grey relational results, is given in Table 9 It is seen that wt% $Y_2O_3$ (43%) significantly affects the hot corrosion rate as compared to halide salt activator (31%) and master alloy powder (26 %).

Table 9. ANOVA Table for raw data

| Parameter | DF | SS | MS | Contribution Ratio (%) |
|---|---|---|---|---|
| Halide salt activator | 2 | 0.01481 | 0.007403 | 31% |
| Master alloy powder | 2 | 0.01213 | 0.006063 | 26% |
| Wt.% Y2O3 | 2 | 0.04786 | 0.023930 | 43% |

Confirmation tests a final step recommended by Grey-relational-based Taguchi approach to verify the experiment conclusion. Estimated overall grey relational Grade is calculated as follows [15]:

$$\gamma_{opt} = \gamma_m + \Sigma (\gamma_i - \gamma_m) \qquad (7)$$

where $\gamma_m$ is the mean of total Grey-relational Grade, $\gamma_i$ is the mean of overall Grey relational grade at optimum level (A2, B3, C3), i the number of process parameter having significant Contribution in multiple performance characteristics. The summary results and comparison between experimental and predication results are shown in Table 10 . Figure 2 shows the cross-sectional and top-view morphology shows a thin oxide layer formed on surface which mainly contains (Cr, Al and



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Y), which are playing a significant role in a protection. According to XRD pattern , it is found that ($Cr_2O_3$, $Al_2O_3$ and $Y_2O_3$) oxides are mainly formed during hot corrosion and thus forms a protective oxide layer at the surface due to which further hot corrosion is prevented and the presence of yttria oxide layer in the scale improve the scale adhesion property and the scale formed are tightly adherent [16].

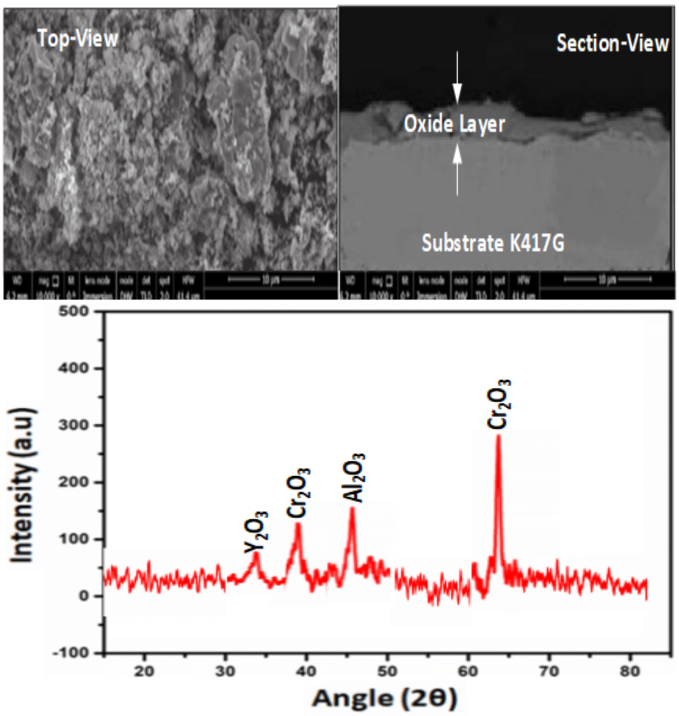


Figure 2. SEM/XRD Analysis for hot corroded pack coated K417G at optimum setting conditions

Table 10. Summary results of confirmation experiments

| method | level | Kp (10-10mg cm4 s-1) | |
|---|---|---|---|
| | | experimental | predication |
| Initial condition | 111 | 1.14 | 1.10 |
| Optimal condition | 233 | 0.51 | 0.552972 |

## Conclusion

The results acquiring from this study can be drawn as follows:

1. According to grey relational analysis, the optimum.
2. parameters with grade value of 0.7 (closest to 1 is optimum) were A2B3C3 i.e., Activator (NaCl), master alloy (94Cr-6Al) and wt.%$Y_2O_3$ (4%).
3. According to the ANOVA results, Wt.% $Y_2O_3$ (43%) is the most significant controllable as compared to halide salt activator (31%) and master alloy powder (26 %).
4. According to XRD pattern, it is found that ($Cr_2O_3$, $Al_2O_3$ and $Y_2O_3$) oxides are mainly formed during hot corrosion.



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