

Application of Response Surface Methodology for Modelling and Optimization of Hot Corrosion Rate of Nimonic 75 Coated by Ce-doped Aluminizing-Titanizing

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Abstract

Response surface methodology (RSM) was used to determine the optimum conditions (wt.% of Na_2SO_4 , %wt. of V_2O_5 , and Temperature) that give the minimum hot corrosion rate (K_p) ($\text{g}^2 \text{cm}^{-4} \text{s}^{-1}$) for Nimonic75 coated by Ce-doped Aluminizing- titanizing. Experiments were designed according to central composite design in response surface methodology with these three factors using MINITAB 16 and MATLAB 2014a Software. The variation of hot corrosion rate (K_p) with hot corrosion parameters was mathematically modeled using response surface methodology.

The optimum conditions obtained were 40 wt.% of Na_2SO_4 , 40 %wt. of V_2O_5 , and 900°C . This resulted in ($K_p=1.430987 \times 10^{-10} \text{g}^2 \text{cm}^{-4} \text{s}^{-1}$) as obtained from the predicted model, which fitted well with the laboratory verification result ($K_p=1.4311 \times 10^{-10} \text{g}^2 \text{cm}^{-4} \text{s}^{-1}$). This was supported by the high value of coefficient of determination ($R^2=99.81\%$) of the Predicted model. The high correlation coefficient ($R^2=98.991\%$) between the model and the experimental data show that the model was able to predict the hot corrosion rate from hot corrosion conditions.

Keywords: Response surface methodology (RSM), Hot corrosion, Diffusion coating, aluminizing, titanizing.

Introduction

Many important engineering systems operating at high temperature fail due to hot corrosion which is the main failure modes of components in the hot sections of gas turbines, boilers, and so forth. Hot corrosion is basically the result of attack by fuel and/or ash compounds of Na, V, S, and Cl that are present in the coal or in fuel oil used for combustion in the applications such as boilers, gas turbines, fluidized bed combustion, and industrial waste incinerators. In some situations, these impurities may be ingested from the service environment.

The operating temperatures in gas turbines are relatively high and are expected to increase further with the advances in materials

development and cooling schemes for the new generation gas turbine engines. The combination of such high temperatures with an aircraft environment that contains contaminants such as sodium, sulphur, vanadium, and various halides requires special attention to the phenomena of hot corrosion. No alloy is immune to hot corrosion attack indefinitely. Supper alloys such as Nimonic 75 have been developed for high temperature applications. However, these alloys may not be able to meet both high temperature strength requirements and high temperature corrosion resistance simultaneously for longer life. Super alloys find their largest application in the gas turbine industry, constituting over 50% of the gas turbine weight. Superalloys exposed to high temperature tend to suffer degradation due to hot corrosion during service. In utility gas turbine, contaminants in the fuel and air can cause serious hot corrosion problem. It is found that the corrosion involving a $\text{Na}_2\text{SO}_4\text{-V}_2\text{O}_5$ combination resulted in formation of $\text{NaV}_6\text{O}_{15}$ and NaV_3O_8 deposits. The molten salt was very corrosive and increased the acidic solubility of the protective oxides [1, 2,3].

One of the solutions to this problem is applying a thin layer of anticorrosion and antioxidation coating. So protective coatings are used to counter the latter. The diffusion coating processes have been used widely to deposit high-temperature oxidation and corrosion resistance coatings, such as aluminizing, titanizing and siliconizing. In the service environment the coatings are expected to form protective oxides such as Al_2O_3 , TiO_2 , SiO_2 . So the coatings are designed to serve as a reservoir for the elements forming or contributing to form these surface oxide. In general, Hot corrosion resistance of the materials used in the high-temperature regions can be improved by the application of protective coating since it alters the surface without affecting the bulk material properties [4,5].

The purpose of the present study was to investigate the effects of (wt.% of Na_2SO_4 , %wt. of V_2O_5 , and Temperature) on the hot corrosion rate (K_p) ($\text{g}^2 \text{cm}^{-4} \text{s}^{-1}$) of Nimonic75 coated by Ce-doped aluminizing- titanizing using response surface methodology and to develop a mathematical model to optimize three important parameters: wt.% of Na_2SO_4 , %wt. of V_2O_5 , and Temperature .

Experimental Procedure

Coating System

The experimental work was performed by using samples of Nimonic75. The spectrochemical analysis of candidate material is shown in Table 1 samples were cut into squares shapes with dimensions (20mm×20×mm×5mm) with small hole of 2mm diameter was drilled in each sample for holding. All surfaces , including the edges were wet ground using 120, 220, 320, 600, 800, and 1200 grit silicon carbide 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 bags. The dimensions of all samples were measured. The pack mixture used for aluminum-titanium diffusion coating consisting of 16 Wt.%Al powder (50-60 μm in particule size) as an aluminum source, 6 Wt.%Ti powder (70-80 μm in particule size) as a titanium source, 2Wt.% NaF and 2Wt.%NaCl as activator and the balance was alumina-powder (70-120 μm in particule size). All pack powders was sized by sieving method and 1Wt.% of the pack silica filler was replaced by cerium (Ce) .

The sample 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. Figure 1 shows the apparatus used for pack cementation (University of Technology / Department of Production Engineering & Metallurgy). Pack cementation process was carried out at 1050 °C for 6 h under an Ar atmosphere. After coating, the samples were ultrasonically cleaned , and weighed. It was found that the diffusion coating time of 6 h at 1050 °C give a coating thickness of 65-66 μm using QuaNix® 1500 coating thickness gauge (University of Technology /Materials

Engineering Department) . Figure 2 illustrates the experimental setup used for coating process.

Hot Corrosion Test

For hot corrosion tests, Na_2SO_4 and V_2O_5 powders were selected as a corrosive salts. Samples were deposited with each of these salts until a total coating weight of 5 mg/cm^2 was reached according to A.Anderson et. al procedure [6] .The samples were measured and weighed first , then placed on a hot plated heated to 110°C. An air gun sprayed on the saturated aqueous –salt solutions in air mist and a coat of fine salt particles formed on the samples surfaces after the mist settled and the water evaporated. The process was repeated until the dry particles were deposited up to 5 mg/cm^2 . Hot corrosion test was performed in a static air at (700-900°C) for 50 hr at 5 hr cycle in a programmable tube furnace .The experimental setup is shown in Figure 3 (University of Technology / Department of Production Engineering & Metallurgy). After testing the samples were cleaned in an ultrasonic bath, first in distilled water and then in ethanol. They were then weighed on a digital balance to determine the change in weight.

The parabolic rate of hot corrosion , K_p , was calculated by linear least-square algorithm to a function in the form of [5] :

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

Where W/A is the weight gain per unit surface area (mg/cm^2) and t indicates the number of cycles representing the time of exposure.

Experimental design and analysis

Basically, an optimization process involves three major steps, which are (1) performing the statistically designed experiments, (2) estimating the coefficients in a mathematical model, and (3) predicting the response and checking the adequacy of the model, as with the set up in this experiment [7] .

Central composite design (CCD) has been applied in this work to study the design of the hot corrosion experiments. CCD has been widely used for fitting a second-order model from experimental runs. The design consists of a $2n$ factorial or fraction (coded to the usual ± 1 notation) augmented by $2n$ axial points ($\pm\alpha$, 0, 0, ..., 0), (0, $\pm\alpha$, 0, ..., 0), ..., (0, 0, ..., $\pm\alpha$), and n_c center points (0, 0, 0, ..., 0). In this case, the main effects and interactions may be estimated by fractional factorial designs running only a minimum number of experiments. The responses and the corresponding parameters were modeled and optimized using analysis of

variance (ANOVA) to estimate the statistical parameters by means of response surface methodology (RSM). If all variables are assumed to be measurable, the response surface can be expressed, in Equation 2, as follows [8]:

$$Y = f(X_1, X_2, X_3, X_4, \dots, X_n) \quad \dots (2)$$

Where Y is the response of the system and X_i is the variables of action called factors. The goal of the RSM is to optimize the response variable (Y) and search for a suitable approximation of the functional relationship between the independent variables and the response surface. Second degree quadratic equation as given by Equation 3 was used for model formation. Applying the relationships in Table 2, the values of the codes were calculated and shown in Table 3, Where X_{max} and X_{min} are maximum and minimum values of X, respectively, β is 2^{n/4}, n= number of variables [8]. (in this study; β=2^{3/4}=1.682):

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \left(\sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} x_i x_j \right) \quad \dots (3)$$

where Y is the predicted response evaluated, x_i and x_j are the variables, β₀ is the constant coefficient, β_i, β_{ii} and β_{ij} are the inter-action coefficients of linear, quadratic, and the second order terms, respectively, and k is the number of studied factors.

For statistical analysis, the experimental variables X_i have been coded as x_i according to the following Equation 4.

$$x_i = \frac{(X_i - X_n)}{\Delta X_i} \quad \dots (4)$$

where x_i is the coded value (dimensionless) of the ith independent variable, X_i is the uncoded value of the ith independent variable, is the X_i at the center point, and ΔX_i is the step change value of the real variable i. From Equation 4, the coded unit can be converted to the uncoded unit. In the CCD the values of +/- alpha are the low and high values of the variables. For three variables, the design in coded and uncoded form is shown in Table 2.

The quality of the fit of the polynomial model was expressed by the value of correlation coefficient (R²) [9]. The experimental plan was generated using the MINITAB16 & MATLAB2014a. Finally, the optimum values

for maximizing the amount of the studied responses were determined using the same software.

Results and Discussion

ANOVA analysis and quadratic model

The statistical software package ‘MINITAB16’ has been used for regression analysis of the experimental data and to draw the response surface plot. ANOVA was used to estimate the statistical characteristics of the model fitting. The complete experimental design and results consisting of coded levels, actual variables, and responses are given in table 4 (MINITAB Worksheet), where the parabolic rate of hot corrosion for coated system in molten salts under 700-900°C were calculated on the basis of 5 hr cycle data. in order to ensure a good model, a test for significance of the regression model and individual model coefficients was needed to be performed accompanying with the lack-of-fit test. Normally, the significant factors can be ranked based on the F-value or p-value (also named ‘Prob. >F’ value). The larger the magnitude of the F-value and correspondingly the smaller the ‘Prob. > F’ value, the more significant is the corresponding coefficient [10].

Table 4 Central composite design consisting of experiments for the study of experimental factors in coded and actual values with responses from observed and predicted results.

As there are many insignificant model terms from the full second quadratic model, they can be sorted out and then an improved model could be obtained. Thus, with the MINITAB16 program, the stepwise elimination procedure was selected to automatically eliminate the insignificant terms. The resulting ANOVA data for the reduced quadratic model of total flux are given in MINITAB session shown in Figure 4. By applying multiple regression analysis on the experimental data, the reduced quadratic equation in terms of code factors was obtained, as shown in MINITAB session in Figure 5, where PRESS=Predicted residual sum of squares (SS) and DF= Degree of freedom, where K_p is the hot corrosion rate. X₁, X₂, and X₃ are the wt.% of Na₂SO₄, %wt. of V₂O₅, and Temperature, respectively, detailed in Table 3. From Figure 4 and 5, significant terms for the response surface model can be determined. Firstly, the linear terms of the wt.% of Na₂SO₄ (X₁), %wt. of V₂O₅ (X₂), and the Temperature (X₃) have different effects on K_p. According to ANOVA Table, was found to be the major factor affecting the hot corrosion rate (K_p), whereas %wt. of V₂O₅ was found to be the second factor and wt.% of Na₂SO₄ the third

factor . The second order term of Temperature (X_3^2) and the interaction of X_1 and X_2 are also significant terms in the model. Ranking of these significant terms is as follows, with $X_3 > X_2 > X_1 > X_1X_2 > X_3^2 > X_1^2 > X_2X_3 > X_1X_3 > X_2^2$.

The predicted R^2 of 98.61% is in reasonable agreement with the adjusted R^2 of 99.64% . The application of the response surface methodology yields, on the basis of parameter estimates, an empirical relationship between the response variables (the hot corrosion rate K_p) and the test variables. These are related to the following quadratic expression in code unit, and after substituting reduced quadratic model to the refined model, the final model in terms of natural variables (where K_p is the hot corrosion rate. X_1 , X_2 , and X_3 are the wt.% of Na_2SO_4 , %wt. of V_2O_5 , and Temperature, respectively) are obtained, as represented below in MINITAB session in Figure 6 .

The quadratic equation obtained with multiple variables can be used to predict the K_p within the limits of the experimental factors. Figure 7 reveals that the predicted response values of the reduced quadratic model are well in agreement with the actual ones in the range of the operating variables.

Combined effect of operating parameters on the response

In order to visualize the relationship between the experimental variables and the response, and to study individual and interaction effects of the three factors consisting of the wt.% of Na_2SO_4 , %wt. of V_2O_5 , and temperature on the hot corrosion rate, response surfaces and contour plots were generated from the final model, as shown in Figure 8, and the contours were plotted in the x-y plane by a projection of the response surface . These figures illustrate the response of different experimental variables and can be used to identify the major interactions between the variables. Each contour curve represents an infinite number of combinations of two test variables. Other factors are kept each time at their respective zero levels . A careful observation of the ANOVA results reveals that the wt.% of Na_2SO_4 , %wt. of V_2O_5 , and temperature have affected the response of the hot corrosion rate . However, the temperature imposes the greatest effect while the wt.% of Na_2SO_4 imposes the least.

Process optimization using response surface methodology

The optimum conditions of (wt.% of Na_2SO_4 , %wt. of V_2O_5 , and Temperature) that give the minimum hot corrosion rate (K_p) ($\text{g}^2 \text{cm}^{-4} \text{s}^{-1}$) for Nimonic75 coated by Ce-doped

Aluminizing- titanizing was obtained using numerical optimization feature of the MATLAB2014a software . The program searches for a combination of factor levels that simultaneously satisfy the requirements placed on each of the responses and factors. The optimum values of the selected variables were obtained by solving the regression equation . The optimum values of input variables from regression equation of for the hot corrosion rate (K_p) were shown in Table 5 . Confirmation experiment was used to verify the optimal combination of the factor settings . Therefore , confirmation experiment was performed using optimal conditions i.e. 40 wt.% of Na_2SO_4 , 40 %wt. of V_2O_5 , and 900°C for the hot corrosion rate (K_p) . The predicted and the experimentally observed values of the hot corrosion rate (K_p) at the optimum conditions found to be in good agreement as is seen from the in Table 5 . It shows the adequacy of the response surface methodology in the prediction of the hot corrosion rate (K_p) ($\text{g}^2 \text{cm}^{-4} \text{s}^{-1}$) for Nimonic75 coated by Ce-doped Aluminizing- titanizing .

Conclusion

From studies quoted above, we come to a conclusion that:

1. The central composite design and response surface methodology enabled the determination of optimal of (wt.% of Na_2SO_4 , %wt. of V_2O_5 , and Temperature) that give the minimum hot corrosion rate (K_p) ($\text{g}^2 \text{cm}^{-4} \text{s}^{-1}$) for Nimonic75 coated by Ce-doped Aluminizing- titanizing .
2. The multiple correlation coefficient of determination R^2 obtained was 0.9981, inferring that the actual data fit quite well with the predicted data applying the quadratic model.
3. The optimum conditions obtained were 40 wt.% of Na_2SO_4 , 40 %wt. of V_2O_5 , and 900°C . By applying these parameter values, the minimal hot corrosion rate (K_p) has been predicted and confirmed experimentally, i.e. $K_p=1.430987 \times 10^{-10} \text{ g}^2 \text{cm}^{-4} \text{ s}^{-1}$ as obtained from the predicted model , which fitted well with the laboratory verification result ($K_p=1.4311 \times 10^{-10} \text{ g}^2 \text{cm}^{-4} \text{ s}^{-1}$).

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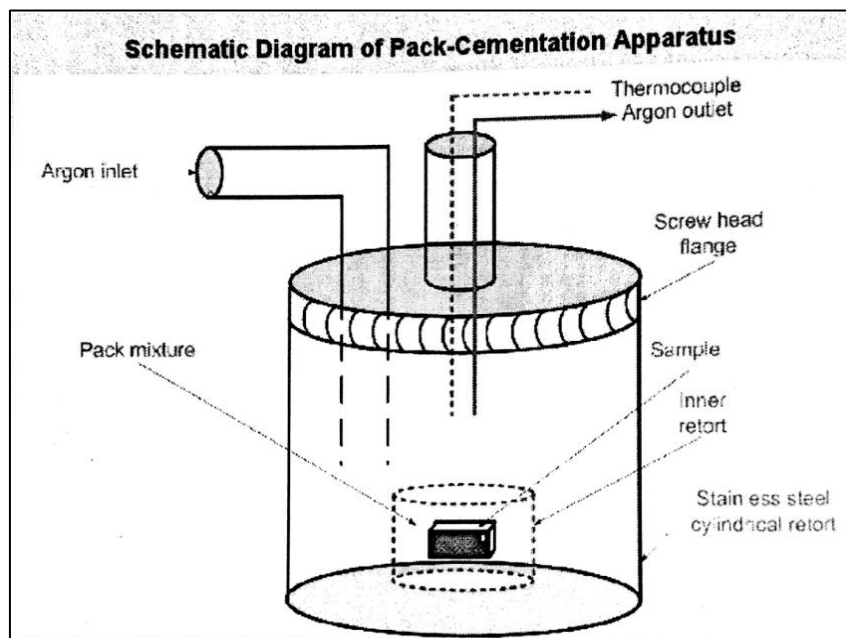


Figure 1a: Schematic diagram of pack cementation apparatus

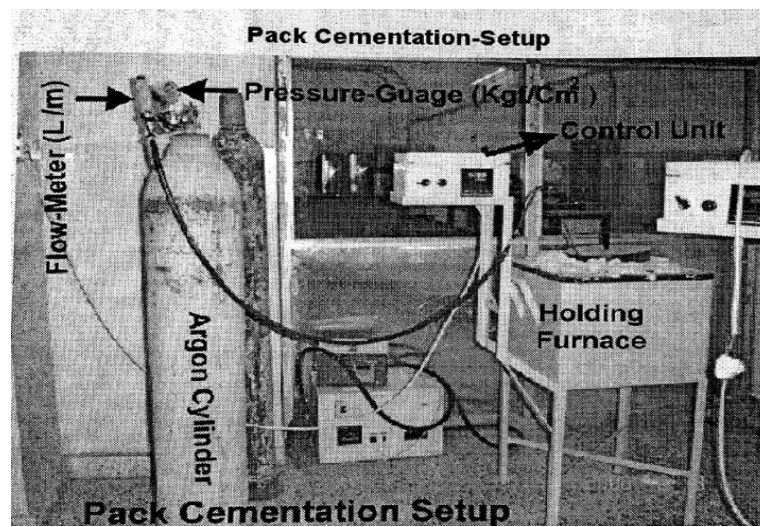


Figure 1b: pack cementation setup

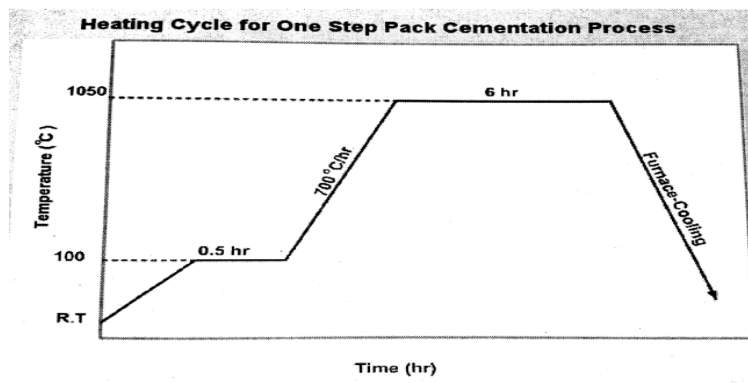


Figure 2: Heating cycle for coating process

Table 1: Chemical composition of substrate material

Element	C	Si	Cu	Fe	Mn	Ti	Al	Cr	Ni	Mo
Wt.%	0.10	0.01	0.02	3.1	0.07	0.33	0.01	19.34	bal	0.03

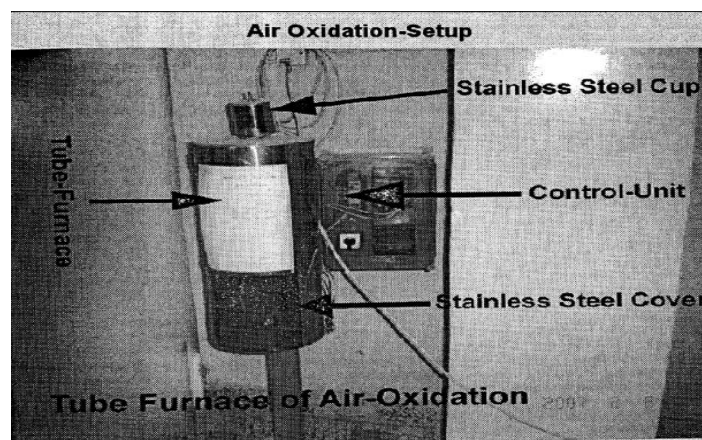


Figure 3: Air oxidation setup for hot corrosion test

Table 2: Relation between coded value and level of the variable [8]

Coded value	Level of variable
$-\alpha$	X_{min}
-1	$[(X_{max} + X_{min})/2] - [(X_{max} - X_{min})/2\beta]$
0	$(X_{max} + X_{min})/2$
+1	$[(X_{max} + X_{min})/2] + [(X_{max} - X_{min})/2\beta]$
$+\alpha$	X_{max}

Table 3: Independent variable and their levels for CCD experimental design
Coded variable levels

Independent Variables	Symbol	Coded variable levels				
		$-\alpha$	-1	0	+1	$+\alpha$
wt.% of Na_2SO_4	X_1	40	44.055	50	55.055	60
wt. % of V_2O_5	X_2	40	44.055	50	55.055	60
Temperature($^{\circ}C$)	X_3	700	740.547	800	859.453	900

Table 4: Central composite design consisting of experiments for the study of experimental factors in coded and actual values with responses from observed and predicted results

Run	Coded Values			Actual values			Response values (observed)		Response values (predicted)
	X_1	X_2	X_3	wt.% of Na_2SO_4	%wt. of V_2O_5	Temperature	hot corrosion rate (K_p) ($g^2 cm^{-4} s^{-1}$)	hot corrosion rate (K_p) ($g^2 cm^{-4} s^{-1}$)	
1	α	0	0	60	50	800	4.85	4.728078508	
2	1	-1	1	55.055	44.055	859.453	1.94	2.055063481	
3	0	0	0	50	50	800	4.05	4.067447243	
4	0	0	0	50	50	800	4.07	4.067447243	
5	0	0	$-\alpha$	50	50	700	3.27	3.209072931	
6	0	0	0	50	50	800	4	4.067447243	
7	0	0	0	50	50	800	4.08	4.067447243	
8	0	0	α	50	50	900	4.52	4.378777078	
9	-1	-1	1	44.055	44.055	859.453	1.28	1.428283737	
10	1	1	-1	55.055	55.055	740.547	6.55	6.544657892	
11	1	1	1	55.055	55.055	859.453	7.21	7.28916814	
12	-1	-1	-1	44.055	44.055	740.547	0.718	0.78177349	
13	0	$-\alpha$	0	50	40	800	0.26	0.055885748	
14	0	α	0	50	60	800	8.03	8.031964261	
15	0	0	0	50	50	800	4.1	4.067447243	
16	-1	1	-1	44.055	55.055	740.547	5.005	5.032878148	
17	-1	1	1	44.055	55.055	859.453	6.12	6.130888396	
18	0	0	0	50	50	800	4.07	4.067447243	
19	$-\alpha$	0	0	40	50	800	3.01	2.929771501	
20	1	-1	-1	55.055	44.055	740.547	1.63	1.762053234	

Analysis of Variance						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	82.8714	82.8714	9.20793	587.869	0.000000
X1	1	3.9037	0.1113	0.11126	7.103	0.023685
X2	1	76.7935	0.0434	0.04339	2.770	0.127019
X3	1	1.6516	0.1304	0.13044	8.328	0.016224
X1*X1	1	0.0822	0.1025	0.10249	6.543	0.028472
X2*X2	1	0.0000	0.0010	0.00100	0.064	0.805951
X3*X3	1	0.1348	0.1348	0.13477	8.604	0.014956
X1*X2	1	0.1412	0.1412	0.14125	9.018	0.013277
X1*X3	1	0.0625	0.0625	0.06248	3.989	0.073724
X2*X3	1	0.1019	0.1019	0.10193	6.507	0.028817
Error	10	0.1566	0.1566	0.01566		
Lack-of-Fit	5	0.1507	0.1507	0.03015	25.623	0.001426
Pure Error	5	0.0059	0.0059	0.00118		
Total	19	83.0280				

Figure 4: Minitab session shows ANOVA Table for reduced quadratic model

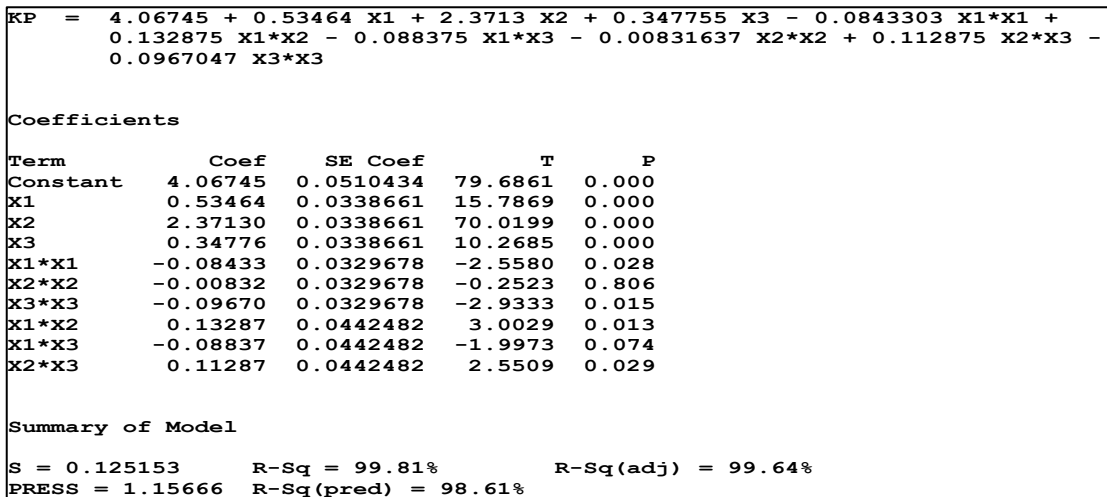


Figure 5: Minitab session shows reduced quadratic model

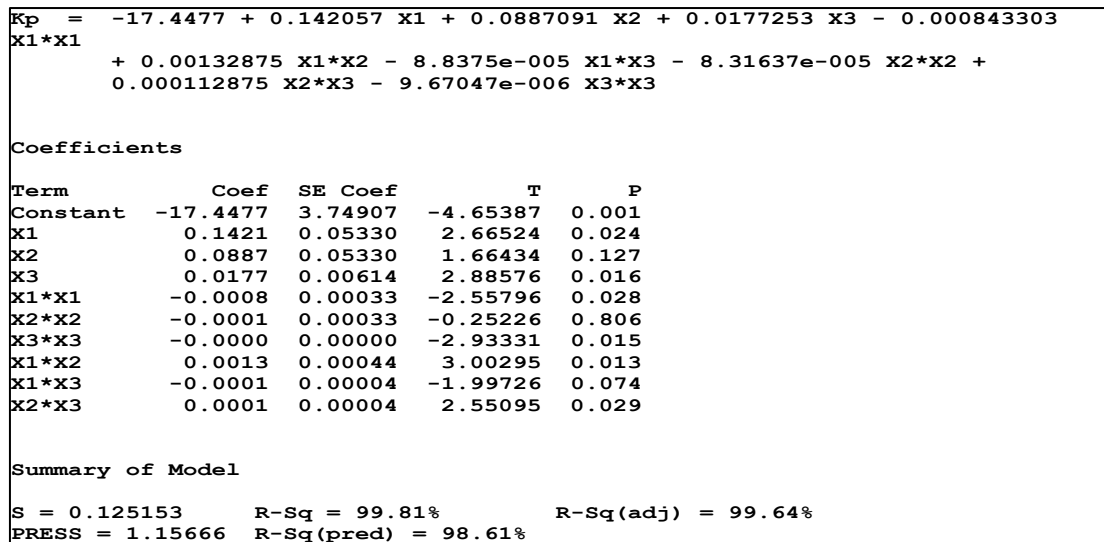


Figure 6: Minitab session shows final model in terms of natural variables

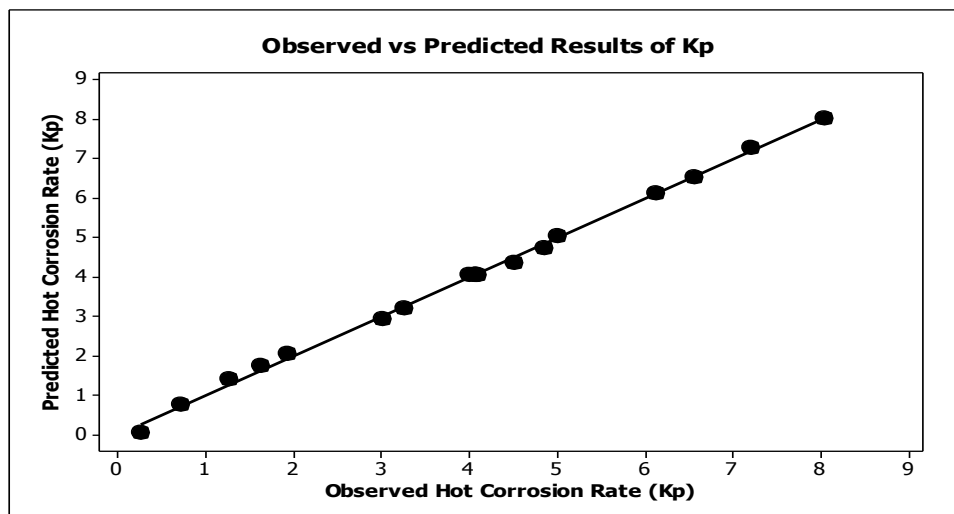


Figure 7: Plot of predicted response vs. actual value for hot corrosion rate (K_p) response from reduced surface quadratic model

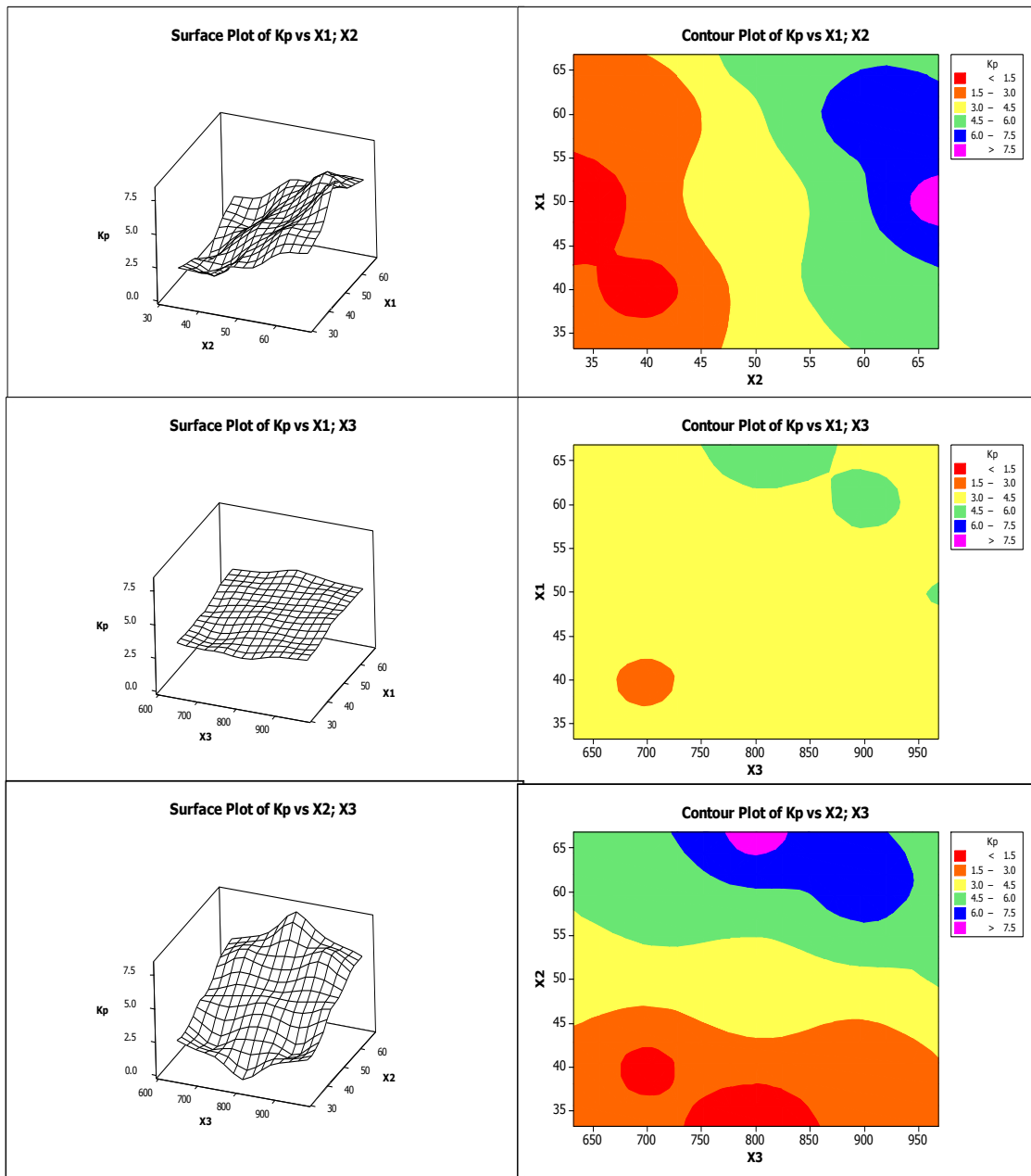


Figure 8: 3D surface and contour plotted for combined effect of hot corrosion parameters

تطبيق منهجية سطح الإستجابة لنمذجة و تحديد العوامل المثلى لمعدل التآكل الساخن في سبيكة نيمونك 75 المطلية بطريقة الألمنة-تيتنة المحورة بالسيريوم

عباس خماس حسين
الجامعة التكنولوجية
قسم هندسة المواد

الخلاصة:

أستخدم في هذا البحث منهجية سطح الإستجابة لتحديد الظروف المثلى للعوامل (النسبة الوزنية لكبريتات الصوديوم، النسبة الوزنية لأوكسيد الفناديوم، درجة الحرارة) التي تبدي أدنى قيمة ممكنة للتآكل الساخن في سبيكة نيمونك 75 المطلية بطريقة الألمنة-تيتنة المحورة بالسيريوم . و تم تصميم التجارب للعوامل الثلاثة . وفقاً لمبدأ التصميم المركب المركزي في منهجية سطح الإستجابة بإستخدام البرمجيات 16 MINITAB و MATLAB 2014a. و تم بناء الموديل الرياضي الذي يعبر عن تغير معدل التآكل الساخن مع عوامل التآكل الساخن بإستخدام منهجية سطح الإستجابة . و تم الحصول على العوامل المثلى و التي تتضمن (40 wt.% of Na_2SO_4 , 40 %wt. of V_2O_5 , and 900°C) حيث أن معدل التآكل الساخن (الذي تم تحديده من خلال الموديل الرياضي الذي تم التنبأ به) عند هذه الظروف كان ($K_p=1.430987 \times 10^{-10} \text{ g}^2 \text{ cm}^{-4} \text{ s}^{-1}$) . و لوحظ أن القيمة التجريبية لمعدل التآكل الساخن عند هذه الظروف كان ($K_p=1.4311 \times 10^{-10} \text{ g}^2 \text{ cm}^{-4} \text{ s}^{-1}$) (و هي في تطابق جيد مع القيمة المحسوبة من الموديل الرياضي . و هذا تم إثباته أيضاً من خلال قيمة معامل التحديد العالية ($R^2=99.81\%$) للموديل الرياضي الذي تم التنبأ به . كما أن قيمة معامل العلاقة ($R^2=98.991\%$) ما بين البيانات التجريبية و بيانات الموديل الرياضي تشير الى إمكانية إستخدام الموديل الرياضي للتنبأ بمعدل التآكل الساخن من خلال ظروف التآكل الساخن .