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FACTORIAL DESIGN BASED OPTIMISATION OF CREVICE CORROSION FOR TYPE 304 STAINLESS STEEL IN CHLORIDE SOLUTIONS

ABSTRACT

The effects of chloride concentration, creviced scaling factor and immersion time on the percentage area and maximum depth of attack for Type 304 stainless steel (SS304) in chloride solutions were investigated. The crevice assembly comprised of coupon (SS-304), polytetrafluoroethylene (crevice former) and fasteners (titanium bolt, nut and washers). The full immersion tests were based on ASTM G-78 using full factorial design to study the effects of chloride concentration (1.5, 3.0 and 4.5 w/w%), crevice scaling factor (8, 16 and 24) and immersion time (15, 30 and 45 days) on the percentage area of attack (Y_1) and maximum depth of attack (Y_2) of SS-304. Data obtained was used to develop and optimize the models of Y_1 and Y_2 in terms of the three factors using Response Surface Methodology (RSM). The R^2 of Y_1 and Y_2 were 0.98 and 0.91, respectively. The minimum Y_1 (5.63%) and Y_2 ($3.32x10^{-7}$ mm) were obtained at 4.5% chloride concentration, 20 scaling factor and 15 days immersion time. The predicted optimal conditions agreed with the experimental results for validation with a maximum absolute relative error of 5.75%.

Keywords: *crevice corrosion, scaling factor, stainless steel, optimisation, chloride solution*

INTRODUCTION

Stainless Steel 304 (SS-304) has extensive industrial usage due to its good mechanical and corrosion resistance properties [1, 2, 3]. In spite of wide industrial use of SS-304, its crevice corrosion failures in chloride containing environments are frequently reported [4, 5, 6, 7] in process equipment and installations whose fabrications and assembly involve unavoidable creviced geometry. Hu *et al.* [6] explained that metals in the passive system like SS-304 in sodium chloride solution and carbon steels in chloride containing bicarbonate solution would experience crevice corrosion when crevice opening ranges between 0.1mm and 0.5mm. In this phenomenon, species exchange between the inside and outside solutions is difficult and then, the crevice solution can be aggressive over time. This unavoidable nature of creviced geometry has made crevice corrosion of SS-304 a remarkable concern in chloride containing environments [7].

In order to control the propagation of crevice corrosion, the potential has to be kept lower than a critical value called the repassivation potential [8]. This has been identified as a very difficult control objective as far as the nature of crevice corrosion of stainless steel is concerned [9]. The dynamics of crevice corrosion are not fully understood, and so more experimental methods are required for *in situ* observation of the process at the metal/solution interface to obtain a simple and flexible crevice initiation and propagation model [9]. This will allow parametric studies of crevice corrosion of stainless steel to integrate repassivation conditions to design and operation of creviced SS-304 assemblies operated in chloride containing environments.

In crevice corrosion system involving SS-304, though, it is generally agreed that a varied concentration of chloride is notorious for causing components failure, a clear information on the relationship of its interactions with other geometrical and operational factors is still lacking [3, 6, 10, 11, 12]. Therefore, the operating conditions within the design criteria that control crevice corrosion in chloride containing environments need to be studied via suitable combinations of experimentation and modelling to determine the optimal values of these parameters [11, 12] so as to eliminate or reduce the occurrences of unanticipated failures in processing industries.

Several modelling studies like ohmic drop and other electrochemical methods [13, 14, 15, 16] have been developed. These were based on process variable that are non-controllable and hence, cannot be implemented in design and operation for controlling crevice corrosion [4, 5]. In all of these approaches, while considering the overall process, the likelihood, timing, and rate of attack were not considered. Another important issue that these models do not address is the effects of geometry within a crevice and the proper crevice scaling factor needed for different applications [17].

Mathematical optimisation by means of an experimental design is most helpful in determining the effects and interactions of important variables on responses [18]. In most corrosion studies reported, the effect of individual parameters on the corrosion process has been studied by keeping the level of other operating factors constant. These studies do not consider the combined effect of factors. Recent research that employed statistical based methodologies to address this gap includes that of Hepsen and Kaya [19] and Gu *et al.* [20]. In this present study, full factorial design and Response Surface Methodology (RSM) were used to evaluate effects of chloride concentration, crevice scaling factor and immersion time on the rate of crevice corrosion of SS-304 in chloride containing environments for the purpose of minimising its occurrence. These factors chosen are critical factors of crevice corrosion of SS 304 in chloride containing environments [4, 5, 6].

MATERIALS AND METHODS

Materials

The engineered crevice assembly designed for the purpose of this study consists of specimen (AISI 304) (coupons), multiple crevice former (polytetrafluoroethylene (PTFE)), titanium bolt, nuts and washers. The SS-304 sheets (3mm thick), teflon sheets (5mm thick), titanium bolt, nuts and washers.

Preparation of chloride containing environments

The test solution was NaCl solution prepared from analytical-grade of NaCl and double distilled water at 1.5% w/w NaCl to simulate the fresh water environment; 3% w/w NaCl to simulate the brackish water environment and 4.5%w/w NaCl to simulate the marine water environment.

Preparation of SS-304 crevice assembly

According to ASTM G-78 [21], multi-crevice assemblies of different crevice scaling factors and 40 crevice sites each were totally immersed in chloride containing environments and monitored for the rate of initiation and propagation of crevice corrosion in terms of percentage crevice sites attacked and maximum depth of attack, respectively at different immersion time intervals. The crevice assembly is as shown in Fig.1. After the crevice corrosion experiments, the corroded surface area of each sample was calculated using Photoshop based software and the depth of attack was measured with surface roughness tester (Surftest SJ210)

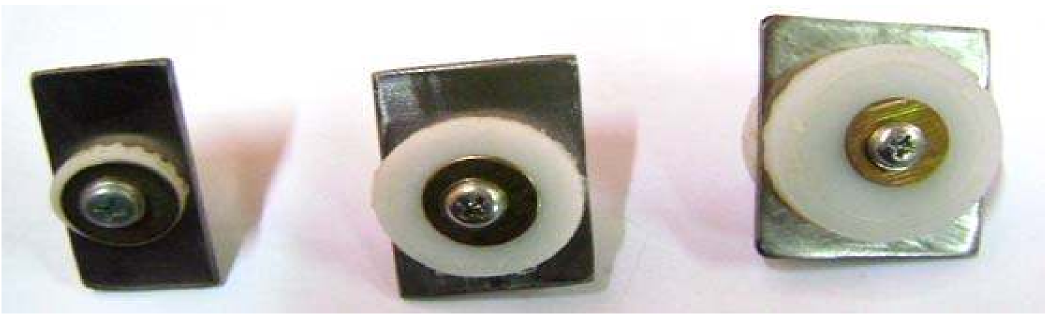


Fig. 1. Components of the multiple crevice assembly for corrosion test

Experimental design and statistical analysis

A 3-factor and 3-level full factorial design was used for the experiments. The working range of input parameters and their levels taken are as shown on Table 1. The detailed dimensioning and fabrication and coupling of multi-crevice assembly are as shown in Table 2. A second order polynomial equation was used to fit the experimental data given in Table 3. The model proposed for the response (Y_i) is Eq. (1)

$$Y_i = a_o + a_1X_1 + a_2X_2 + a_3X_3 + a_{11}X_1^2 + a_{22}X_2^2 + a_{33}X_3^2 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + \varepsilon \quad (1)$$

where Y_i (i = 1, 2) is the predicted response for Creviced Area Attacked (Y_1), and Maximum Depth of Attack (Y_2) to model the rate of initiation and propagation of SS-304 crevice corrosion in chloride containing environments, a_0 is the value of the fitted response at the centre point of the design, a_i, a_{ii}, a_{ij} being the linear, quadratic, and cross product terms, respectively and ε is the random error.

Table 1. *Coded and actual values of the process variables*

Variables	Units	Symbol	Level		
			-1	0	+1
Chloride Concentration	%	A	1.5	3	4.5
Crevice Scaling Factor	-	B	8	16	24
Time	days	C	15	30	45

Table 2. *Detail design parameters of creviced assembly types*

Crevice Assembly Type	Parameters	Values
TYPE A	Width of a single Crevice, w (mm)	1
	Length of a single Crevice, l (mm)	4
	Depth of a Single Crevice, d (mm)	0.5
	Surface Area of a Single Crevice, a (mm^2)	2
	Total Crevice Area, A (mm^2)	80
TYPE B	Width of a single Crevice, w (mm)	1.5
	Length of a single Crevice, l (mm)	8
	Depth of a Single Crevice, d (mm)	0.5
	Surface Area of a Single Crevice, a (mm^2)	4
	Total Crevice Area, A (mm^2)	160
TYPE C	Width of a single Crevice, w (mm)	2
	Length of a single Crevice, l (mm)	12
	Depth of a Single Crevice, d (mm)	0.5
	Surface Area of a Single Crevice, a (mm^2)	6
	Total Crevice Area, A (mm^2)	240

Optimization of crevice corrosion parameters

The objective of this work was to find the optimal combination of input parameters that provided minimum initiation and propagation rate of crevice corrosion of SS-304 in chloride containing environments. Here, the objective is:

Minimise:

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{23} X_2 X_3 + \varepsilon \qquad (2)$$

Subject to:

$A_{min} \le A \le A_{max}$; 3 $B_{min} \le B \le A_{max}$; 4 $C_{min} \le C \le C_{max}$; 5

The responses were monitored and the results were compared with predictions as per the model. To visualize the relationship between the responses and experimental levels for each of the factors and also to deduce the optimum conditions, the fitted polynomial equation was expressed as both surface and contour plots. The optimization routine of Design-expert software 9.0 using factorial design as shown on Table 3 was used to search for a combination of factor levels that simultaneously satisfy the optimization criteria on each one of the responses and process factors considered in this work.

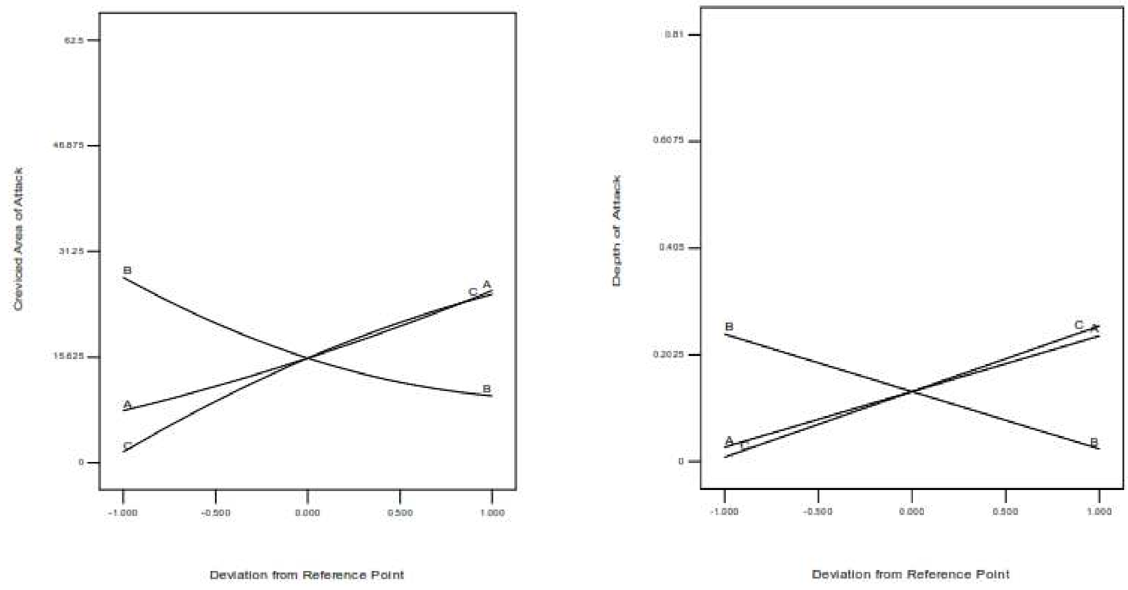
RESULTS AND DISCUSSION

Effects of crevice corrosion parameters on the percentage area and depth of attack

The corresponding percentage area and depth of attacks as a result of 32 experimental runs for various combinations of chloride concentrations, crevice scaling factor and time of immersion are as presented in Table 3. The 3-level factorial design employed in the application of RSM resulted in the 32 experimental runs. In order to clearly see the effect of each factor on the two responses, the factor effect function (Fig. 2) was used to show the effect of each factor graphically based on data on Table 3. The perturbation from the centre point of the experiment (1.5%, 16 mm/mm and 30 days) was plotted to show their corresponding relationship with each response. From the trace plot as shown in Fig. 2a, it can be seen that each of the three variables used in the present study has its individual effect on the percentage area of attack. Gradual increase in chloride concentration and time of immersion increased the percentage area of attack while such increment in the crevice scaling factor reduced the percentage of area of attack on the assembly. The depth of attack followed the same trend as shown on Fig. 2b which is in agreement with the result of Cai *et al.* [22] and Yang *et al* [23] .The maximum percentage area and depth of attack value of 62.5% and 0.81mm, respectively were obtained at run 7 which correspond to 4.5% chloride concentration, 8 mm/mm crevice scaling factor and 45 days immersion time. It was a different result at run 15 where the area and depth of attacks were both zero. Therefore, it can be said that higher crevice scaling factor can actually bring crevice corrosion to zero.

Table 3. *The Experimental conditions and corresponding responses*

Runs	Factors			Responses	
	A(%)	**B (mm/mm)**	**C(days)**	**Y_1 (%)**	**Y_2(mm)**
1	3.0	24	30	10.0	0.02
2	3.0	16	30	15.0	0.10
3	1.5	8	30	15.0	0.03
4	3.0	8	45	40.0	0.56
5	1.5	8	45	27.5	0.10
6	4.5	16	30	27.5	0.15
7	4.5	8	45	62.5	0.81
8	1.5	16	45	12.5	0.05
9	4.5	24	30	17.5	0.05
10	1.5	16	15	0.0	0.00
11	3.0	16	45	25.0	0.30
12	3.0	24	15	0.0	0.00
13	1.5	24	45	7.5	0.05
14	4.5	24	45	22.5	0.10
15	1.5	24	15	0.0	0.00
16	4.5	16	15	10.0	0.10
17	1.5	24	30	2.5	0.00
18	3.0	16	15	2.5	0.01
19	3.0	16	30	15.0	0.10
20	3.0	8	15	7.5	0.10
21	4.5	16	45	35.0	0.50
22	3.0	16	30	15.0	0.10
23	1.5	16	30	7.5	0.02
24	3.0	24	45	17.5	0.04



(a) **(b)**

Fig. 2. Factor plot representing individual variable effect on (a) area of attack and (b) depth of attack

Response equations and statistical analysis

Multiple regression analysis of both the percentage area of attack (Y_1) and depth of attack (Y_2) in term of chloride concentration (A), scaling factor (B) and time of immersion (C) were made in order to develop models in form of Eq. (1). This is a crucial step in RSM in order establish models that correctly represent the experiments which can in turn be optimized . The statistical results from the factorial design for parameter estimation are summarized in Table 4 for both Y_1 and Y_2 while Table 5 represent the reduced parameter factorial design after the non-significant terms at $p < 0.05$ have been eliminated. The model for Y_1 followed a quadratic model where all the factors in their linear forms were significant and the interaction between factors were also significant at $p < 0.05$. A shown on Table 5, the area of attack depends on the chloride concentration, scaling factor and the time of immersion. Any gradual increments in both A and C increased theY_1 while B reduced it. The interaction between chloride concentration (A) and scaling factor (B) as well as scaling factor (B) and time (C) reduced the value of Y_1. However, the interaction between A and C increased the value of Y_1. It is clearly seen here that the influence of the three factors on Y_1 is nonlinear.

The resulting model for Y_2 is a 2-Factor Interaction (2FI) model. Here, the linear terms of the factors as well as the interactions were significant at $p< 0.05$ as indicated on Table 5. The influence of the three factors on the Y_2 followed the trend of that of Y_1. The regression coefficients, standard error of coefficients and p-values are listed in Table 5. By replacing the coefficients in Eq. (1) with their corresponding values from Table 5, the best-fitted model equations are obtained in terms of factors affecting the creviced area attacked (Y_1) and maximum depth of attack (Y_2) are as given in Eq. (6) and (7), respectively. These equations explain the rate of initiation and propagation of crevice corrosion of SS-304 in chloride containing environments, respectively.

Table 4. *Statistical parameters for the factorial design*

Response	Model Term	Coefficient	Standard Error of Coefficient	p-Value	Remark
Area of Attack (Y_1)	b0	15.48	0.78	< 0.0001	Significant
	A	8.89	0.55	*< 0.0001*	Significant
	B	-8.75	0.55	*< 0.0001*	Significant
	C	11.67	0.55	*< 0.0001*	Significant
	A^2	1.1	0.87	*0.2182*	Not Significant
	B^2	3.19	0.87	*0.0014*	Significant
	C^2	-2.23	0.87	*0.0176*	Significant
	AB	-3.13	0.67	*0.0001*	Significant
	AC	3.96	0.67	*< 0.0001*	Significant
	BC	-5.42	0.67	*< 0.0001*	Significant
Maximum Depth of Attack (Y_2)	b0	0.11	0.02	< 0.0001	Significant
	A	0.11	0.014	*< 0.0001*	Significant
	B	-0.11	0.014	*< 0.0001*	Significant
	C	0.12	0.014	*< 0.0001*	Significant
	A^2	-9.84E-03	0.022	*0.666*	Not Significant
	B^2	6.83E-03	0.022	*0.7643*	Not Significant
	C^2	0.05	0.022	*0.0362*	Not Significant
	AB	-0.082	0.017	*0.0001*	Significant
	AC	0.089	0.017	*< 0.0001*	Significant
	BC	-0.097	0.017	*< 0.0001*	Significant

Table 5. *Statistical reduced parameters for the factorial design*

Response	Model Term	Coefficient	Standard Error of Coefficient	p-Value	Remark
Area of Attack (Y_1)	b0	15.87	0.73	< 0.0001	Significant
	A	8.89	0.56	*< 0.0001*	Significant
	B	-8.75	0.56	*< 0.0001*	Significant
	C	11.67	0.56	*< 0.0001*	Significant
	B^2	3.4	0.86	*0.0007*	Significant
	C^2	-2.02	0.86	*0.0286*	Significant
	AB	-3.12	0.68	*0.0001*	Significant
	AC	3.96	0.68	*< 0.0001*	Significant
	BC	-5.42	0.68	*< 0.0001*	Significant
			R^2 = 0.98		
Maximum Depth of Attack (Y_2)	b0	0.13	0.011	< 0.0001	Significant
	A	0.11	0.015	*< 0.0001*	Significant
	B	-0.11	0.015	*< 0.0001*	Significant
	C	0.12	0.015	*< 0.0001*	Significant
	AB	-0.082	0.018	*0.0001*	Significant
	AC	0.089	0.018	*< 0.0001*	Significant
	BC	-0.097	0.018	*< 0.0001*	Significant
			R^2 = 0.91		

$$\% \text{ Area Attacked } (Y_1) = +15.87 + 8.89A - 8.75B + 11.67C + 3.40B^2 - 2.02C^2 - 3.12AB + 3.96AC - 5.42BC \quad (6)$$

$$\text{Maximum Depth of Attack}(Y_2) = +0.13 + 0.11A - 0.11B + 0.12C - 0.082AB + 0.089AC - 0.097BC \quad (7)$$

Eq. (6) and (7) were used to evaluate the influence of process variables on the creviced area attacked (Y_1) and maximum depth of attack (Y_2), respectively. Analysis of variance (ANOVA) method was employed to further estimate the significance and accuracy of the models. The corresponding results are presented in Table 6. As it can be seen in the Table 6, the models had F- values of 135.20 and 40.95 for Y_1 and Y_2, respectively that were significant at $p < 0.05$. Also, the coefficient of determination (R^2) of the two models are 0.9806 and 0.9076, respectively. These high values of R^2 is an indication of the agreement between the actual experimental values and the predicted values from the model. These values shows that

96% of the total variation in the data could be explained by the models. This shows that the models are reliable for predicting the process.

Table 6. *ANOVA of Response Surface Quadratic Model for Percentage Area Attacked*

Response	Source	Sum of Square	DF	Mean Square	F- Value	p-value	Remark
Area of Attack (Y₁)	**Model**	6004.92	8	750.61	135.20	< 0.0001	Significant
	Linear	5250.35	3	5250.35	945.66	< 0.0001	Significant
	Square	116.03	2	116.03	20.90	< 0.0001	Significant
	Interaction	657.29	3	657.29	118.39	< 0.0001	Significant
	Residual	127.70	23	5.55			
	Lack of Fit	127.70	18	7.09			
	Pure Error	0.00	5	0.00			
	Total	6132.62	31				
	R^2 = 0.9806						
Maximum Depth of Attack (Y_2)	**Model**	0.98	6	0.16	40.95	< 0.0001	Significant
	Linear	0.69	3	0.69	173.64	< 0.0003	Significant
	Interaction	0.29	3	0.29	72.08	< 0.0004	Significant
	Residual	0.10	25	0.00			
	Lack of Fit	0.10	20	0.00			
	Pure Error	0.00	5	0.00			
	Total	1.08	31				
	R^2= 0.9076						

Optimisation studies of crevice corrosion damage behaviour of SS-304 in chloride containing environment

In attempt to find the minimum response values for the percentage of area of attack (Y_1) and the depth of attack (Y_2) of SS304 in chloride containing environment, the numerical optimization function based on the desirability index in the design expert 9.0 was employed to locate the values of chloride concentrations, creviced scaling factor and time of immersion. The optimal values of the systems were computed for all levels of chloride concentration (A), creviced scaling factor (B) and time of immersion (C). The composite desirability was at the maximum index of 1.000 and the percentage area of attack and maximum depth of attack reached the minimum value of 1.26 x 10^{-5} mm^2 and 7.24 x10^{-9} m, respectively at chloride concentration (2.14 wt %), creviced scaling factor (15.52 mm/mm) and time of immersion(15.58 days).

The response surface plots based on the predictive models in Eq. (6) - (7) for the percentage area of attack and depth of attack, respectively were obtained and illustrated in Fig. 3 and Fig. 4, respectively. Effects of chloride concentration and creviced scaling factor on percentage area of attack of SS 304 is as shown on Fig. 3a, chloride concentration and time of immersion on Fig. 3b while Fig. 3c illustrates the effects of creviced scaling factor and time of immersion. Similar illustrations for depth of attack are presented in Fig. 4. Fig. 3a depicts that area of attack of SS304 are minimized at lower chloride concentration and short immersion time. High creviced scaling factor with either low chloride concentration or short immersion time minimized area of attack for SS304 as shown on Fig. 4b and 3c, respectively. Similar explanation holds for Fig.4a – Fig. 4c for depth of attack of SS304.

The suitability of the models developed for predicting the optimum response values were tested using three practical examples. These examples were the chloride concentrations of 4.5, 3.0 and 1.5% representing the marine, brackish and fresh water conditions, respectively. Here, it is envisaged that if these conditions were encountered, this section shows how the two other factors may be adjusted to obtain a minimum effect on the SS304. The simulated optimum factors were also experimented for comparisons. The result of this validation experiment is as presented on Table 7. Each experiment was done in triplicate and the means are presented. As depicted on the Table, the minimum effects were obtained with the fresh water conditions. The implication of this results is that, components inspection must always be done latest by the fifteenth (15) day in order to avert equipment deteriorations that can lead to the eventual failure. Likewise, the relatively low absolute error between the model predicted responses and the measured values from the experiment depicts the ability of the model to represent the real process.

The micrographs of samples of creviced surfaces are presented in Fig.5. The polished surface without immersion in the chloride solution is as shown in Fig. 5a while the surface after immersion in brackish environment is in Fig.5b. Finally, Fig. 5c depicts the surface after being immersion in the chloride solution at an optimised condition. The effects of these optimisation is clearly seen when Fig. 5b is compared to Fig. 5c.

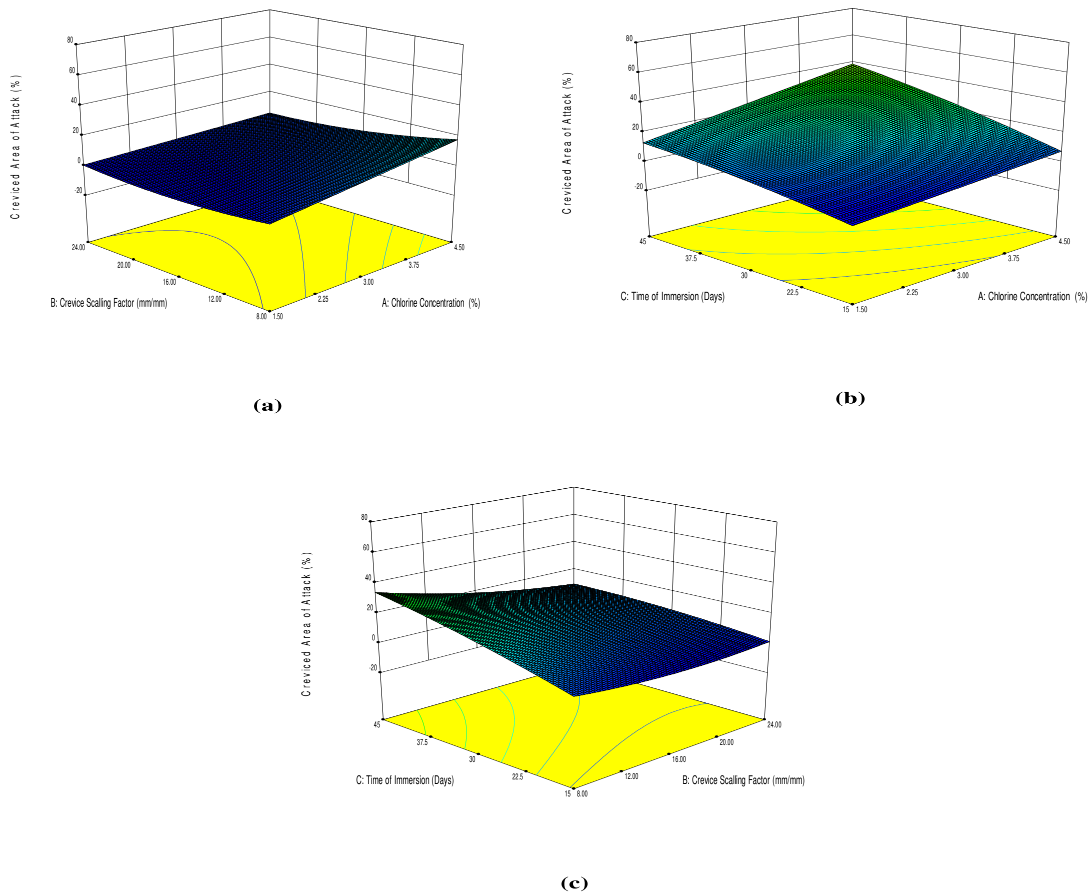
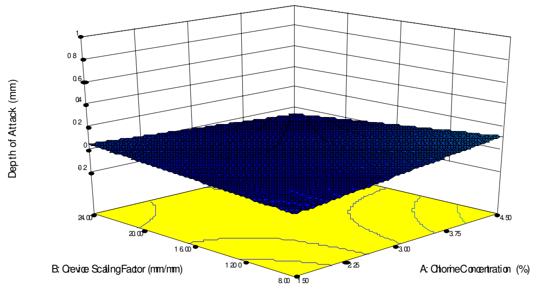
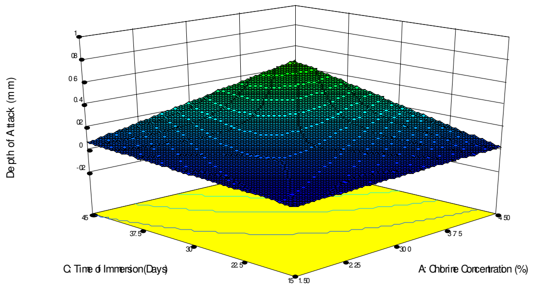


Fig. 3. The combined effects factors on the percentage area of attack of SS304 in chloride environment
(a) chloride concentration and creviced scaling factor, C = 15.58 days
(b) chloride concentration and time of immersion, B = 15.52 mm/mm
(c) creviced scaling factor and time of immersion, A = 2.14 %



(a)



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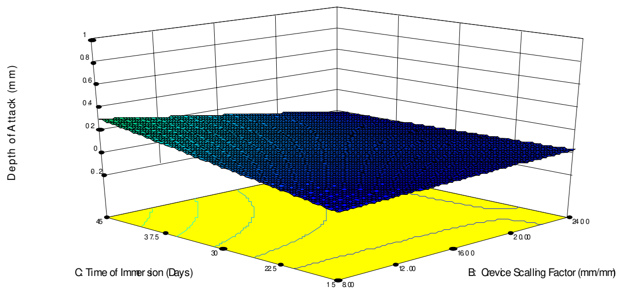
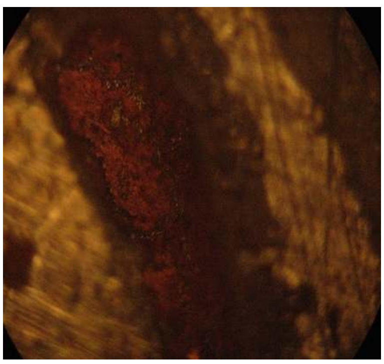


(c)

Fig. 4. The combined effects factors on the depth of attack of SS304 in chloride environment (a) chloride concentration and creviced scaling factor, C = 15.58 days (b) chloride concentration and time of immersion, B = 15.52 mm/mm (c) creviced scaling factor and time of immersion, A = 2.14 %

a



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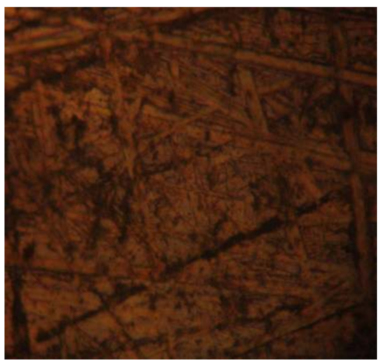

c

Fig. 5. Micrographs of Creviced Surface (a) Polished surface without immersion (b) Crevice surface at non - optimised condition (c) Creviced surface at optimised condition

Table 7. *The optimal parameters and responses for crevice corrosion of SS-304 in chloride containing environments*

Crevice Factors			Optimum Predicted Response		Optimum Measured Responses		Average Measured Values		Absolute Relative Errors (%)	
A	B	C	$Y_{1(pred)}$ (%)	$Y_{2(pred)}$ (mm)	$Y_{1(exp)}$ (%)	$Y_{2(exp)}$ (mm)	Y_1	Y_2	Y_1	Y_2
4.5	**20**	**15**	5.64	$3.16x10^{-7}$	5.58	$3.20x10^{-7}$	5.63	$3.32x10^{-7}$	0.17	5.06
					5.60	$3.35x10^{-7}$				
					5.72	$3.40x10^{-7}$				
3.0	**21**	**15**	1.42	$7.20x10^{-4}$	1.50	$7.18x10^{-4}$	1.48	$7.21x10^{-4}$	4.23	0.14
					1.47	$7.20x10^{-4}$				
					1.48	$7.24x10^{-4}$				
1.5	**16**	**15**	$2.76x10^{-5}$	$2.26x10^{-7}$	$2.80x10^{-5}$	$2.40x10^{-7}$	$2.79x10^{-5}$	$2.39x10^{-7}$	1.09	5.75
					$2.78x10^{-5}$	$2.38x10^{-7}$				
					$2.78x10^{-5}$	$2.40x10^{-7}$				

CONCLUSION

Application of the response surfaces methodology to optimise the parameters influencing the crevice corrosion of SS-304 in chloride containing environment has been carried out. A three factor factorial experimental design-based optimization modelling has been carried out to minimise the percentage area and depth of attack for SS304 in chloride environment. The results reveal that the interaction of chloride concentration , creviced scaling factor and time of immersion play important roles in the corrosion of SS304. The predictions of the regression model based on experimental design agreed well with the experimental results and can be the basis for crevice corrosion maintenance of SS304 in chloride environment.

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