Determining the Minimum Corrosion Conditions for the Stir Zone of Friction Stir Welded AA6061 Aluminium Alloy Joints

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ABSTRACT

Joining of aluminium is commonly done in automobile industries because of its light weight and high specific strength. In recent days, friction stir welding (FSW) is widely preferred to join aluminium than fusion-welding processes. In this joint, grains are very fine in stir zone (SZ) compared to the other zones. Due to this extreme change in the microstructure at the SZ, the mechanical properties (tensile strength, hardness, etc) of the FSW joints are superior but the corrosion resistance of SZ is very poor. The concentration of chloride ion, exposure time and pH value are reported to be the more influencing corrosion test parameters. The present work aims to determine combination of these pitting corrosion test parameters to attain a minimum corrosion rate at the SZ of friction stir welded aluminium alloy, AA6061-T6, by response surface methodology (RSM). From the results obtained, chloride ion concentration is reportedly had higher effect on corrosion rate than the other two parameters considered.

Keywords: AA6061 aluminium alloy, Stir zone, Response surface methodology, Pitting corrosion test.

1.0 INTRODUCTION

In automobile industries, aluminium alloys are extensively used due to high strength, low density and excellent corrosion resistance [1-3]. Due to rapid formation of oxide films on aluminium alloy, it is quite difficult task to join these materials by fusion welding methods [4-6]. In order to overcome this problem, the solid state welding techniques are commonly used to join the aluminium alloys.

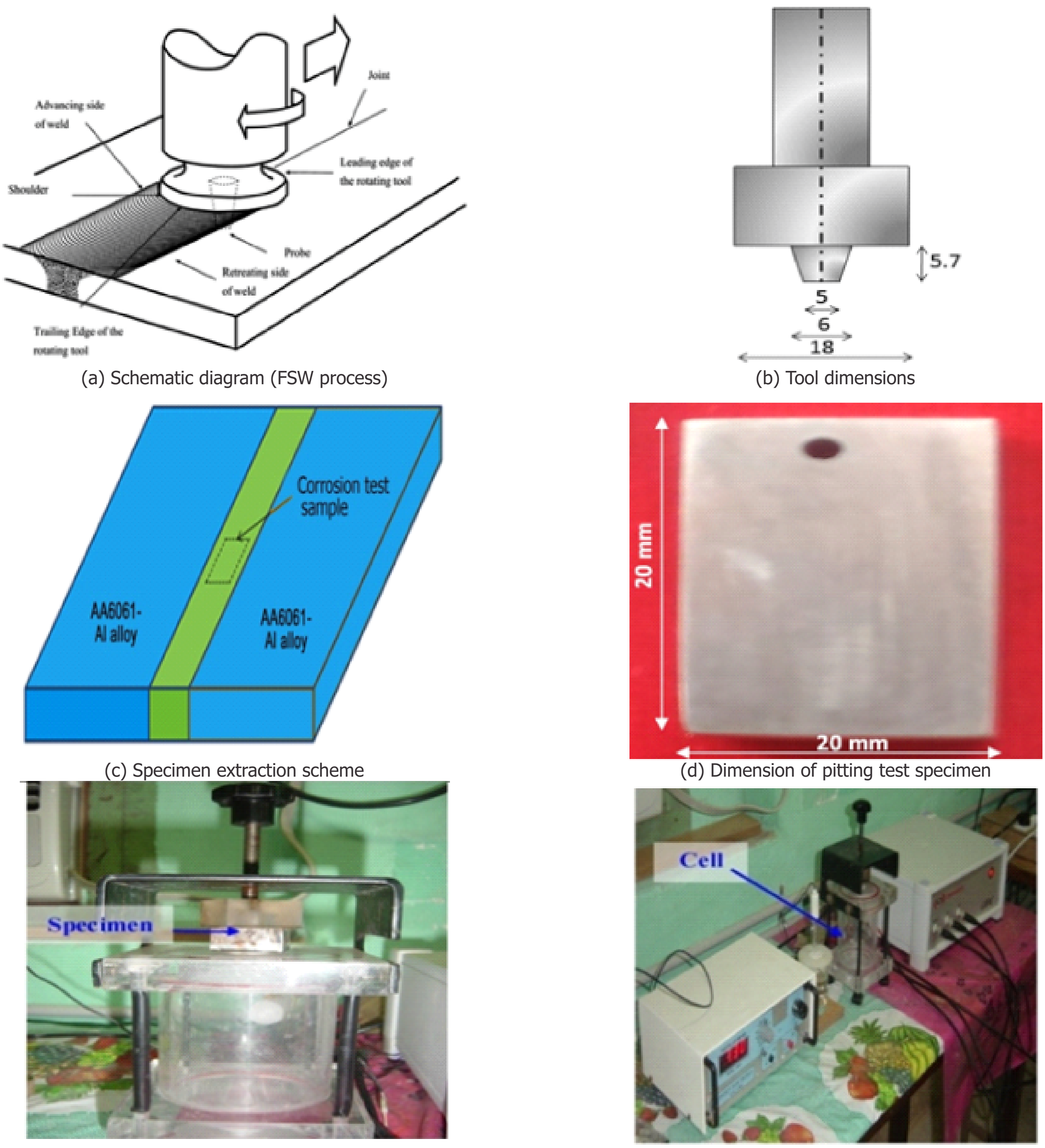
Friction stir welding (FSW) is a non-fusion joining techniques specially developed to join aluminium and its alloys [7]. FSW joint exhibits four different regions and stir zone (SZ) is the most important region, which has dynamically recrystallized microstructure. Hence it is most important to study the corrosion behaviour of the SZ. Few researchers investigated the corrosion behaviour of FSW joints and reported that the corrosion behaviour of the FSW joint depends on FSW parameters [8, 9].

The electrochemical corrosion test is used to measure the electrochemical potential of a material and also it can be used to estimate the relative activity of a material in a particular environment. Different electrochemical testing methods are available to evaluate the corrosion rate of a material such as potentiostatic, potentiodynamic, cyclic polarization, etc., [10]. Among these potentiodynamic polarization measurements are quite appropriate for determination of the pitting susceptibility, corrosion current density and corrosion rate of aluminium alloys [11]. Some researchers carried out the potentiodynamic corrosion test on aluminium and magnesium alloys [12, 13].

The rate of corrosion of the material is varied by means of concentration of Cl- ion, pH of the solution and exposure time of a material [14]. Depending on the level of these factors, the rate of corrosion is varied. The life of the components is depending upon the rate of corrosion occur on the joints. Hence, in this respect the present work is aimed to determine the combination of pitting corrosion test parameters to attain the minimum corrosion rate in the SZ.

2.0 EXPERIMENTAL WORK

AA6061-T6 aluminium rolled plates are used in this investigation and the thickness of the plate is 6 mm. The chemical composition of parent metal (AA6061-T6 aluminium alloy) in weight percentage is 1% Mg, 0.6% Si, 0.25% Cu, 0.2% Cr and balance Al.



(a) Schematic diagram (FSW process)

(b) Tool dimensions

(c) Specimen extraction scheme

(d) Dimension of pitting test specimen

(e) Corrosion test cell

(f) Gill AC potentiostat instrument

Fig.1 : Experimental details

Table 1 : Process parameters and dimensions of tools

Rotational speed of tool, rpm	Tool travel speed mm/min	Axial force, kN	Tool shoulder diameter, mm	Tool pin diameter, mm	Tool Pin length, mm	Tool pin profile
1000	30	5.5	18	5-6	5.7	Taper cylindrical

Prior to FSW joint the parent metals were cut to the dimensions of 150 mm x 75 mm. Schematic representation of AA6061 aluminium joint and dimensions of tool are shown in **Fig. 1(a) & 1(b)** respectively.

The FSW parameters and tool dimensions used in this investigation is presented in **Table 1**. From this fabricated joints, the coupons were extracted from weld nugget region for conducting potentiodynamic corrosion test with the dimensions of 20 x 20 x 6 mm. The scheme of extraction of pitting corrosion test samples is shown in **Fig.1(c)**. Then the coupons were polished with SiC paper and it was properly cleaned with acetone. Finally it was dried by blowing warm air. The photograph of the polished corrosion test specimen is shown in **Fig. 1(d)**. The sample placed in a pitting corrosion test cell is shown in **Fig. 1(e)**. The Gill-AC potentiostat instrument was used to conduct the potentiodynamic polarisation test in NaCl solution at various conditions as shown in **Fig. 1(f)**. The optical micrograph of parent metal and SZ of FSWed joints are shown in **Fig. 2**. Corroded surfaces were analysed by scanning electron microscope (JEOL-JSM-5610LV).

3.0 SELECTION OF EXPERIMENTAL DESIGN MATRIX

Identifying the important corrosion test parameters fixing their testing range, selection of appropriate design matrix were presented in our previous study [15].

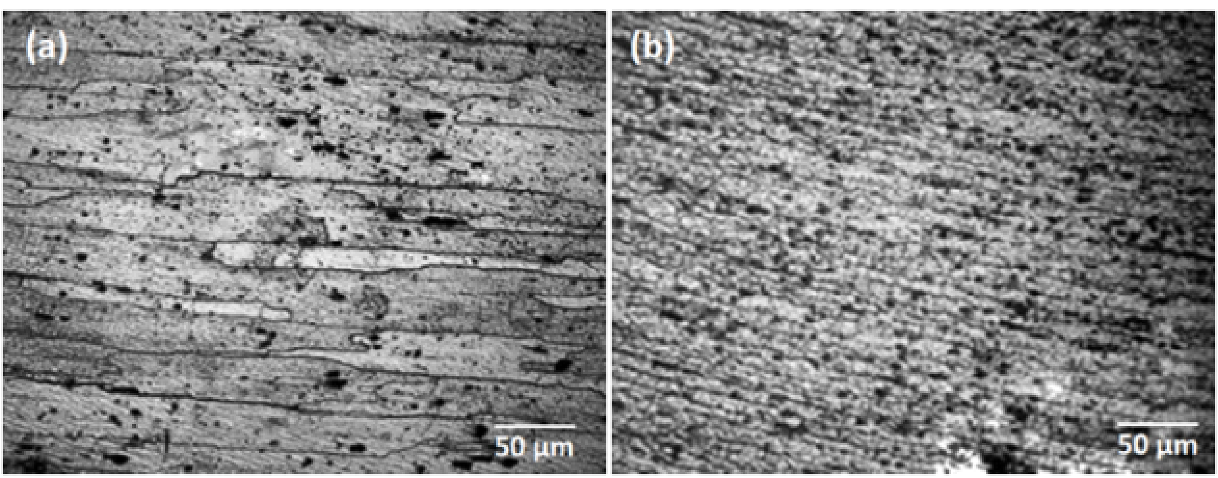


Fig 2 : Optical micrograph of AA6061 (a) parent metal and (b) SZ of friction stir welded joint.

Table 2. Main factors and levels

Sl. No.	Factor	Notation	Unit	Levels				
				-1.68	-1	0	+1	+1.68
1	Chloride ion concentration	C	mol	0.2	0.36	0.6	0.84	1
2	pH value	P	-	3	4.62	7	9.38	11
3	Time of Exposure	T	Hr	4	5.62	8	10.38	12

Table 3 : The design matrix chosen and experimental results obtained

Experimental number	Coded values			Actual values			Corrosion rate (mm/year)
	Conc.(C)	pH (P)	Time (T)	Conc. (C)	pH (P)	Time (T)	
1	-1	-1	-1	0.36	4.62	5.62	0.0042
2	+1	-1	-1	0.84	4.62	5.62	0.0057
3	-1	+1	-1	0.36	9.38	5.62	0.0023
4	+1	+1	-1	0.84	9.38	5.62	0.0040
5	-1	-1	+1	0.36	4.62	10.38	0.0027
6	+1	-1	+1	0.84	4.62	10.38	0.0044
7	-1	+1	+1	0.36	9.38	10.38	0.0011
8	+1	+1	+1	0.84	9.38	10.38	0.0028
9	-1.682	0	0	0.20	7.00	8.00	0.0015
10	+1.682	0	0	1.00	7.00	8.00	0.0044
11	0	-1.682	0	0.60	3.00	8.00	0.0048
12	0	+1.682	0	0.60	11.00	8.00	0.0021
13	0	0	-1.682	0.60	7.00	4.00	0.0051
14	0	0	+1.682	0.60	7.00	12.00	0.0027
15	0	0	0	0.60	7.00	8.00	0.0035
16	0	0	0	0.60	7.00	8.00	0.0035
17	0	0	0	0.60	7.00	8.00	0.0035
18	0	0	0	0.60	7.00	8.00	0.0034
19	0	0	0	0.60	7.00	8.00	0.0036
20	0	0	0	0.60	7.00	8.00	0.0035

The limits of factors used in this investigation are presented in **Table 2**. The actual and coded values are presented in **Table 3**. The lower and upper range of parameters have been coded as -1.682 and +1.682, respectively.

4.0 DEVELOPING AN EMPIRICAL RELATIONSHIP

The rate of corrosion was derived from the potentiodynamic polarization curve, for the maximum and minimum corrosion rate conditions and the related polarization curves are shown in **Fig. 3**. To relate the pitting corrosion test factors and the rate of corrosion, a second order quadratic polynomial equation was developed. The response (rate of corrosion) is a function of Cl- ion concentration (C), pH value (P), and exposure time (T). Hence it is expressed as

$$\textit{Corrosion rate} = f\ \{C, P, T\} \quad (1)$$

The relationship should contain all factors; therefore the preferred relation is given below

$$Y = b_0 + \Sigma b_i x_i + \Sigma b_{ii} x_i^2 + \Sigma b_{ij} x_i x_j \quad (2)$$

The polynomial expression used in this investigation for the three factors is given in our previous study [16]. The significance of each co-efficient was evaluated by student's t-test and the results are presented in **Table 4**; Values of "Prob >F" less than 0.05 indicate that the model terms are significant.

Table 4 : ANOVA test results

Source	Sum of squares ($x10^{-6}$)	Df	Mean squares ($x10^{-6}$)	F value	p-value Prob>F	
Model	26.270	9	2.9190	549.80	< 0.0001	significant
C	9.645	1	9.6450	1816.45	< 0.0001	
P	9.418	1	9.4180	1773.55	< 0.0001	
T	6.247	1	6.2470	1176.38	< 0.0001	
CP	0.005	1	0.0050	0.94	0.3547	
CT	0.005	1	0.0050	0.94	0.3547	
PT	0.020	1	0.0200	3.77	0.0810	
C2	0.580	1	0.5800	109.25	< 0.0001	
P2	0.008	1	0.0082	1.54	0.2422	
T2	0.263	1	0.2636	49.64	< 0.0001	
Residual	0.053	10	0.0053			
Lack of Fit	0.033	5	0.0066	1.66	0.2969	Not significant
Pure Error	0.020	5	0.0040			
Cor Total	26.330	19				

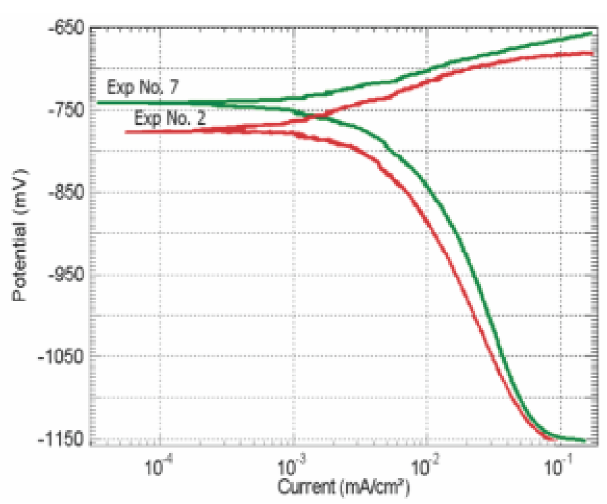


Fig. 3 : Potentiodynamic polarization curves recorded during corrosion test.

Corrosion rate (mm/year) =

$$0.0035 + (0.0008xC) - (0.0008xP) - (0.0007xT) + (0.00002xCxP)+(0.00002xCxT) + (0.00005xPxT) - (0.0002xC^2) - (0.00002xP^2) + (0.0001xT^2) \quad (3)$$

The significant and main interaction factors were found by analysis of variance (ANOVA). The F-value of 549.80 infers the model to be significant. Values of "Prob > F" less than 0.0500 indicate that terms considered in the model are significant. In this model, C, P, T, C^2, T^2 are significant.

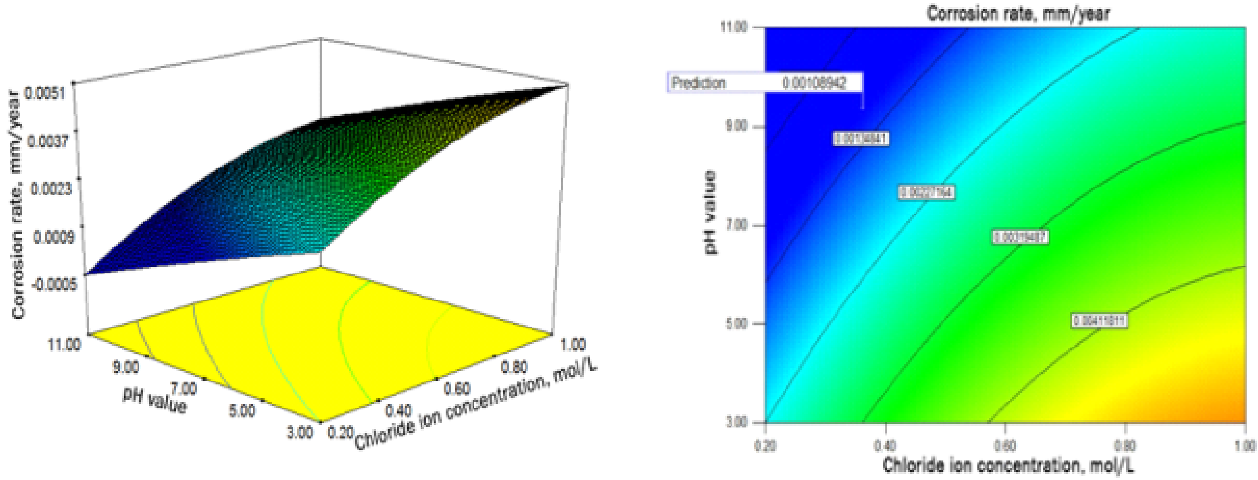
5.0 DETERMINING MINIMUM CORROSION CONDITIONS

A contour plot is a graphical method for representing a three dimensional (3D) surface in a two dimensional (2D) format. The contour plot is an alternative to a 3D surface plot. From the 3D surface and contour plot (**Fig.4a**), it can be observed that the minimum rate of corrosion observed in a Cl-ion concentration of 0.36 mol and a pH value of 9.36. In **Fig.4b**, the condition is required to achieve a minimum corrosion rate is 0.36 mol of Cl- concentration and an exposure time of 9.36 hr.

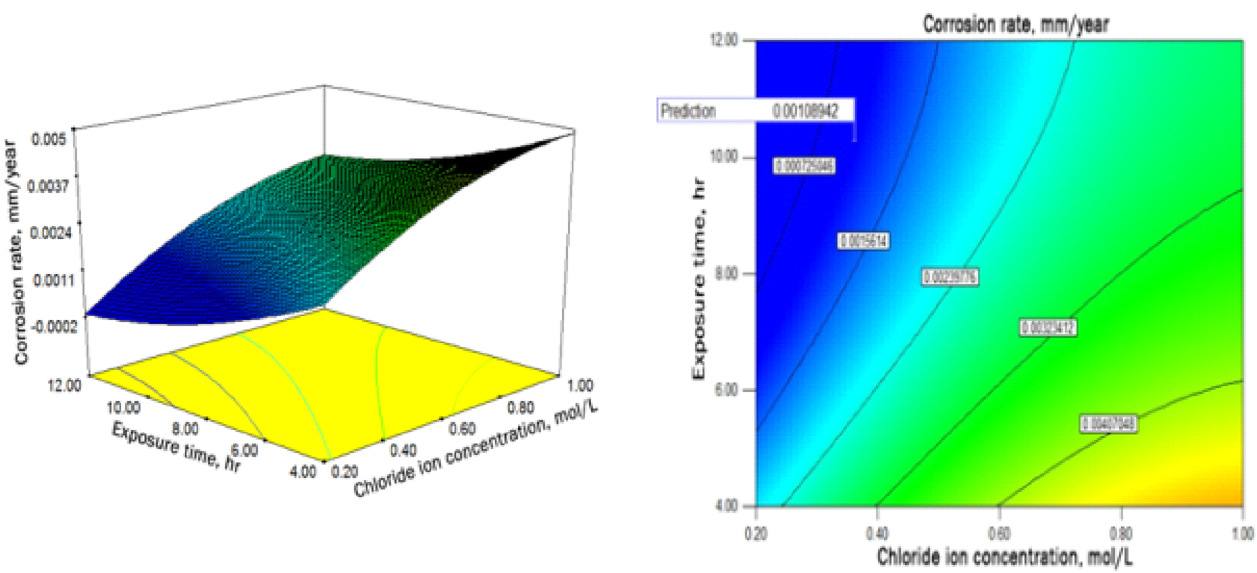
Fig.4c is the relationship between the pH value and the time of exposure, in this minimum achievable rate of corrosion observed in a pH value of 9.36 and an exposure time of 10.28 hr. The rate of corrosion decreases linearly with increase in value of pH and exposure time. It may be due to aluminium dissolution in NaCl solutions taking place by water reduction then it produce aluminium hydroxide ($Al(OH)_3$) and H_2 gas. These effects are insensitive to O_2 concentration [17].

Solutions with high acidic are aggressive to AA6061 aluminum alloy, therefore a very high rate of corrosion observed at pH 3. The metal get started to passivate after some time it will prevent the metal from further corrosion. The rate of corrosion typically increased with the increase in concentration of NaCl solution. The increase in rate of corrosion with increasing concentration of Cl- ion possibly attributed to the participation of Cl- ions in the dissolution reaction. The concentrations of Cl- ions are aggressive for Al alloys. Adsorption of Cl- ions covered the Al surface then transforms in to $Al(OH)_3$.

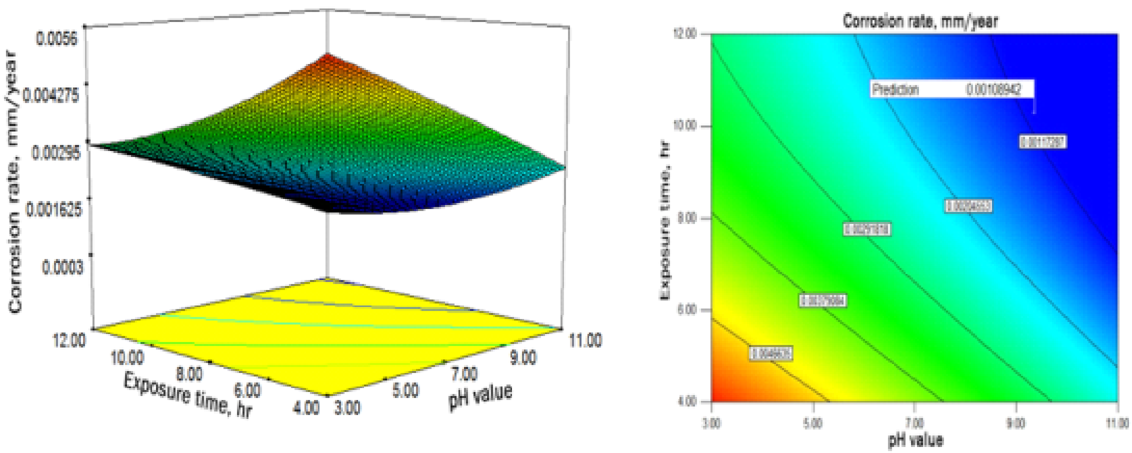
By examining the 3D surface and contour plots as shown in **Fig.4 (a-c)**, the minimum achievable corrosion rate value is found to be 0.001089 mm/year. The respected parameters that give up this minimum values are chloride ion concentration of 0.36 mol, pH value of 9.36 and exposure time of 10.28 hr. From the 'F' ratio value, it can be resulted that the exposure time is contributing the minor factor to corrosion attack, followed by pH value and Cl- ion concentration for the range considered in this investigation.



(a) Interaction effect of Cl- ion concentration and pH.



(b) Interaction effect of Cl- ion concentration and immersion time.



(c) Interaction effect of pH and immersion time.

Fig. 4 : Response surface graphs and contour plots

Table 5 : Validation test results

Sl. No.	Chloride ion concentration, (mol)	pH	Exposure time, (hr)	Actual corrosion rate (mm/year)	Predicted corrosion rate (mm/year)	Variation (%)
1	0.4	5	6	0.0040	0.0039	+2.53
2	0.5	4	11	0.0037	0.0036	+2.74
3	0.9	9	7	0.0041	0.0042	-2.41

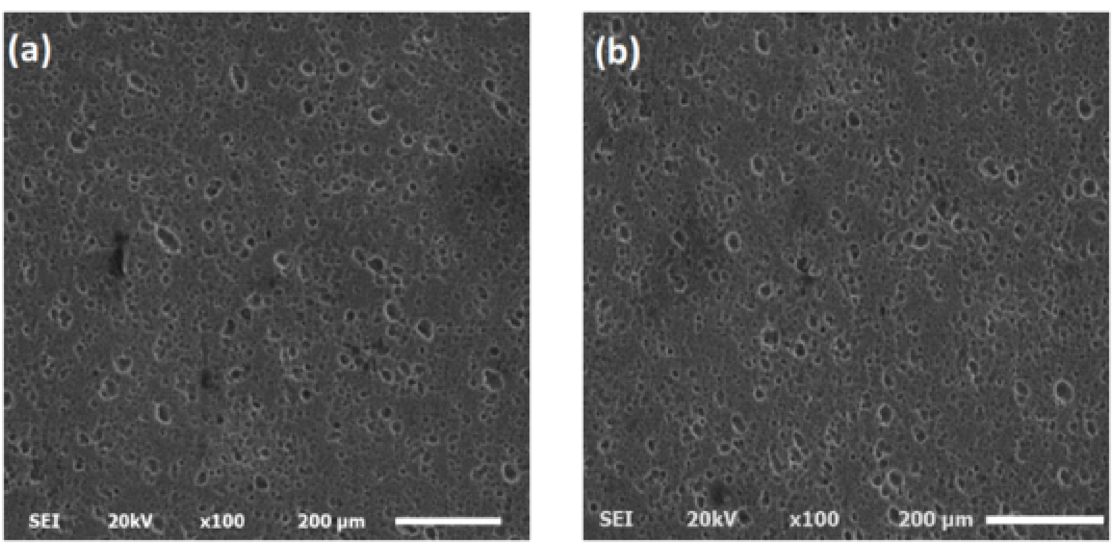


Fig. 5 : Scanning electron micrograph of the corrosion test specimens (a) Minimum corrosion attack (b) Maximum corrosion attack.

To confirm the developed relationship, three confirmation tests were conducted by varying the concentration of Cl- ion, pH and exposure time; the values were selected randomly within the range of test parameters presented in **Table 2**. The actual response was calculated from the average of three calculated results. **Table 5** represents the predicted values, experimental values and the variations. The confirmation test results exposed that the established empirical relationship is quite precise as the deviation is ±3 %.

SEM images of the corrosion test specimens exhibited minimum and maximum corrosion rate are shown in **Fig.5**. In both the conditions, the stir zone of FSW joint doesn't corrode severely; this may be due to passivation of oxide films on the surface shielding the metal from corrosion. From all these conditions, minimum corrosion attack is observed at a Cl- ion concentration of 0.36 mol, pH of 9.38 and exposure time of 10.38 hr and maximum corrosion attack is observed at a Cl- ion concentration of 0.84 mol, pH of 4.62 and exposure time of 5.62 hr.

6.0 CONCLUSIONS

(i) Developed empirical relationship is capable of predicting the rate of corrosion at the stir zone of Al alloy joints with 95% of confidence level.

(ii) Response surface method was used to determine the combinations of pitting corrosion test parameters to attain minimum rate of corrosion in the SZ of Al alloy joints.

(iii) From this investigation, it is found that the minimum corrosion rate of 0.001089 mm/year was attained under the test conditions of 0.36 mol chloride ion concentration, 9.36 pH and 10.28 hr exposure time.

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