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An Investigation on Application of Experimental Design and Linear Regression Technique to Predict Pitting Potential of Stainless Steel

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This study using experimental design and linear regression technique was implemented in order to predict the pitting potential of stainless steel in marine environments, with the target materials being AL-6XN and STS 316L. The various variables (inputs) which affect stainless steel's pitting potential included the pitting resistance equivalent number (PRNE), temperature, pH, Cl^- concentration, sulfate levels, and nitrate levels. Among them, significant factors affecting pitting potential were chosen through an experimental design method (screening design, full factor design, analysis of variance). The potentiodynamic polarization test was performed based on the experimental design, including significant factor levels. From these testing methods, a total 32 polarization curves were obtained, which were used as training data for the linear regression model. As a result of the model's validation, it showed an acceptable prediction performance, which was statistically significant within the 95% confidence level. The linear regression model based on the full factorial design and ANOVA also showed a high confidence level in the prediction of pitting potential. This study confirmed the possibility to predict the pitting potential of stainless steel according to various variables used with experimental linear regression design.

Keywords: *Experimental design, Linear regression, Stainless steel, Pitting potential*

1. Introduction

Austenitic stainless steel is defined as more than 11% Cr-containing steel. It has excellent corrosion resistance as it forms a natural oxide film in the air. A so-called passivation film is formed. However, in an extremely corrosive environment such as a marine environment, local corrosion occurs due to the destruction of the passive film. It is a major concern in various industries. The reason is that this degrades the mechanical properties, and the durability of the structure decreases.

The pitting potential is a representative index to evaluate the localized corrosion sensitivity for stainless steel in an application environment. If the surface potential is shifted in a noble direction than the pitting potential in the application environment, it continuously causes severe localized corrosion such as pitting and crevice. Therefore, it is necessary to predict the pitting potential in the design and application environment in advance. In addition, if it is possible to select a key among various corrosion factors that may occur in the environment, it is possible to reduce the sensitivity of stainless steel to localized corrosion by controlling the factor. In previous studies, various studies have been conducted on the factors affecting the pitting potential of stainless steel. The general effects of each factor on the passive film properties of stainless steel are as follows. Cl^- ion in seawater is a representative factor that destroys the passive film [1], and various models for passivity breakdown have been suggested. An increase of hydrogen ions (pH decrease) in a solution interferes with the dehydrogenation reaction required for passive growth [2]. By contrast, a strongly alkaline environment improves the passive film performance for localized corrosion [3]. Sulfate and nitrate are known to act as inhibitors against localized corrosion [4]. Bobić *et al.* [5] studied the pitting properties of AISI 304 according to the addition of sulfate and nitrate in a natural seawater solution (0.2 mol/dm^3 NaCl), and reported that sulfate and nitrate had a positive effect on the pitting potential and critical pitting temperature (CPT). By contrast, Moyaed *et al.* [6] examined the changes of CPT according to the addition of sulfate (0, 0.2, 0.5, 0.75 M) in a 1M NaCl solution for 904L, and reported that the CPT decreased by up to 12 °C with the

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Table 1. Chemical composition of the materials

	C	Si	Mn	S	P	Ni	Cr	Mo	Cu	N
STS 316L	0.01	0.56	1.06	0.001	0.03	10.07	16.17	2.05	-	0.001
AL-6XN	0.02	0.27	0.56	0.03	0.01	24.8	20.2	6.83	0.61	-

addition of sulfate. However, most studies are based on single variables. From the results, it is very difficult to predict the pitting potential in an environment in which various variables act in a complex. In addition, it is very difficult to interpret a problem through human abilities. To solve this problem, a data-based prediction model using statistical techniques and machine learning is being applied in the industrial field.

The experimental design was first applied to the agricultural field by Ronald Fisher in the 1920s, and it has been widely applied in various industrial fields since then. Among the various experimental design methods, the full factorial design can identify the main effects, significance, and interactions between each factor. In ASTM G16-05, the two-level full factorial design is introduced as an excellent method for evaluating the influence of various factors on metal corrosion. In addition, the significance for factors can be verified through analysis of variance (ANOVA) on the response value from the full factorial design.

In machine learning, regression analysis is to explain the tendency of variables. In other words, it is to investigate the functional relationship between variables and to derive a mathematical model. It is divided into linear and nonlinear regression. Among them, linear regression is to generate a predictive function model through a 'linear model' that assumes that the relationship between various variables is linear. This can show very high predictive performance in a problem in which the relationship between variables is linear.

This study aims to predict the pitting potential with variables, and the process consisted of two steps. First, it is to determine the significant factor affecting the pitting potential among corrosive factor candidates. This step is to select a significant factor among various variables by using factor design, one of the experimental design methods. At this stage, full factorial design and ANOVA were applied. Second, it is to derive a linear regression model based on the engineering experiment data (training data) with a full factorial design. Finally, it is to confirm the possibility of predicting the pitting potential through the linear regression model.

2. Experimental Methods

2.1 Materials

The chemical composition of STS 316L and AL-6XN are shown in Table 1. The pitting resistance equivalent number (PREN) can be calculated using equation (1).

$$PREN = \%Cr + 3.3(\%Mo) + 16(\%N) \quad (1)$$

The PREN of STS 316L and AL-6XN were calculated as 24 and 45, respectively.

2.2 Potentiodynamic polarization curve

To measure the pitting potential at the factor level on the full factorial design, a potentiodynamic polarization test was performed in accordance with ASTM G61-86. For the working electrode, a specimen was polished to SiC paper #600, and the exposure area was 1.36 cm^2. A three-electrode corrosion cell was configured using Ag/AgCl sat. KCl for the reference electrode and a 20 mm × 20 mm platinum mesh for the counter electrode. The initial stabilization time was set to 3,600 s. Polarization was performed at a scan rate of 0.5 mV/s until the potential corresponding to a current density of 1.0 mA/cm^2 at the open circuit potential (OCP) of –0.25 V. Then reverse polarization was performed until the OCP. The pitting potential was measured at a point where the current density increased sharply after the current density stagnation section (passivation area). As needed, the reverse

Table 2. Designed factors and their levels for screening design

Factors	Unit	Level	
		Min. (-1)	Max. (1)
Temperature (A)	℃	20	60
Electrolyte (B)		Fresh water	Seawater (3.5% NaCl)
pH (C)		Acid(2)	Alkali(12)
Sulfate (D)	g/L	0	2
Nitrate (E)	g/L	0	2

polarization was not indicated in some figures.

2.3 Screening design

The statistical analysis for the data was performed using Minitab19® software. Experimental design methods are largely divided into screening and optimization (full factorial design) methods. In particular, the screening design aims to screen factors that are considered to have a significant influence among the many factors. Therefore, before full factorial design, a screening design was performed for STS 316L to screen major factors influencing the pitting characteristics in various candidate factors. The factors used in screening design with maximum (+1) and minimum (-1) levels are listed in Table 2. It was composed of five factors and two levels, and the detail is shown in Table 3.

2.4 Full factorial design and ANOVA

Full factorial design means that the experiment is performed in all combinations of the factors and is appropriate for determining interactions effect as well as the main effect. This study describes a two-level full factorial design for major factors selected in screening design. A full factorial model composed of four factors and two-levels, including the pitting resistance index of stainless steel besides environmental

Table 3. Screen design matrix

Row	Factors					E_{pit}
	Temp., °C	Electrolyte	pH	Sulfate, g/L	Nitrate, g/L	
1	60	Seawater	Acid	2	0	0.114
2	40	Fresh water	Acid	0	0	0.426
3	20	Seawater	Alkali	0	1	0.323
4	40	Fresh water	Acid	1	1	0.8
5	60	Fresh water	Alkali	0	0	0.651
6	20	Seawater	Acid	1	0	0.395
7	40	Seawater	Alkali	1	1	0.365
8	40	Seawater	Alkali	2	2	0.344
9	20	Fresh water	Alkali	0	2	0.761
10	60	Fresh water	Acid	2	1	0.26
11	20	Seawater	Acid	2	2	0.316
12	20	Fresh water	Alkali	2	0	0.756
13	60	Seawater	Acid	0	2	0.131
14	60	Fresh water	Alkali	1	2	0.669

Table 4. Full factorial design matrix

Row	Block	Factors				E_{pit}
		PREN	Temp., °C	Cl^-, g/L	pH	
1	1	45	20	40	2	0.863
2	1	45	60	20	2	0.744
3	1	24	60	40	2	0.04
4	1	24	60	40	6	0.193
5	1	45	20	20	2	0.819
6	1	45	20	40	6	0.984
7	1	24	20	20	2	0.432
8	1	45	60	20	6	0.984
9	1	45	20	20	6	0.977

Table 4. Continued

Row	Block	Factors				E_{pit}
		PREN	Temp., °C	Cl^-, g/L	pH	
10	1	24	60	20	2	0.171
11	1	45	60	40	6	0.975
12	1	24	20	40	6	0.411
13	1	45	60	40	2	0.729
14	1	24	20	40	2	0.341
15	1	24	20	20	6	0.328
16	1	24	60	20	6	0.2
17	2	45	20	20	6	0.974
18	2	45	60	20	2	0.743
19	2	24	60	20	2	0.176
20	2	45	20	20	2	0.824
21	2	24	20	40	6	0.39
22	2	45	20	40	2	0.83
23	2	45	60	40	2	0.761
24	2	45	60	40	6	0.958
25	2	45	20	40	6	0.974
26	2	45	60	20	6	0.988
27	2	24	20	20	2	0.411
28	2	24	60	20	6	0.166
29	2	24	20	40	2	0.364
30	2	24	60	40	6	0.132
31	2	24	60	40	2	0.128
32	2	24	20	20	6	0.372

factors was designed. The total number of experiments required for four factors and two-levels is 16. The experiment was performed twice for the same combination level of factors to achieve reliability. Block was set to remove errors by uncontrollable external factors. Finally, the full factorial design model was set up with 32 experiments in total, as shown in Table 4. The influence and significance of each factor were evaluated by analysis of variance (ANOVA) with a 95% confidence level.

3. Results and Discussion

3.1 Potentiodynamic polarization experiment

Fig. 1 shows the cyclic polarization curve of 316L under some conditions of the screening design model in Table 3. The cyclic polarization curves showed a passive behavior in the anodic polarization. Then the current density increased sharply at each critical pitting potential. In every condition, the open circuit potential was more negative than the pitting potential, but the value appeared differently at the level of

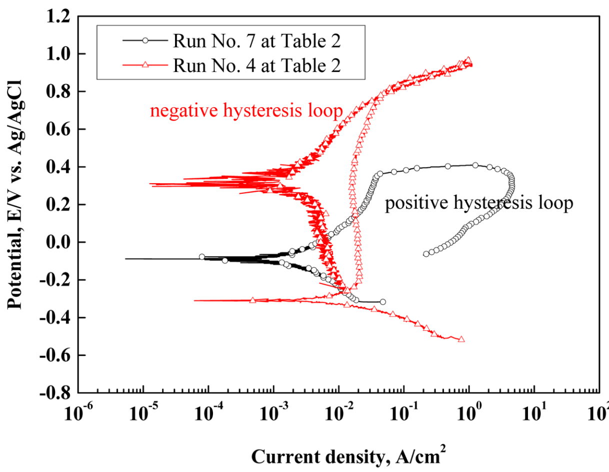

Fig. 1. Cyclic polarization curves of 316L for partial conditions of Table 3

each factor. As shown in Fig. 1, the hysteresis loop area tended to increase as the pitting potential was lowered. It is caused by a current density difference between forward and reverse directions at the same potential [7]. The hysteresis loop area means the stability of the passive film. The area tends to increase when the passive film is unstable [8].

3.2 Statistical analysis

Fig. 2 presents the Pareto chart for the experiment results of the screening design in Table 3. Pareto chart intuitively indicates the ratio of the variance dependent on each factor

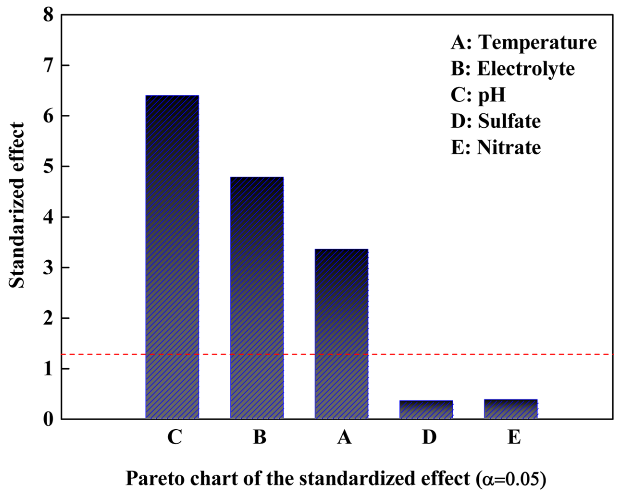

Fig. 2. Pareto chart of standardized effect for factors in screening design

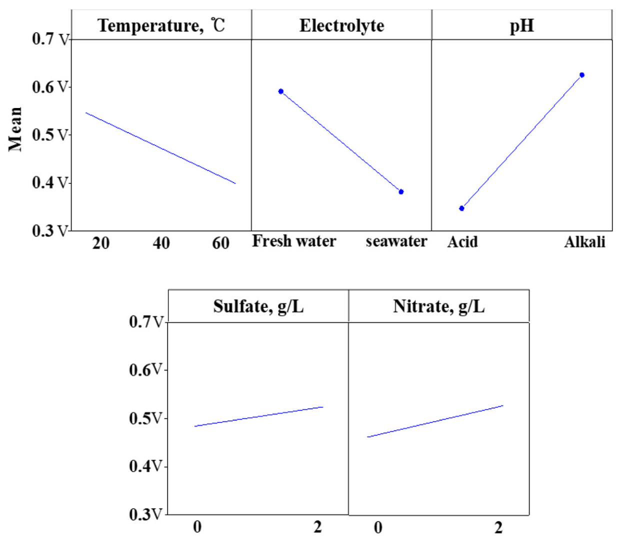

Fig. 3. Main effect plot for pitting potential in screening design

and the total variance. If the bar size for each factor is larger than the total average (1.397), the factor is determined to have a significant influence on the output. Interactions with low reliability were excluded because experiments are conducted for many factors in screening design. Furthermore, major factors were selected at the confidence interval (CI) of 80% due to the low resolution of experimental values. Significant factors that influenced the pitting potential were found to be electrolyte, temperature, and pH. The factor levels acting as the harsh environment on pitting in each factor were seawater (Cl^- ion), high temperature, and acidity. However, sulfate and nitrate had no significant effect on the pitting potential under the range of conditions in this study. The pitting potential tended to move slightly the noble direction by the addition of sulfate and nitrate. However, the value is significantly smaller than that of other factors and was insignificant in the ANOVA with 80% confidence interval.

Fig. 3 depicts the main effect diagram for factors. The factor with a large slope on the centerline (mean pitting potential) indicates a large influence on the pitting potential. The order of influence size for factors was B (electrolyte), C (pH), and A (temperature). The pitting potential with each factor level was negative in an acidic environment including Cl^- ions as expected and increased with the temperature. The major factors influencing the pitting potential based on the screening design were Cl^- ion concentration, pH change in acidic condition, and temperature.

Fig. 4 describes the polarization curve for some conditions of the full factorial model. Fig. 4a-c show the polarization curves for the maximum and minimum values of the characteristic factors. Other factors are at the same level. Fig. 4a is the polarization curve according to the level of the pitting resistance index. The current densities in each potential or area at different pitting resistance index values were similar, but as the pitting resistance index decreased, the pitting potential decreased rapidly. Fig. 4b is a polarization curve according to temperature change. As the temperature increased, the current density at each potential increased and the pitting potential decreased. Fig. 4c is a polarization curve according to the acidity change. As the acidity increased, the open circuit potential and pitting potential moved in the negative direction and the current density at each potential increased.

Fig. 5 shows some polarization curves with the Cl^- ion concentration. Fig. 5a is a polarization curve under the Cl^- ion concentration at 60 ºC and pH 2 for AL-6XN. As the

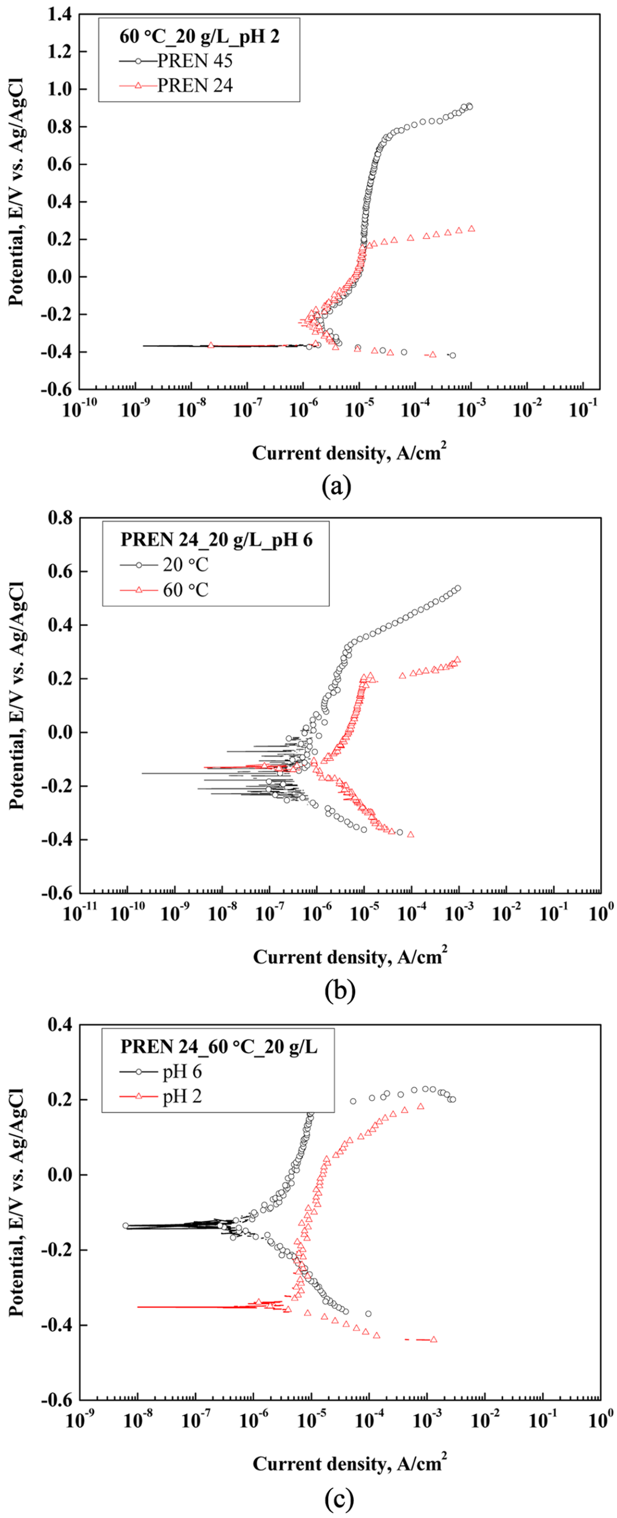

Fig. 4. Potentiodynamic polarization curves in different factor level: (a) PREN, (b) temperature, (c) pH

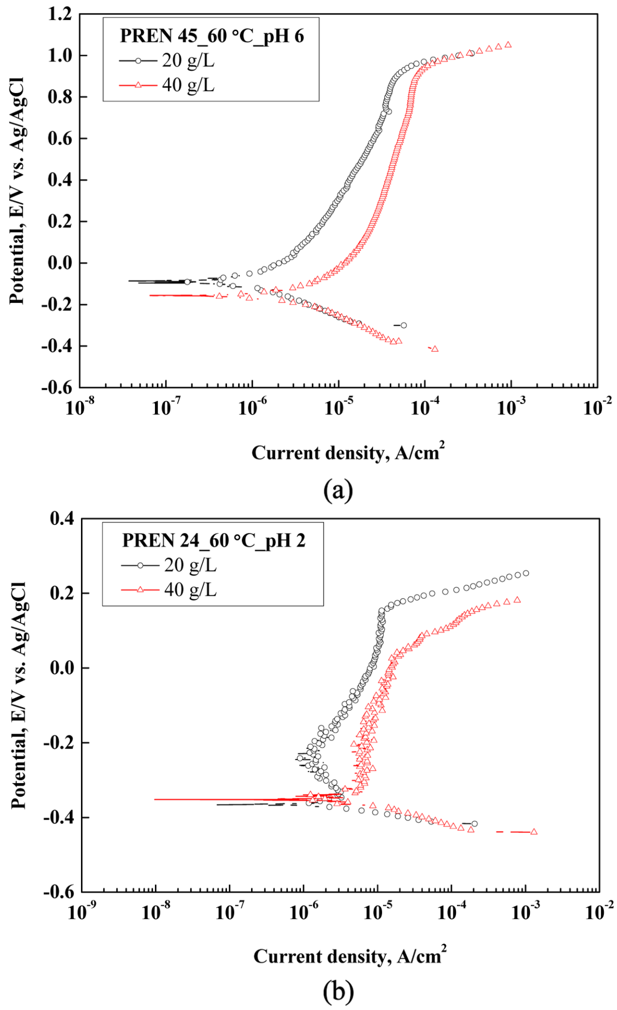

Fig. 5. Potentiodynamic polarization curves with different level of Cl^- concentration in (a) PREN 45_60 °C_pH 6 and (b) PREN 24_60°C_pH 2

Cl^- ion concentration increased, the current density at each potential increased. However, the open circuit potential and pitting potential showed approximate values. This is that each stainless steel has a range of critical Cl^- ion concentration required for passivity breakdown at a certain potential [3,9], and AL-6XN does not have a critical point in the Cl^- ion concentration range of 20 ~ 40 g/L. by contrast, in Fig. 5b, STS 316L showed a big difference in pitting potential with the Cl^- ion concentration. The pitting potentials were 0.171 V and 0.04 V at the Cl^- ion concentration of 20 g/L and 40 g/L. Ramana *et al.* [10] investigated the pitting potential with the change of Cl^- ion concentration in various acidic conditions at 60 °C. They reported that a rapid decrease of pitting potential occurred as the Cl^- ion concentration increased from 17,725 ppm to 35,450 ppm. This is very similar to the polarization test condition in Fig. 5b. Thus, 316L was found to have a critical Cl^- ion concentration in the range of 20 to 40 g/L in an acidic environment.

The influence of each factor was confirmed by ANOVA. It is a method of inter-group comparison of variance between two or more groups resulting from differences in the variance, total mean, and mean of each group in statistics. The mean of square (MS) is the sum of squares (SS) divided by the degree of freedom (DF). The F value is defined as the ratio of the mean squared error to the MS value. The F value indicates the influence of a factor in the model, and a higher F value is interpreted as a larger influence. The most important value in ANOVA is P-value. If the P-value is lower than 0.05 at 95% confidence level ($\alpha = 0.05$), the factor has significance for the response value.

Figs. 6 and 7 depicts the Pareto chart and main effect plot for pitting potential based on the ANOVA result of Table 5 with 95% confidence level. The insignificant terms at the confidence level 95% were pulled. The significant factors for the pitting potential were PREN (*A*), temperature (*B*), and pH (*D*). The interaction effect were found to be PREN •temperature (*A•B*) and PREN•pH (*A•D*). In contrast to the previous screening design where the Cl^- ions showed the largest effect, its effect was not significant compared with other factors in the full factorial model. This was confirmed through the polarization curve in Fig. 5. However, the mean pitting potential did not significantly differ in the range of full factorial design. Its effect was statistically insignificant in the pitting potential analysis when compared with other factors. The Cl^- ion concentration of 20 ~ 40 g/L did not have a significant effect on the pitting potential.

Fig. 8 describes the interaction effect plot between each factor. Interaction effect indicates that the effect of a factor depends on the level of another factor. The PREN confirmed the interaction effect with the temperature and pH at each level. The reason is that the PREN is a pitting resistance index for stainless steel, and the higher PREN, the higher the CPT tends to be [11]. The CPT of stainless steel can be different depending on the external environment and surface passivation properties(thickness, passivation processing).

Table 5. The result of ANOVA for full factorial design

Source	DF	Adj SS	Adj MS	F-value	P-value
Model	5	3.40789	0.68158	478.70	〈 0.001
Linear	3	3.28062	1.09354	768.04	〈 0.001
A	1	3.04551	3.04551	2138.98	〈 0.001
B	1	0.15208	0.15208	106.81	〈 0.001
D	1	0.08303	0.08303	58.31	〈 0.001
A·B	1	0.06845	0.06845	48.08	〈 0.001
A·D	1	0.05882	0.05882	41.31	〈 0.001
Error	26	0.03702	0.00142		
Total	31	3.44491			

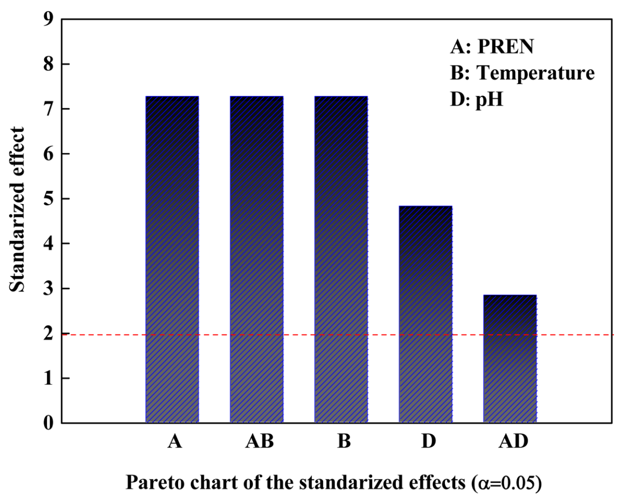

Fig. 6. Pareto chart of standardized effect for factors in the full factorial design

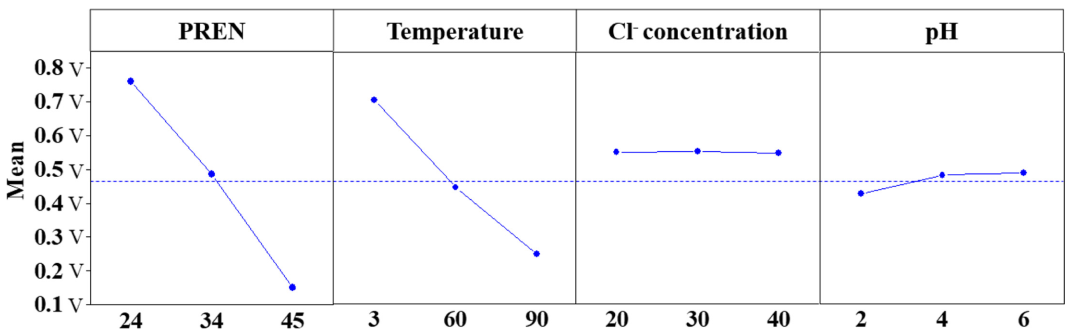

Fig. 7. Main effect plot for pitting potential in the full factorial design

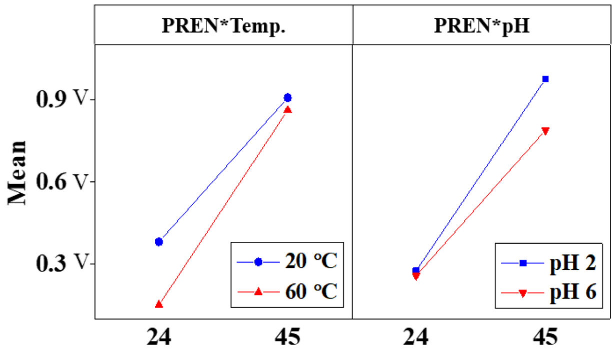

Fig. 8. Interaction effect plot for pitting potential in the full factorial design

However, previous study reported that the maximum CPT of 316L was 30 °C [12]. Abd El Meguid *et al.* [13] investigated the CPT of AL-6XN in a 4% NaCl solution at various ranges of pH (1 ~ 10) and temperatures (30 ~ 100 °C), and reported that the CPT was 64 ~ 89 °C. Therefore, it is considered that the interaction of between PREN and temperature depended on the CPT. Since the CPT of 316L is in the range of the 20 ~ 60 °C, the interaction effect on the pitting potential was large. However, the AL-6XN showed a small interaction effect with the temperature because the CPT is larger than the maximum level (60 °C).

3.3 Prediction of pitting potential using a linear regression model

A linear regression model was conducted to predict the pitting potential at the level of the factors. The influence of each factor is applied in the coefficient of the regression model, and the strong the influence, the larger value the coefficient is applied. As shown in equation (2), each factor level was normalized to have values of 0 and 1.

$$X = \frac{PREN - 24}{45 - 24},\ Y = \log(Cl^{-}_{g/L}),\ Z = \frac{pH - 2}{6 - 2} \quad (2)$$

Equation (3), shows a linear regression model for pitting potential based on the full factorial design and ANOVA result.

$$\begin{aligned} CPP &= 561.7 - 450.3X - 563.3Y + 0.016Z \\ &+ 452.3X \bullet Y + 0.1715X \bullet Z \end{aligned} \quad (3)$$

In the ANOVA with 95% confidence interval (P value < 0.05), the Cl^- ion concentration was not significant, thus factor and related interactions were pulled.

Fig. 9 depicts the normal probability diagram for pitting potential. It shows residuals for predicted value from the linear regression model and the actual value. If the residual is closer to the red line in Fig. 9, it means a higher fitness of the linear regression model. The linear regression model showed a very high fitness (R^2) of 98.63%.

To validate the linear regression model, potentiodynamic polarization test was conducted with the conditions in Table 6, and the result is shown in Fig. 10. In Table 6, validation tests 1 and 2 have the same factor level of test conditions included in the full factorial design model, and validation test 3 is factor level that are not included in the full factorial design. The value calculated from the regression model showed a very approximate value as the actual experimental value, and satisfied the confidence interval and prediction

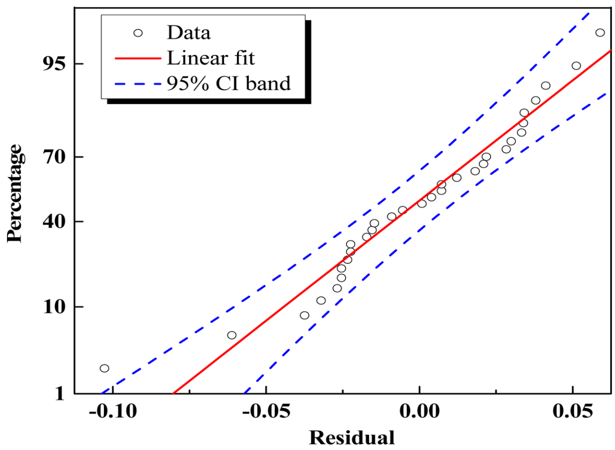

Fig. 9. Normal probability diagrams for pitting potential

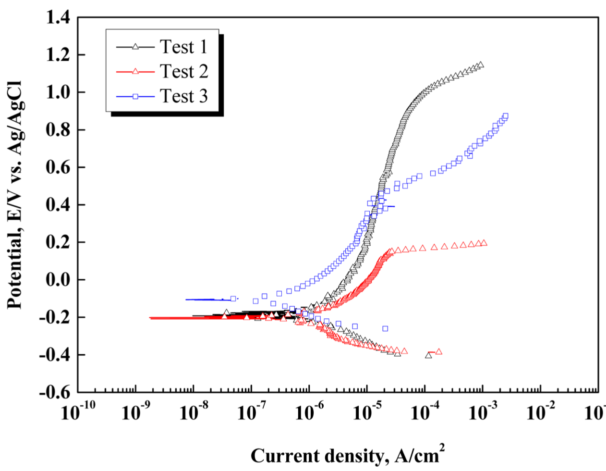

Fig. 10. Potentiodynamic polarization curves for validation test in Table 4

Table 6. Results of validation experiment for linear regression

	Factors			Regression value, V	Experimental value, V	CI 95%	PI 95%
	PREN	Temp, ℃	pH				
1	24	60	2	0.124	0.14	0.087 ~ 0.158	0.037 ~ 0.208
2	45	20	6	0.981	1.01	0.963 ~ 1.025	0.91 ~ 1.077
3	34	45	2	0.489	0.448	0.469 ~ 0.508	0.408 ~ 0.569

interval (PI) of 95%.

4. Conclusions

This study investigated the effects of environmental parameters on the pitting potential for 316L and AL-6XN using the experimental design and ANOVA. In the screening design, the main environmental factors that influence the pitting potential of austenitic stainless steel were temperature, pH, and Cl^- ions. The sulfate and nitrate were showed insignificant at 80% confidence level.

The pitting potential showed a very large effect in each environment according to the change of the pitting resistance index. The pitting potential was greatly affected by temperature and pH among the environmental parameters, and temperature showed the largest effect among them. The Cl^- ion concentration in the range of 20 ~ 40 g/L did not have a significant effect on the pitting potential.

The linear regression model based on the full factorial design and ANOVA had a high confidence level in the prediction of pitting potential.

The predictability of pitting potential for austenitic stainless steel was confirmed using experimental design and linear regression technique.

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