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Studying of Heat Treatment Influence on Corrosion Behavior of AA6061-T6 by Taguchi Method

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In this paper, the Taguchi method has been applied to optimize the heat treatment parameters for the corrosion resistance of 6061 aluminum alloy. The experimental design consisted of four parameters (aging temperature, aging time, quenching environment and NaCl concentration), each at three levels. Tafel polarization measurements were carried out to determine the corrosion resistance of the heat treatment samples. According to the mean of signal-to-noise ratio analysis, the corrosion resistance of AA6061-T6 was influenced significantly by the levels in the Taguchi orthogonalarray. The optimized parameters for corrosion resistance are 2 h for aging time, 200 °C for aging temperature, ice water for quenching media and environment with 0.5% for NaCl concentration. The percentage of contribution for each parameter was determined by the analysis of variance. The results showed that the NaCl concentration is the most significant parameter affecting the corrosion resistance of the AA6061.

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1. INTRODUCTION

Aluminum and its alloy are widely used for different applications in the industries and marine environment because of their excellent properties [1-3]. Generally, aluminum has excellent corrosion resistance due to its thin surface oxide film formation. Although, in an aggressive media, mostly chloride and acidic, the protective films breaks, however, the oxide coating is not totally removed but is thinned and recreate by the oxidation of the underlying metal [4-7]. Moreover, in this aggressive environment, the recreation of the thin film layer may be delayed and an active region is formed. Hence, pits are initiated and may spread further. AA6061-T6 aluminum alloy (solutionized and artificially aged) displays high strength, excellent extrudability, reasonable weldability and good corrosion resistance. This alloy finds extensive application in ship building and in the fabrication of tank containers for transporting various liquids, where is often joined during manufacturing process.

According to the best of our knowledge, the effect of aging and Cu content on intergranular corrosion (IGC) susceptibility of AA6061-T6 aluminum alloy is well documented. It was claimed that IGC susceptibility was a result of microgalvanic coupling between grain boundary precipitates and the adjacent depleted zone. The exact nature and properties of these precipitates in relation to IGC were not reported [8-12].

A traditional optimization approach would be to design experiments that identify all possible combinations for a given set of variables. This approach is named the full factorial design, and involves a large number of experiments, that can be costly and time consuming. Recently, the Taguchi method has been effectively applied to improve product quality and manufacturing process. The Taguchi method uses a special design of orthogonal arrays to investigate all the designed parameters with a minimum of experiments at a relatively low cost [13].

Many studies have been reported on corrosion behavior of aluminum and its alloys in marine

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environments [1-8]. However, very few have focused on the optimization of the heat treatment parameters on corrosion resistance of these alloys. In this study, the Taguchi method (by Qualitek-4 software) for design of experiment was used to optimize the heat treatment parameters (i.e. T6 process), for modification of corrosion resistance (i.e. E_{pit}) of AA6061-T6 aluminum alloy. This consideration led to the selection of four influential parameters, i.e. aging temperature, aging time, NaCl concentration, quenching environment with three different levels. The results of the parameter response analysis were used to derive the optimal levels combinations. Confirmation experiments were conducted to verify the analytical results. The percentage contribution of each parameter was determined by an analysis of variance (by Minitab V.14 program).

2. PROBLEM FORMULATION

The nominal composition of the alloy AA6061 (% by weight) is 1.23% Mg, 0.6% Si, 0.23% Cu, 0.46% Fe, 0.18% Cr, 0.06% Mn and the rest Al. The solution of heat treatment for received samples was done in furnace at 530 °C for 1.5 h in air, followed by quenching in various environments. After quenching, samples were artificially aged at three aging temperatures (180, 200, 220°C) for different aging times as summarized in Table 1. After any aging treatment, the alloy specimens were polishing, the working surfaces of samples were wet ground with emery paper up to 2000 grit (using a polishing machine model POLIMENT I, Buehler polisher) and then degreased with ethanol, cleaned with distilled water, and finally dried in air.

The "quality characteristic" of concern is the corrosion resistance of samples that was assessed by polarization tests carried out at 25 °C using an EG&G 273 A potentiostat with Power suite 2.20.0 software and its PAR calculations. A three electrode cell with the 6061-T6 aluminum samples as a working electrode, saturated calomel electrode as a reference electrode, and a platinum plate as a counter electrode was used in the tests.

A delay time of 6 hours applied to allow the specimens surface to achieve a steady state before the potential scan was commenced. The scan rate for the potentiodynamic polarization experiments was 1 mV/s. The potential scans in the NaCl electrolyte were started at 250 mV with respect to open circuit potential (OCP) and continued to +0 mV. Potentiodynamic polarization curves were recorded in NaCl solution. It was necessary to note that for AA6061-T6 aluminum alloy in NaCl solutions, the pitting potential (E_{pit}) was close to the corrosion potential (E_{corr}).

3. TAGUCHI DESIGN OF EXPERIMENT

3. 1. Design of Orthogonal Array and Signal-to-noise Analysis

Four parameters (i.e. aging temperature, aging time, NaCl concentration and quenching environment) with three levels were selected and were shown in Table 1.

The parameters and levels were used to design an orthogonal array L9 (34) for experimentation. Nine Taguchi experiments were conducted twice to ensure the credibility of experimental data for a signal-to-noise analysis. In process design, it is almost impossible to remove all errors caused by the variation of characteristics. An increase in the variance of corrosion resistance decreases the quality reliability of sample. To minimize the effect of corrosion resistance variation on the analysis of experimental data, the signal-to-noise (S/N) ratio was employed, which converts the trial result data into a value for the response to evaluate AA6061-T6 aluminum alloy samples quality in the optimum setting analysis. The S/N ratio consolidates several repetitions into one value, which determines the amount of variation present. This is because the S/N ratio can reflect both the mean and the variation of the quality characteristics. There are several S/N ratios available depending on the types of characteristics [13]: lower is the best (LB), nominal is the best (NB), and higher is the best (HB). In the present study, a corrosion resistance is treated as a characteristic value. Since the corrosion resistance of samples intended to be maximized, the S/N ratio for HB characteristics was selected, which can be calculated as follows:

$$S/N_{HB} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{R_{pi}^{2}}\right) \tag{1}$$

where, n is the repetition number of each experiment under the same condition for design parameters, and R_{pi} is the polarization resistance and E_{pit} of an individual measurement at the ith test. After calculating and plotting the mean S/N ratios at each level for various parameters, the optimal level, that is the largest S/N ratio among all levels of the parameters, can be determined.

TABLE 1. Parameters and levels used in this experiment

		Levels		
		Low (1)	**Medium (2)**	**High (3)**
A	Aging temperature (°C)	180	200	220
B	Aging time (h)	1	2	3
C	NaCl concentration (%)	0.5	1.5	3.5
D	Quenching environment	Ice water (0 °C)	Water (25 °C)	Oil

3. 2. Analysis of Variance (ANOVA) The analysis of variance (ANOVA) on the experimental results was carried out to evaluate the source of variation during corrosion process. Accordingly, it is relatively easy to identify the effect order of parameters on heat treatment and the contribution of parameters on corrosion resistance of samples. In this study, variation due to both the four parameters and the possible error was taken into consideration. The ANOVA was established based on the sum of the square (SS), the degree of freedom (D), the variance (V), and the percentage of the contribution to the total variation (P). The five parameters symbols typically used in ANOVA [14, 15] are described below:

1. Sum of squares (SS). SS_p denotes the sum of squares of parameters A, B, C, and D; SS_e denotes the error sum of squares; SS_T denotes the total sum of squares. The total sum of square SS_T from S/N ratio can be calculated from S/N ratio as following:

$$SS_T = \sum_i^m \eta^2 - \frac{1}{m}\left(\sum_{i=1}^m \eta_i\right)^2 \quad (2)$$

where, m is the total number of the experiments, and η_i is the S/N ratio at the ith test. The sum of squares from the tested parameters, SS_p, can be calculated as:

$$SS_p = \sum_{j=1}^p \frac{(S_{\eta j})^2}{t} - \frac{1}{m}\left(\sum_{i=1}^m \eta_i\right)^2 \quad (3)$$

where, p represents one of the tested parameters, j the level number of this specific parameter p, t the repetition of each level of the parameter p, and $S_{\eta i}$ the sum of the S/N ratio involving this parameter and level j.

2. Degree of freedom (D). D denotes the number of independent variables. The degree of freedom for each parameter (D_p) is the number of its levels minus one. The total degree of freedom (D_T) is the total number of the result data points minus one. The degree of freedom for the error (D_e) is the number of the total degrees of freedom minus the total of degree of freedom for each parameter.

3. Variance (V). Variance is defined as SS_p divided by D_p as fallowing:

$$V_p(\%) = \frac{SS_p}{D_p} \times 100 \quad (4)$$

4. The corrected sum of squares (SS'_p). SS'_p is defined as the sum of squares of parameters minus the error variance times the degree of freedom of each parameter:

$$SS'_p = SS_p - D_p V_e \quad (5)$$

5. Percentage of the contribution to the total variation (P). P_p denotes the percentage of the total variance of each individual parameter:

$$P_p(\%) = \frac{SS'_p}{SS_T} \times 100 \quad (6)$$

4. RESULTS AND DISCUSSIONS

4. 1. Corrosion Resistance of AA6061-T6 Aluminum Alloy The calculation of the corrosion resistance of AA6061-T6 aluminum alloy samples is based on the corrosion potential, the corrosion current density, the anodic/cathodicTafel slopes ($\beta_a$and β_c) and pitting potential (E_{pit}) which were derived from the measured polarization curves. Based on the approximately linear polarization at the corrosion potential (E_{corr}), Table 2 shows value of corrosion resistance (R_p) which was determined from the relationship [12]:

$$R_p = \frac{\beta_a \beta_c}{2.3 i_{corr}(\beta_a + \beta_c)} \quad (7)$$

4. 2. Determination of Optimal Conditions Based on Equation (1), two corrosion resistance measurements for each experiment were converted into one S/N ratio. Table 3 compares the calculated mean S/N ratios with the corrosion resistance and Epit data and level numbers. In the following discussion, the S/N ratios are employed as a response index to compare the corrosion resistance and E_{pit} of different samples instead of directly using the values of corrosion resistance and Epit. The response of each parameter to its individual level was calculated by averaging the S/N ratios of all experiments at each level for each parameter. The determined parameter responses i.e. R (max-min) = high S/N-low S/N for each parameter, are summarized in Tables 4 and 5.

Figure 1 shows S/N response graph for corrosion resistance. The greater is the S/N ratio, the smaller is the varience of R_p around the desired value. Therefore, the optimum condition is A2, B1, C1 and D1. In other words, based on the S/N ratio, the optimal conditions for corrosion resistance is the A at level 2 (180°C), B at level 1 (2 h), C at level 1 (0.5% of NaCl) and D at level 1(ice water).

Figure 2 shows S/N response graph for pitting potential. By attention to negative values of E_{pit}, The smaller is the S/N ratio, the greater is the varience of E_{pit} around the desired value. Therefore, the optimum condition is A2, B1, C1 and D1. In other words, based on the S/N ratio, the optimal conditions for pitting resistance the A at level 2 (180°C), B at level 1 (2 h), C at level 1 (0.5% of NaCl) and D at level 1(ice water).

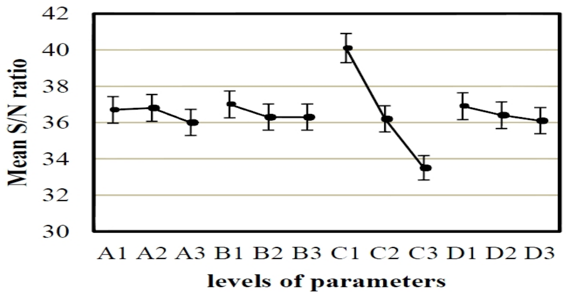

Figure 1. S/N graph for corrosion resistance (Rp), A: aging temperature (°C), B: aging time (h), C: NaCl concentration (%) and D: quenching environment.

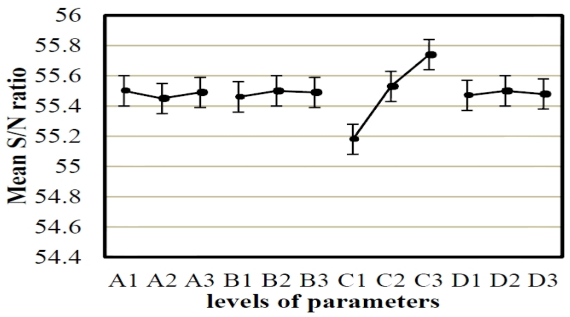

Figure 2. S/N graph for corrosion resistance (E_{pit}), A: aging temperature (°C), B: aging time (h), C: NaCl concentration (%) and D: quenching environment.

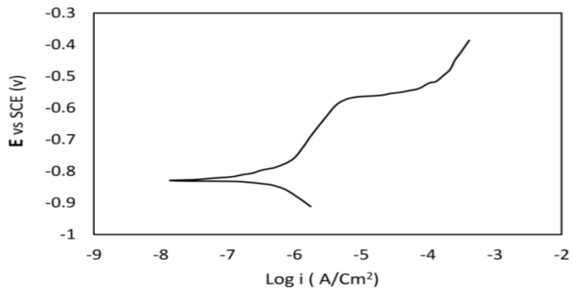

Figure 3. Polarization curve of 6061-T6 aluminum alloy in optimal condition.

4. 3. Parameter Contributions

The contribution of each parameter on the corrosion resistance and Epit of samples can be determined by performing analysis of variance based on Equations (2)–(6). The results of analysis of variance (ANOVA) are summarized in Tables 6 and 7.

The data given in Table 6 shows that the contribution of the four parameters, i.e. aging temperature, aging time, NaCl concentration and quenching environment is 1.55%, 1.62%, 95.5% and 1.33%, respectively. The contribution of NaCl concentration (95.5%) is more than the sum of the contributions of all the other three parameters (4.5%). Moreover, the contribution of aging time is more than aging temperature.

The data given in Table 7 shows that contribution of NaCl concentration (97.4%) is more than the sum of the contributions of all the other three parameters (2.6%). Also, the contribution of aging temperature is more than aging time. It is evident from Tables 6 and 7 that ANOVA analysis not only specifies how important a parameter is to the corrosion resistance of coatings by numbers but also shows their relative effect. By ranking their relative contributions, the sequence of the four parameters affecting the corrosion resistance is NaCl concentration, aging temperature, aging time and quenching environment. It is also worthwhile mentioning that, in the ANOVA analysis, if the percentage error (P_e) contribution to the total variance is lower than 15%, no important parameter is missing in the experimental design. In contrast, if the percent contribution of the error exceeds 50%, certain significant parameters have been overlooked and the experiments must be re-designed [16]. As shown in Tables 6 and 7 the percentage error (P_e) is 0%. This indicates that no significant parameters are missing in the experimental design.

4. 4. Prediction and Confirmation Experiments

Once the optimal level of the design parameters has been selected, the final step is to predict and verify the improvement of the quality characteristic using the optimal level of the design parameters. The predicted S/N ratio using the optimal level of the design parameters can be calculated as [17]:

$$S/N\ predicted = S/N_m + \{(S/N1 - S/N_m) + (S/N2 - S/N_m) + (S/N3 - S/N_m) + (S/N4 - S/N_m)\} \quad (8)$$

where, S/N_m is the total mean S/N ratio and S/N_1, S/N_2, S/N_3 and S/N_4 is the mean S/N ratio at the optimal level.

In the case of R_p and E_{pit}, the values of S/N_m calculated from Tables 4 and 5 are about 36.49 and 55.48. In addition, S/N ratio for A2, B1, C1 and D1 can be obtained from Tables 4 and 5. Therefore, the predicted S/N ratio for R_p, i.e. 41.23, and for E_{pit}, i.e. 55.12, can then be obtained and the corresponding estimated R_p and E_{pit} can also be calculated by $S/N = -10\ log(y^2)$. Finally, estimated R_p (115.2 KΩ.cm^2) and E_{pit} (-570 mV) can be obtained.

The confirmation experiment is the final step in verifying the conclusions from the previous round of experimentation. If the results of the confirmation runs are not consistent with the expected conclusions, a new Taguchi method design is required.

The confirmation experiment was performed by setting the experimental condition of the four parameters as: 200 °C for aging temperature; 1 hour for aging time; 0.5% for NaCl concentration and ice water for quenching environment that are given in Figure 3.

TABLE 2. Results of Tafel and cyclic polarization tests.

Experiment no.	E_{corr} (mv)		i_{corr} (μA/cm^2)		βc- (mV/decade)		β_a (mv/decade)		E_{pit} (mV)		R_p (kΩ.cm^2)	
	Test1	Test2	Test1	Test2	Test1	Test2	Test1	Test1	Test1	Test2	Test1	Test2
#1	-825	-815	0.207	0.195	0.130	0.125	0.090	0.085	-571	-575	111.8	112.8
#2	-809	-820	0.360	0.340	0.125	0.117	0.087	0.084	-597	-605	61.8	62.9
#3	-840	-832	0.512	0.482	0.117	0.110	0.089	0.077	-611	619	44.6	45.5
#4	-815	-810	0.353	0.305	0.123	0.120	0.086	0.076	-593	-595	65.7	66.4
#5	-828	-832	0.465	0.415	0.120	0.116	0.089	0.079	-608	-612	48.8	49.2
#6	-820	-829	0.220	0.210	0.128	0.123	0.084	0.082	-570	-580	103.8	102.5
#7	-824	-836	0.428	0.415	0.115	0.110	0.087	0.075	-612	-614	47.2	46.7
#8	-815	-809	0.235	0.243	0.119	0.128	0.085	0.087	-570	-582	92.9	93.3
#9	-819	-825	0.375	0.360	0.125	0.120	0.090	0.089	-590	-606	60.6	61.7

TABLE 3. The S/N ratios

Experiment no.	A	B	C	D	R_p	*S/N* ratio	E_{pit}	*S/N* ratio
#1	1	1	1	1	112	41.1	-573	55.16
#2	1	2	2	2	62	35.8	-601	55.58
#3	1	3	3	3	45	33.0	-615	55.77
#4	2	1	2	3	66	36.4	-594	55.47
#5	2	2	3	1	49	34.1	-610	55.71
#6	2	3	1	2	102	40.0	-575	55.18
#7	3	1	3	2	47	33.5	-613	55.74
#8	3	2	1	3	93	39.1	-576	55.21
#9	3	3	2	1	61	35.7	-598	55.53

TABLE 4. The parameter response (R_p)

Symbol	Mean S/N ratio			
	Level (1)	Level (2)	Level (3)	R (max – min)
A	36.7	36.8	36.0	0.80
B	37.0	36.3	36.3	0.70
C	40.1	36.2	33.5	6.60
D	36.9	36.4	36.1	0.77

TABLE 5. The parameter response (E_{pit})

Symbol	Mean *S/N*ratio			
	Level (1)	Level (2)	Level (3)	R (max – min)
A	55.50	55.45	55.49	0.05
B	55.46	55.50	55.49	0.04
C	55.18	55.53	55.74	0.56
D	55.47	55.50	55.48	0.03

TABLE 6: Results of the ANOVA for corrosion resistance (R_p)

Symbol	Degree of freedom (D)	Sum of squares (SS¢)	Variance (V)	Corrected sum of squares (SS¢)	Contribution (P, %)	Rank
A	2	1.045	0.5225	1.045	1.55	3
B	2	1.091	0.5457	1.091	1.62	2
C	2	64.36	32.18	64.36	95.5	1
D	2	0.904	0.452	0.904	1.33	4
Error	9	0	0			
Total	17	67.39				

TABLE 7. Results of the ANOVA for pitting potential (E_{pit})

Symbol	Degree of freedom (D)	Sum of squares (SS¢)	Variance (V)	Corrected sum of squares (SS¢)	Contribution (P, %)	Rank
A	2	0.0042	0.0021	0.0042	0.87	2
B	2	0.0027	0.0014	0.0027	0.56	3
C	2	0.47	0.235	0.47	97.4	1
D	2	0.0015	0.0008	0.0015	0.31	4
Error	9	0	0			
Total	17	0.4825				

TABLE 8: Results of cycle polarization test for confirmation run.

Samples	R_p/(kΩ.cm^2)	E_{pit} / (mv)
Prediction	115.2	-570
Experiment	١١4.1	-575

Table 8 shows the comparison of the predicted R_p and Epit with the experimental results using the optimal conditions. As can be seen there is a good agreement between the predicted and experimental corrosion resistance. Consequently, corrosion resistance in the heat treatment T6 of AA6061 Al alloy can be increased and improved through the Taguchi method approach.

5. CONCLUSION

The Taguchi method for the design of experiment has been used for optimizing heat treatment process parameters on corrosion behavior. The NaCl concentration is found to be the major parameter affecting the corrosion resistance of the samples. Also, the aging temperature as well as aging time have a similar and to be the second important parameters affecting the corrosion resistance of the samples. However, quenching environment have smallest effect on the corrosion resistance and pitting potential. The percentage contributions of the NaCl concentration, aging time, aging temperature and quenching environment on the corrosion resistance are 95.5, 1.62, 1.55 and 1.33%, respectively. The contribution of NaCl concentration is more than the sum of the contributions of all the other three parameters. It is evident that, among the selected parameters, NaCl concentration has the major influence on the corrosion resistance of AA6061-T6 Al alloy. It can be seen that the aging time is second important parameter that effects on corrosion resistance of the AA6061-T6 while, in pitting potential the aging temperature is second important parameter that effects on E_{pit}. The effect of NaCl concentration on E_{pit} (97.4%) is more than R_p (95.5%). The optimized processing parameters are 200 °C for aging temperature; 1 h for aging time; 0.5% for NaCl concentration and ice water for quenching environment. There is good agreement between the predicted and experimental corrosion resistance.

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Analysis of Variance

چکیده

در این مقاله از روش تاگوچی برای بهینه‌سازی پارامترهای عملیات حرارتی به منظور بهبود مقاومت خوردگی آلیاژ 6061 آلومینیم استفاده شد. طراحی آزمایش با استفاده از چهار پارامتر (دمای پیرسازی، زمان پیرسازی، محیط خنک کننده و غلظت نمک طعام) هر کدام حاوی سه سطح صورت گرفت. آنالیز پلاریزاسیون تافل برای تعیین مقاومت به خوردگی نمونه‌ها پس از عملیات حرارتی استفاده گردید. با استفاده از آنالیز میانگین نسبت سیگنال به نویز، مقاومت خوردگی نمونه‌هایی از آرایه های ارتوگونال در سطوح متفاوت روش تاگوچی مشخص گردید. زمان پیرسازی 2 ساعت در دمای پیرسازی 200 درجه سانتی گراد با محیط خنک کننده آب یخ در محیط با 0/5 درصد وزنی نمک طعام شرایط بهینه برای بهترین مقاومت به خوردگی را دارد. سهم هریک از پارامترها با استفاده از آنالیز واریانس مشخص گردید. نتایج نشان داد که غلظت نمک طعام مهمترین پارامتر اثرگذار بر مقاومت خوردگی آلیاژ AA6061 است.

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