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Optimal Parameters in Precipitation Hardening of 6061 Aluminium Alloy Using Box-Behnken Design

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Abstract

Precipitation hardening process is important to increase the hardness of 6061 aluminium alloy. In order to achieve the desired hardness, this process has several controlled parameters including solution soaking temperature, solution soaking time, aging temperature and aging time. The identification of each appropriate parameter is important in the precipitation hardening process of aluminium alloy. Because there has been no method or exact pattern, therefore this research work applies the response surface methodology with Box-Behnken design to determine these appropriate parameters. The experimental result is found that there are three factors significantly affecting on the hardness obtained from the precipitation hardening process. The experimental data is well fit a quadratic model due to high coefficient of determination (R^2=0.92). When this regression model is taken to construct the three-dimensional surface response plot and the contour plot, it is found that the optimum condition is the solution treatment time of 5 hours and 18 minutes, aging temperature of 181°C and the aging time of 10 hours which yield the maximum predicted Vicker hardness of 132.5 HV.

Keywords : *6061 aluminium alloy, Box-Behnken design, precipitation hardening*

1. Introduction

The application of aluminium alloy in industries continuously increases, especially after 1960. It has been employed more than other metals, the second order compared to steel (1) because it has light weight, easy fabrication, high strength to weight ratio and no toxic for health as well as low cost. Moreover, the aluminium alloy is capable of self rust resistance or passive state, high deformability due to having face-center-cubic (FCC) crystal structure, which planes can slip to many directions while carried out by applied forces (2, 3).

The increase of hardness or strength in aluminium alloy takes place from the secondary particles, which precipitates and evenly distributes on the matrix of aluminium alloy through the precipitation hardening. The natural aging is performed at room temperature and the artificial aging is performed under controlled temperature (4-7). The heat-treatable aluminium alloy must have major alloying elements, which can be highly dissolved at high temperature and their dissolution rate are considerably reduced at low temperature to cause metallic compound separated out from the base structure. The different major alloying elements in various aluminium alloy provide different compounds like magnesium silicate (Mg_2Si) coming from magnesium-silicon aluminium alloy (6000 series) etc.

The 6061 aluminium alloy containing major alloying element as 0.8-1.2% magnesium and 0.4-0.8 % silicon can be strengthened or hardened by both heat treatment and cold working and has medium strength, corrosion resistance, high deformability and good weldability (8). These cause it to be widely used in various industries such as automobiles, ocean ships, general constructions, and secondary industries.

The precipitation hardening is a process of heat treatment for hardening alloys, divided into three stages consisting of solid solution soaking, quenching and aging at a given temperature. For 6061 aluminium alloy having addition of magnesium and silicon, it tends to apply solution soaking temperature of 529 ^{o}C (2) in order to form a homogeneous solid solution and is then dropped into water to have retained magnesium and silicon in a large quantity in the solid solution phase. Subsequently, it is artificial aged to precipitate Mg_2Si compounds within the alloy structure. These crystals forming coherent forces with the structure cause a distortion of the atom lines yielding the movement of atoms to be difficult while the alloy is being subjected to the applied force from the outside. This result enhances the mechanical properties, particularly hardness and strength of alloy (9, 10).

The hardness and the strength are dependable on the soaking temperature in addition to it will depend on the aging temperature and aging time due to the mechanism of precipitation as diffusion. However, if temperature is too high or time is too long will cause to reduce the hardness and the strength as well because of non-coherent precipitation (11). Although currently, there are several study results involving the heat treatment by precipitation of 6061 aluminium alloy, most are the study using one-factor-at-a-time experiment, which is an experiment that must be conducted many repeated runs. It is low efficient method and not able to assess the interaction influence of various factors. Moreover, the application of statistical principles in design of experiment has been limited. This interesting research is to study the effect of factors on the hardness, determine the optimal parameters in the precipitation hardening process, and construct a fit model to predict the hardness by using the Box-Behnken design.

2. Application of Box-Behnken Design (12, 13)

The Box-Behnken experimental design, developed by Box and Behnken in 1980, is a useful method for developing second-order response surface models. It is based on the construction of balanced incompleted block designs and applicable when at least there are three levels for each factor crucial to the problem of interest. In Box-Behnken experimental design, the level of each factor is fixed at the center level while combinations of all levels of the other factors are applied. Table 1 illustrates the concept, the level of factor C is fixed and then, the combinations of all levels of factor A and B are applied and subsequently, the same procedures are performed for the factors B and A, respectively. The last column of the design matrix contains center point values.

Table 1. Three-factor Box-Behnken experimental designs.

Rank	Box-Behnken Experimental Design		
	A	B	C
1	-1	-1	0
2	1	-1	0
3	-1	1	0
4	1	1	0
5	-1	0	-1
6	1	0	-1
7	-1	0	1
8	1	0	1
9	0	-1	-1
10	0	1	-1
11	0	-1	1
12	0	1	1
13	0	0	0
14	0	0	0
15	0	0	0

The Box-Behnken design is a efficiently mathematical and statistical method useful in modeling and problem analysis of a complex process. It creates the response equations as functions of various independent variables to determine the optimal responses and is a simple approach to manage and to describe the results when compared with other methods. In this research it is to find the appropriate process parameters in the precipitation hardening of 6061 aluminium alloy (the complicated process) consisting of at least three factors. Therefore, Box-Behnken design is selected as the experimental design different from the research in title “optimal parameters for diffusion bonding of semi-solid casting aluminium alloy” (14) ,which use the central composite design, CCD due to having just two factors affecting on the tensile strength. In addition, Box-Behnken design has less the number of runs causing decreasing time and expenditure, is popular used in industries and creates the optimal predictive mathematic model of precipitation hardening process of 6061 aluminium alloy.

3. Material and Experimentation

3.1 Material and equipments

1) The 6061 aluminium alloy specimen in the number of 45 rods of 25.4 mm in diameter and 25.4 mm length. The chemical composition and the hardness are received from this experiment as shown in Table 2. The cross section area of specimen is machined to have a smooth surface to prevent an error of hardness measurement.

Table 2. Chemical composition of 6061 aluminium alloy (wt.%).

Specimen	Si	Fe	Ti	Cu	Mg	Zn	Mn	Hardness (HV)
Al 6061	0.8	0.7	0.15	0.4	1.2	0.25	0.15	100

2) Heat treatment process is performed using the Linn High Therm induction furnace model: HK 30.27.

3) The measurement of specimen hardness carried out using Vicker hardness tester model : Zwick/ Roell ZHU.

3.2 Design of experiment

1) Selection of factors, levels and response variable

Choices of parameters and their values are in accordance with the standard (15), related researches, primary experiments and limitations of instruments used in the experiment. Three parameters are to be investigated: solution soaking time, aging temperature, aging time, which can be adjusted from a controller of the heat treatment furnace. When the response variable as hardness is selected, the appropriate parameters are then determined. The changing value of parameter levels in the experiment is defined to be three levels: low (-1) middle (0) and high (1) as shown in Table 3.

Table 3. Factor levels and factor limitations.

Factors	Level		
	-1	0	1
Solution soaking time (hr): (X_1)	4	5	6
Aging temperature (°C): (X_2)	127	177	227
Aging time (hr): (X_3)	6	8	10

2) Box-Behnken design

This research applies Box-Behnken design to find out the appropriate parameters and to create a model relating between the hardness and the parameters in precipitation process of 6061 aluminium alloy by defining the factor level in 3 levels: low, middle and high. The experimental model is created by Minitab R.16 program, which is a program for statistical analysis and widely used in industries. This model has 15 experiments by each repeated three times to estimate errors and to assess the results from the experiment. Therefore, it has a total of 45 experiments as shown in Table 4 by using the level values of each factor as shown in Table 3 representative the number -1, 0, +1 to create testing model. The experimental procedure is referred to a column of run order or an order of experiment.

Table 4. Box- Behnken design model received from Minitab program.

StdOrder	RunOrder	Solution Time	Aging Temp	Aging Time
13	1	0	0	0
27	2	0	1	1
7	3	-1	0	1
40	4	0	1	-1
19	5	1	1	0
…	…	…	…	…
…	…	…	…	…
39	44	0	-1	-1
3	45	-1	1	0

3) Precipitation hardening of 6061 aluminium alloy

The precipitation hardening process (T6 treatment) consists of three main steps: the solution heat treatment, the water quenching, and the artificial aging (Figure 1).

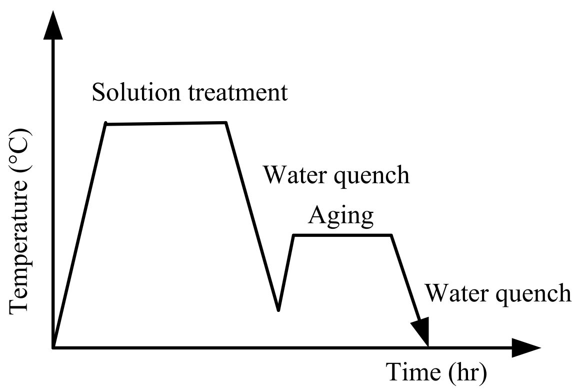

Figure 1. The stages of precipitation hardening process of 6061 aluminium alloy.

The temperature of solution treatment is recommend at 529°C (16). The aging temperature and the aging time of each experiment are identified by the condition of each run order as shown in Table 4; therefore, the number of experiments are 45 runs. For example, Run 1 is the solid solution soaking time of 5 hours, the aging temperature of 177°C, and the aging time for 8 hours and the standard order 13 has the following steps :

(1) The specimen is placed in an electric furnace, the solution treatment is set at 529°C for 5 hours, causing the aluminium alloy becoming a single solid solution phase.

(2) Then, the specimen is water quenched at room temperature.

(3) The specimen is reheated in the furnace by setting the aging temperature of 177°C with the aging time of 8 hours to form Mg_2Si precipitation within the base

structure or matrix.

(4) After aging, the specimen is immediately cooled in water to stop the precipitation process.

(5) The specimen hardness is immediately measured by using the Vicker hardness tester with 10 kg load for 10 seconds. Each specimen is measured 4 times by measuring 2 points on x-axis and y-axis, which are calculated as a mean hardness value (17).

(6) All experiment is performed repeatedly as shown in Table 4.

4. Results and Discussion

4.1 Checking of data and adequacy of model

The quality of data is first investigated before analyzed by verifying the model adequacy checked in 3 assumptions (18-20) ie., independent test, normality test and variance stability test.

1) Normality test is a test whether the residuals have the normal distribution (Figure 2). It is noticed that the points distribute along with the straight line with no any abnormality; therefore, the residuals have the normal distribution.

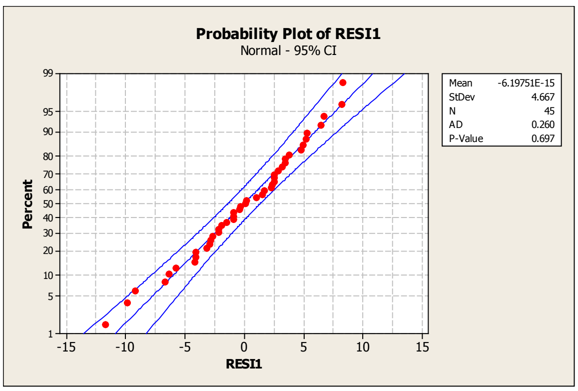

Figure 2. The normal probability plot of standard residuals.

2) Independent test is a test of the residual independence by observing whether distribution of points on the scatter plot has any patterns (Figure 3). The residuals of experimental result in precipitation hardening of 6061 aluminium alloy have no exact patterns and are evenly distributed. This indicates that the data is independent.

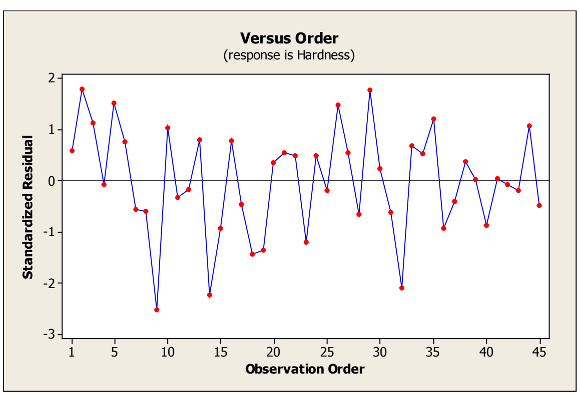

Figure 3. The scatter plot of standard residuals vs observation order.

3) Variance stability test is carried out by using the scatter plot of each factor level (Figure 4). It shows the residual points, the result from precipitation hardening, are evenly distributed on both the positive and negative sides; therefore, the data have the variance stability. From checking the model adequacy, the residuals follow the 3 assumptions: (1) the normal distribution, (2) the independence, and (3) the variance stability. It can be concluded that the hardness data from this experiment are valid and fit for analysis of the variance to determine the coefficient of determination (R^2).

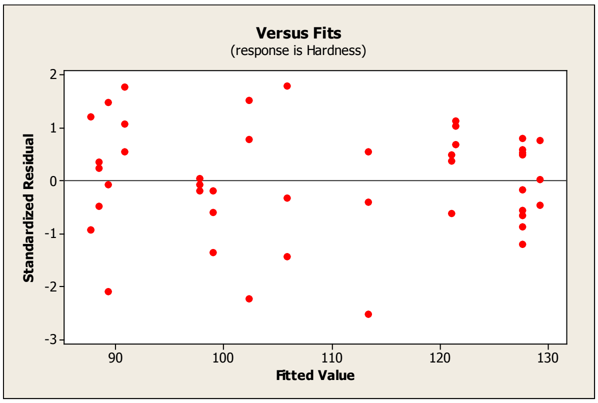

Figure 4. The scatter plot of standard residual vs fitted value.

4.2 Estimated regression coefficients of the hardness

The regression analysis in Table 5 indicates that factors affecting hardness of 6061 aluminium alloy in the precipitation hardening process are solution soaking time, aging temperature and aging time, the square term of solution soaking time, the square term of aging temperature and interaction between the solution soaking time and aging temperature, have the p-value less than the 0.05 significant level. The regression equation of hardness is shown in Eq. (1). From Table 5, it can be confirmed that the most influencing parameters for hardness are the solution soaking time, aging temperature and aging time. The significance of the developed model is investigated using the analysis of variance (ANOVA) technique. The determination coefficient (R^2) indicates the goodness of fit of the model. In this case, the determination coefficient ($R^2 = 0.919$) shows that only less than 10% of the total variance is not explained by the model. The value of the adjusted determination coefficient (adjusted $R^2 = 0.906$) is also high, which indicates the high accuracy of the model.

$$\begin{aligned}\text{Hardness} = \; & -392.078 + \text{solution soaking time} * 56.7514 + \text{aging temperature} * \\ & 3.90818 + \text{aging time} * 2.02917 - \text{solution soaking time} * \\ & \text{solution soaking time} * 6.36859 - \text{aging temperature} * \\ & \text{aging temperature} * 0.0117074 + \text{solution soaking time} * \\ & \text{aging temperature} * 0.0610000 \end{aligned} \tag{1}$$

Table 5. Estimated regression coefficients for hardness.

Term	Coef	SE Coef	T	P
Constant	127.649	1.393	91.648	0.000
Solution Time	3.862	1.025	3.768	0.001
Aging Temp	3.437	1.025	3.353	0.002
Aging Time	4.058	1.025	3.959	0.000
Solution Time*Solution Time	-6.369	1.504	-4.233	0.000
Aging Temp*Aging Temp	-29.269	1.504	-19.455	0.000
Solution Time*Aging Temp	3.050	1.450	2.104	0.042
S = 5.02186 R-Sq = 91.92% R-Sq(adj) = 90.64%				

4.3 Analysis of variance of hardness

Analysis of variance is the investigation of variance sources of the model. From analysis of variance of hardness forming from the precipitation hardening at 0.05 significant level (Table 6), it is found that p-value of an interaction effect term and a square term are 0.000 and 0.042 respectively, which are less than 0.05 specified significant level. It is shown that a curvilinear effect is present in the true response surface model (21-23). Therefore, this second-order model can be fit for predicting the hardness forming from the precipitation hardening. When considering lack of fit, it is found that the p-value is 0.170 more than 0.05; therefore, it could be concluded that this model is adequate for variables in the equation and enables be used to predict the hardness forming from the process.

Table 6. Analysis of variance of hardness.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	6	10899.1	10899.1	1816.51	72.03	0.000
Linear	3	1036.9	1036.9	345.64	13.71	0.000
Square	2	9750.5	9750.5	4875.25	193.32	0.000
Interaction	1	111.6	111.6	111.63	4.43	0.042
Residual Error	38	958.3	958.3	25.22		
Lack-of-Fit	6	224.7	224.7	37.46	1.63	0.170
Pure Error	32	733.6	733.6	22.92		
Total	44	11857.4				

4.4 Response surface of hardness

The surface plot (Figure 5) and the contour plot (Figure 6) can be obtained in equation 1. It is found from Figure 5 when the solution soaking time and the aging temperature are located at the middle levels, maximum hardness are reached. This is because the saturated solution phase of 6061 aluminium alloy contains the largest amount of precipitates. The contour plot in Figure 6 is the relationship between the solution soaking time and the aging temperature in precipitation hardening and has a non-linear characteristic. The most inner oval of the plot has the hardness of 120 HV and the other outer ovals have the decreasing hardness i.e.110, 100, and 90 HV respectively.

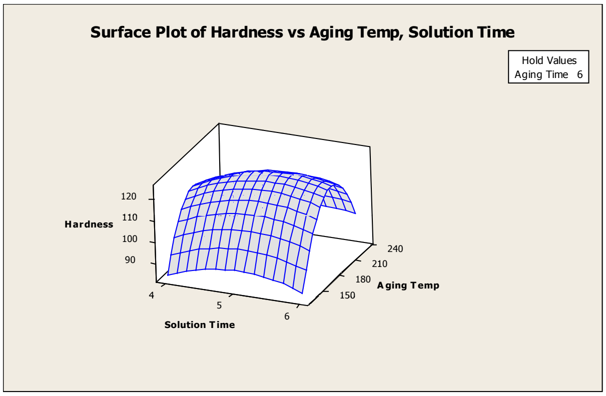

Figure 5. The response surface plot of hardness.

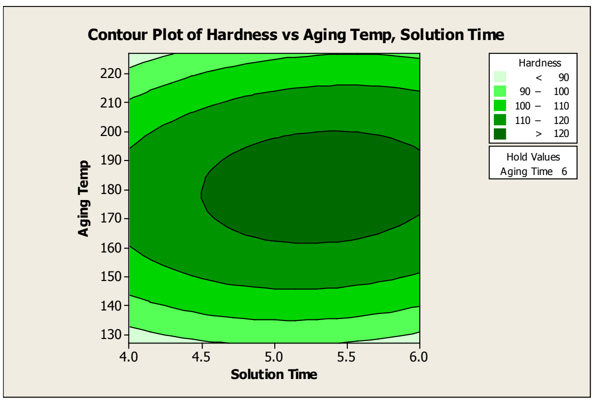

Figure 6. The contour plot of hardness.

4.5 Optimization

In order to receive the maximum hardness, the most fitting factors are determined by using a function of response optimizer in Minitab R.16 to measure the composite desirability (D) ranging from 0 to 1. If D=1, it means that the received response satisfies perfectly. From determining the most fit factors by using the function of response optimizer, it is found that the optimal condition of hardness from the precipitation hardening is the solution soaking time of 0.3131, aging temperature of 0.0707 and the aging time of 1.0 (Figure 7). When transformed to be the actual values, they are the solution soaking time of 5 hours and 18 minutes, the aging temperature of 181°C and the aging time of 10 hours yielding the hardness of 132.5 HV with the desirability (D) of 1.0.

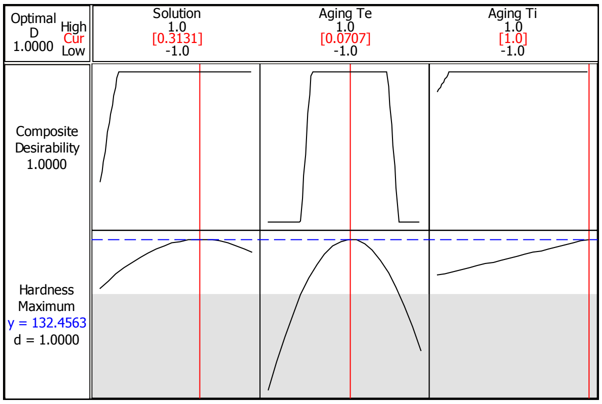

Figure 7. Optimization plot of precipitation hardening.

4.6 Confirmation test

Confirmation of the result can be performed by employing the regression equation and the experiment in order to find the appropriate parameters in the precipitation hardening process of 6061 aluminium alloy. From experimental data, it is found that the appropriate parameters are the solution soaking time of 5 hours and 18 minutes, aging temperature of 181°C, the aging time of 10 hours with 30 replications. Then, the result is compared with the actual experimental values equal to 130.5 mean hardness with 5.80 standard deviation. The confirmation test has the p-value of 0.08 greater than the 0.05 level of significance. It means that it is statistically insignificant. After the predictive equation is confirmed by comparing with the value measured from the actual experiment, it shows that the result has minor error. This equation enables to be used to predict the hardness of 6061 aluminium alloy through the precipitation hardening process.

5. Conclusion

According to the application of design of experiment in response surface methodology with Box-Behnken design in the precipitation hardening, it is seen that three factors: the solid solution soaking time, the aging temperature, the aging time, and the interaction of 2 factors have the influence on the hardness and the fit predictive model of hardness can be expressed as: Hardness = $-392.078 + 56.7514*X_1 + 3.90818*X_2 + 2.02917*X_3 - 6.36859*X_1^2 - 0.0117074*X_2^2 + 0.0610000*X_1X_2$. The coefficient of determination (R^2) is 0.92. When the function of response optimizer is taken to find out the best process condition, the optimization of precipitation hardening is reached when the solution soaking time is 5 hours and 18 minutes, the aging temperature is 181°C and the aging time is 10 hours yielding maximum Vicker hardness value of 132.5 HV with the desirability (d) of 1.0. After confirmation test, the mean hardness of the experiment is close to the hardness of predictive model at 95 % confident interval.

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