Modeling and Optimization of Precipitation Hardening Heat Treatment Factors of the Al2024 Alloy Using a Two-Level Full Factorial Design

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ABSTRACT

In the current study, the sources of variation in the mean hardness value of heat treated aluminum 2024 samples were identified by using metallurgical study and design of experiments methodology (full factorial method). Hardness measurements and microstructural investigations of the samples were carried out by using Brinell hardness test and optical microscopy, respectively. The effects of several control factors including solution treatment temperature, aging temperature, and aging time on the hardness were evaluated. The main and interactions effects of the factors were studied by means of analysis of variance (ANOVA) technique. Moreover, the best model which can estimate the hardness of the heat treated Al2024specimens was found whereas the aging temperature and interaction of aging time and aging temperature had significant effects on the hardness value. Finally, the results of the statistical analysis were used to find the optimum conditions of the factors in order to get the maximum hardness of the samples. After solution treatment (530 °C) and 12 h of aging (180 °C), the optimum hardness of the heat treated Al2024 alloy samples was about 133 Brinell, which is in good agreement with the value estimated by the model (~131 Brinell).

1. Introduction

Aluminum and its alloys are among the most commonly used metals due to their special features. Outstanding properties that make Al alloys one of the economical and interesting candidates in various applications are light weight, corrosion resistance, good formability, excellent fatigue resistance, recyclability and high strength to weight ratio [1]. Among Al alloys, due to its superior mechanical properties, good weldability, as well as heat treatability, Al2024 is one of the most common used alloys in numerous engineering applications such as aircraft structures, corrosion resistance coatings and electrically conductive systems [2].

In most cases, mechanical properties of this alloy must be improved from the as-prepared sheets or rods. The strength and hardness of the Al2024 alloys could be enhanced by the formation of extremely small uniformly dispersed Al-Cu precipitates as a second phase within the Al matrix [3]. This must be accomplished by an appropriate heat treating

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process [4]. This process involves three distinct steps: solution treatment to minimize segregation in the alloy, quenching to create a supersaturated solid solution and aging to facilitate the formation of coherent precipitates which strengthen the Al alloy by interfering with dislocation movement [5]. Microstructural evolution during the heat treatment influences hardness, strength and ductility of the alloy. Different factors are important to reach sophisticated mechanical properties in this processing [6]. For example, at a constant aging temperature, increasing aging time over a distinct value causes dropping the properties of alloy down (over aging) [7]. Therefore, factors such as solution treatment temperature, aging temperature and time are all the main factors affect the hardness of the samples during the heat treatment.

On the other hand, doing all the experiments to investigate the influences of the all involved parameters is not cost-effective. The technique of defining and investigating all possible conditions in an experiment including multiple factors is known as the design of experiments (DOE) [8]. The purpose of DOE is to perform the minimum number of experiments to get the most information on involving factors [9-11]. By optimization of the variation range of the main factors, the distinguished factors which have substantial influence on the output results are found. There are several DOE methodologies among which factorial design iswidely used in experiments involving several factors in two levels where it is necessary to precisely study the overall main factor effects and interactions of different factors[12-14]. This method is based on statistical design of the experiments and has become a practical tool for improving the quality of outputs by establishing optimum process settings or design parameters [15]. In a full factorial design, the effect of all possible combinations of levels across all factors is studiedand hence allows the investigator to study the effect of each factor on the response variable, as well as the effects of interactions between factors on the response variable.

In the present study, the full factorial design is employed to study the effects of heat treatment parameters including solution treatment temperature, aging temperature and aging time on the hardness of the Al2024 alloy. Finally, the optimal values for attaining a higher hardness using heat treatment were searched and a model was proposed to estimate the hardness during heat treatment.

2. Experimental Procedure

Homogenized Al2024 specimens were used with dimensions of 2×2×1cm. Table 1 shows the chemical composition of the alloy. A tubular furnace was used for the heat treatment of the Al2024 samples. The process consists of solution treatment for 60 min and quenching in mixed ice water media. To prevent natural ageing of the alloy, the specimens were immediatelyplaced in the aging furnace. The Brinell test was performed by InstronWolpert testing machine with five replications to determine the mean hardness value of the as-received and heat treated Al2024 specimens. This test consists of applying a constant load of 62.5 Kgf, for 10 sec using a 2.5 mm diameter tungsten carbide ball. Before hardness measurements, the surface of the specimens was mechanically polished. Also, metallographic samples were prepared by polishing and etching with chlorine etchant (2ml HF 48%, 3 ml HCl, 5 ml HNO_3 and 190 ml distilled water) and then analyzed by optical microscope (Olympus-PME3).

A 2^3 full factorial design with three center point observations was used to investigate the effect of the factors in the heat treatment process. Adding the center points provides some information to test the curvature and then evaluates the presence of noises in the system. The sequence of the experiments was randomized to reduce the risk of introducing experimental bias. Optimum condition was obtained by finding an appropriate prediction model and analyzing the surface and contour plots. Final result of the optimum condition was considered for checking the reliability of the selected model. Design of the experiments and statistical analysis of the obtained results were carried out by using Design Expert 7 software which is one of the most famous softwares in DOE.

The main controllable factors affecting the

Table 1. The chemical composition of the as-received Al2024 alloy

Element	Si	Mn	Cu	Mg	Fe	Cr	Ni	Zn	V	Ti	Al
Weight percent	0.13	0.30	3.80	1.35	0.35	0.05	0.01	0.30	0.008	0.004	balanced

Table 2. Experiment factors and the associated levels

Factors	Symbol	Range and levels		
		Low	Center	High
Solution treatment temperature (°C)	A	490	510	530
Aging temperature (°C)	B	180	190	200
Aging time (hr)	C	8	10	12

Table 3. Experimental Design for the heat treatment process of the Al2024 alloy

Experimental Number	Run Order	Solution treatment temperature (°C)	Aging temperature (°C)	Aging time (hr)	Measured hardness (Brinell)	Predicted hardness (Brinell)
1	4	530	200	8	125	125.75
2	6	530	180	12	133	131.25
3	11	510	190	10	116	116.67
4	8	530	200	12	107	107.25
5	5	490	180	12	127	128.75
6	3	490	200	8	124	132.25
7	2	530	180	8	117	117.75
8	10	510	190	10	116	116.67
9	9	510	190	10	118	116.67
10	1	490	180	8	119	115.25
11	7	490	200	12	105	128.75

hardness during heat treatment of the Al alloy are solution treatment temperature, aging temperature and aging time varied between 490-530 °C, 180-200 °C and 8-12 h, respectively. The experiment factors along with the associated levels are presented in Table 2.

3. Results and Discussion

3. 1. Statistical Analysis

The experimental design which is performed by Design Expert 7 software is presented in Table 3. Also, the measured hardness values of the heat treated samples at different experiment conditions arepresented in this table.

The significance of the main effects, the contribution percent and then optimum conditions of the different parameters on the hardness resulted from the statistical analysis of variance have been given in Table 4. Based on the analysis of variance (ANOVA), the contribution of each parameter in affecting the quality characteristic is evaluated. The ANOVA is a collection of statistical models used in order to analyze the differences between group means. In the ANOVA, the observed variance in a particular variable is partitioned into components attributable to different sources of variation. Fisher's t-test was used to recognize an appropriate model for prediction of the hardness. Generally, a model with an appropriate 'F' value and low 'P' value is an acceptable model [16]. If significance level of an effect was greater than 95%, the effect was considered to be significant. This effect meant that the model with a P-value (the probability of obtaining a test statistic at least

Table 4. Analysis of variance (ANOVA) table

Variation source	Sum of squares	Degree of freedom	Mean square	F-value	P-value Prob> F	Contribution percent	
Model	645.50	4	161.37	42.37	0.0005	94.42	significant
A-Solutionizing Temperature	6.13	1	6.13	1.61	0.2606	0.90	
B-Aging Temperature	153.12	1	153.12	40.21	0.0014	22.40	
C-Aging Time	21.13	1	21.13	5.55	0.0651	3.09	
BC	465.13	1	465.13	122.13	0.0001	68.04	
Curvature	19.09	1	19.09	5.01	0.0735	2.79	not significant
Residual (error)	19.04	5	3.81			2.78	
Lack of Fit	16.37	3	5.46	4.09	0.2025		not significant
Pure Error	2.67	2	1.33				
Cor Total	683.64	10					

as extreme as the one that was actually observed) lower than 0.05 could be considerable. It is concluded from the ANOVA results that the model is statistically significant. It can be seen that the F value for quadratic term of the model is suchthat it is not significant. Therefore, the linear model is selected as an appropriate prediction model. Each of the factors has one degree of freedom and total four degrees of freedom for the model is created. In this table, the pure error has two degrees of freedom and 1 degree of freedom is remained for the curvature. By assigning these three degrees of freedom the model has more power than no center point.

Based on the ANOVA results, the aging temperature and its interaction with the aging time can have significant effects (P-value < 0.05). Eq. 1 is a mathematical model obtained by regression analysis which relates the hardness (H) of the heat treated Al2024 alloy to the solutionizing temperature (A), aging temperature (B), aging time (C and interaction of aging time and temperature (BC).

$$H(Brinell)=- 535.81250+0.043750*A+ 3.37500*B+71.62500*C-0.38125*B*C \quad [1]$$

Where temperature and time units in the model are Celsius and hour, respectively. This equation can be used to estimate the hardness of the Al2024 alloy during the heat treatment process. The coefficient value of variation (C.V.) for Eq. 1 was 1.64%, which indicated both the precision and reliability of the experiments. The model also presents a high determination of coefficient R^2=0.9713 (explaining 97.13% of the variability in the response). The observed and the predicted values obtained from Eq.1 areshown in Fig. 1. According to the analysis of variance, the adjustment R-square (0.9484; adjustment R-squared that penalizes the addition of extraneous predictors to the model), the model is appropriate and meaningful.

Adequacy of the selected model is important and it can be identified by ANOVA. By analyzing the residuals from this experiment, adequacy of the assumptions of analysis of variance are investigated (Fig.2). The normal probability plot shown in Fig.2a indicates that the residuals follow a normal distribution and the points follow a straight line. Also, Fig. 2 b shows residuals versus the predicted plot. This is a plot of the residuals versus the ascending predicted response values and tests the assumption of constant variance. The plot is a random scatter (constant range of residuals across the graph) and represents that the variance of data is constant.

The effect of each factor in the model is shown in Fig. 3. Comparison of parts a-d of this figure shows that the effect of the interaction of aging temperature and time is very high. From a metallurgical point of view, it should be said

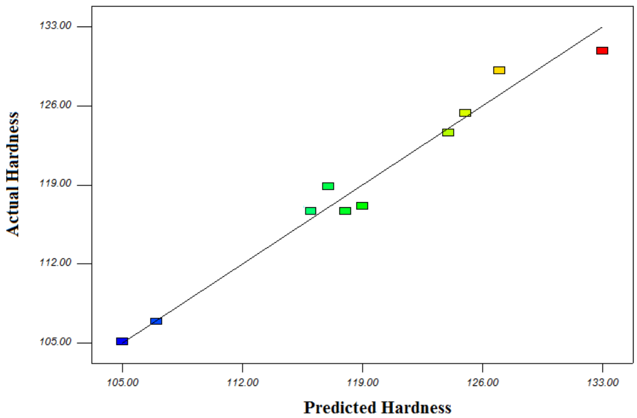

Fig. 1. Real hardness valuesvs predicted values by the model

(a)

(b)

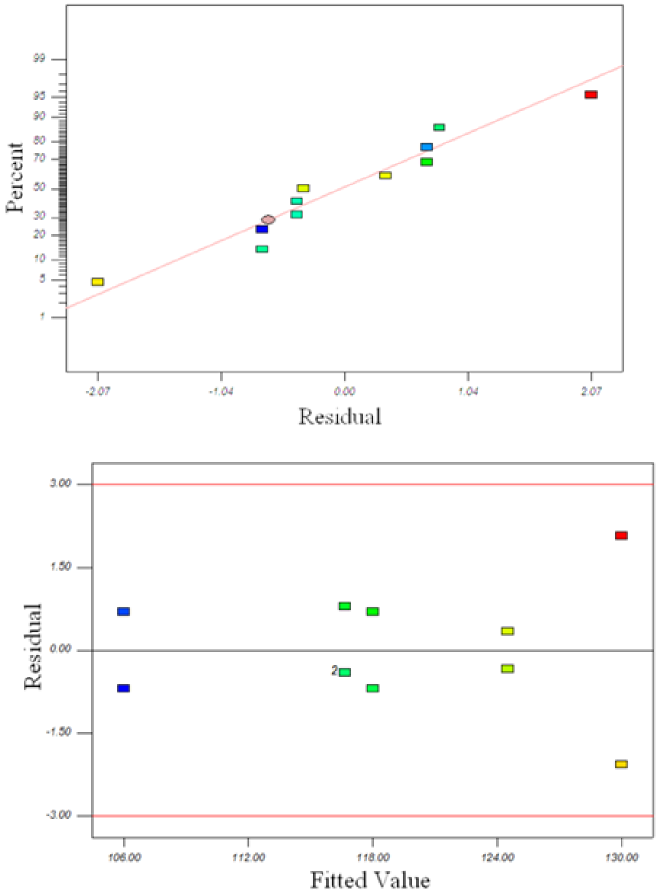

Fig. 2. Residual plots (a) normal probability plot of the residuals and (b) residuals versus predicted plot

that these two factors are highly associated together and virtually isolated investigation of each one is not correct. Also, Fig. 3 shows that the solution treatment temperature has the least influence on the hardness value. Therefore, the high and low levels of the solution treatment temperature do not significantly change the hardness.

The 3D response surface plots are graphical representation of the regression equation generally used to visualize the relationship between the response and experimental levels of each variable and the type of interactions between the variables to deduce the optimum

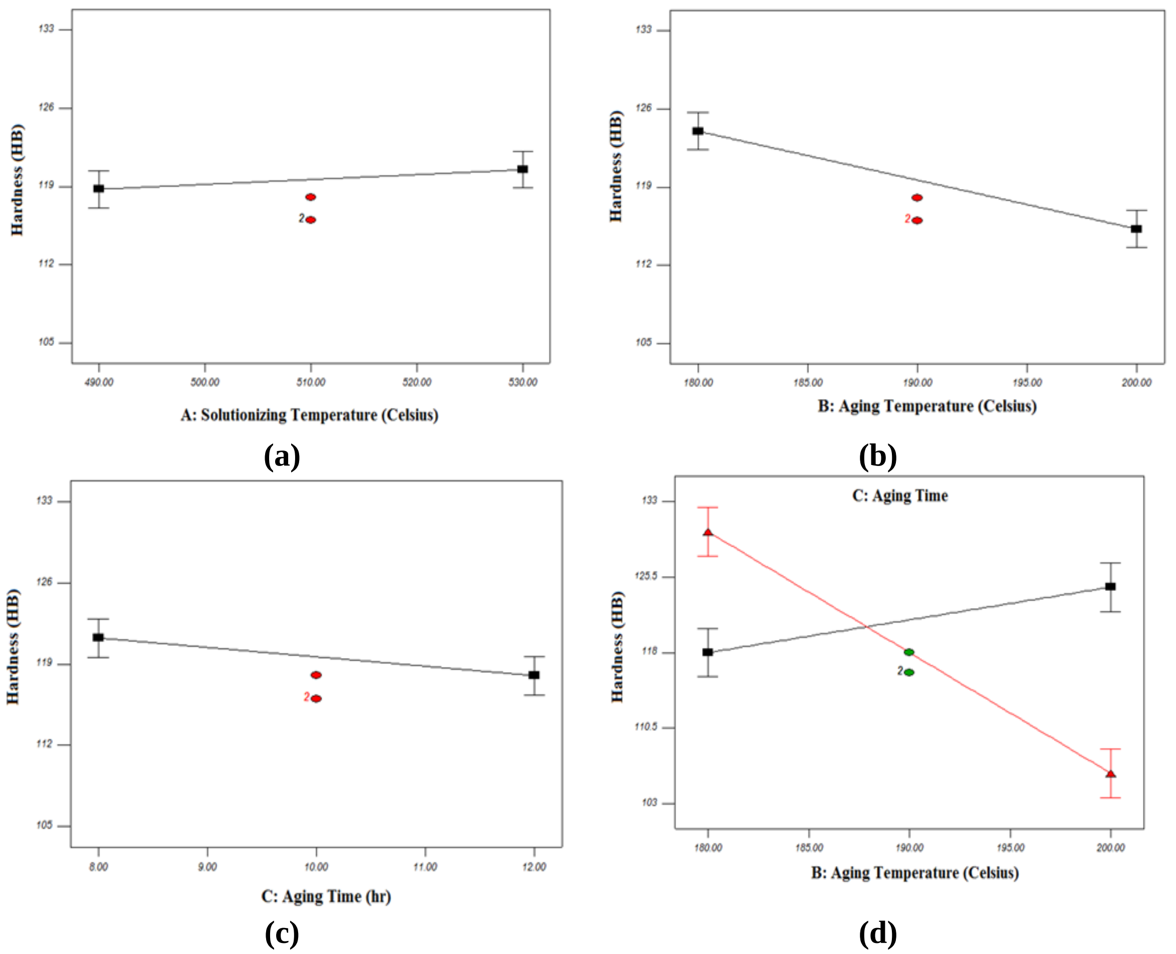

Fig. 3. Main effects plot for hardness versus (a) solution treatment temperature, (b) aging temperature, (c) aging time and (d) aging time and temperature interaction plot

conditions [16]. Fig. 4 shows the 3D response surface relationship between two factors among solution treatment temperature, aging time and aging temperature at the center level of other factor with the hardness of heat treated Al2024. It is obvious from Fig. 4a that the highest hardness could be achieved with the minimum levels of solution treatment temperature and aging time at the center level of aging temperature. As the result indicates in Fig. 4b, the hardness increased with an increase of the solution treatment temperature and decrease of the aging temperature. It can be concluded from Figs.4a and b that the solution treatment temperature is not a determining factor for hardness in this investigation. Fig.4c illustrates the interaction effects of the aging time and temperature on hardness. The maximum hardness of the specimen is obtained in the aging temperature of 180 °C and the aging time of 12 h, which is colored by red in this figure.

The curved lines in Fig. 5 show the response for the interaction of time and aging temperature. In this figure, the solution treatment temperature is constant and equals 510 °C.

The optimum calculated factors experiment were solution treatment temperature 530°C, aging temperature of 180°C and the aging time of 12 h which was introduced by Design Expert 7. The proposed optimum conditions were examined for 2 replicates which result in hardness 130 and 133 Brinellboth of which are acceptable.

3. 2. Metallurgical observations

Fig. 6a shows the microstructure of the as-received Al2024 specimen. As can be seen, the coarse precipitates of the Al_2CuMg phase are observed in the Al matrix. This phase is formed

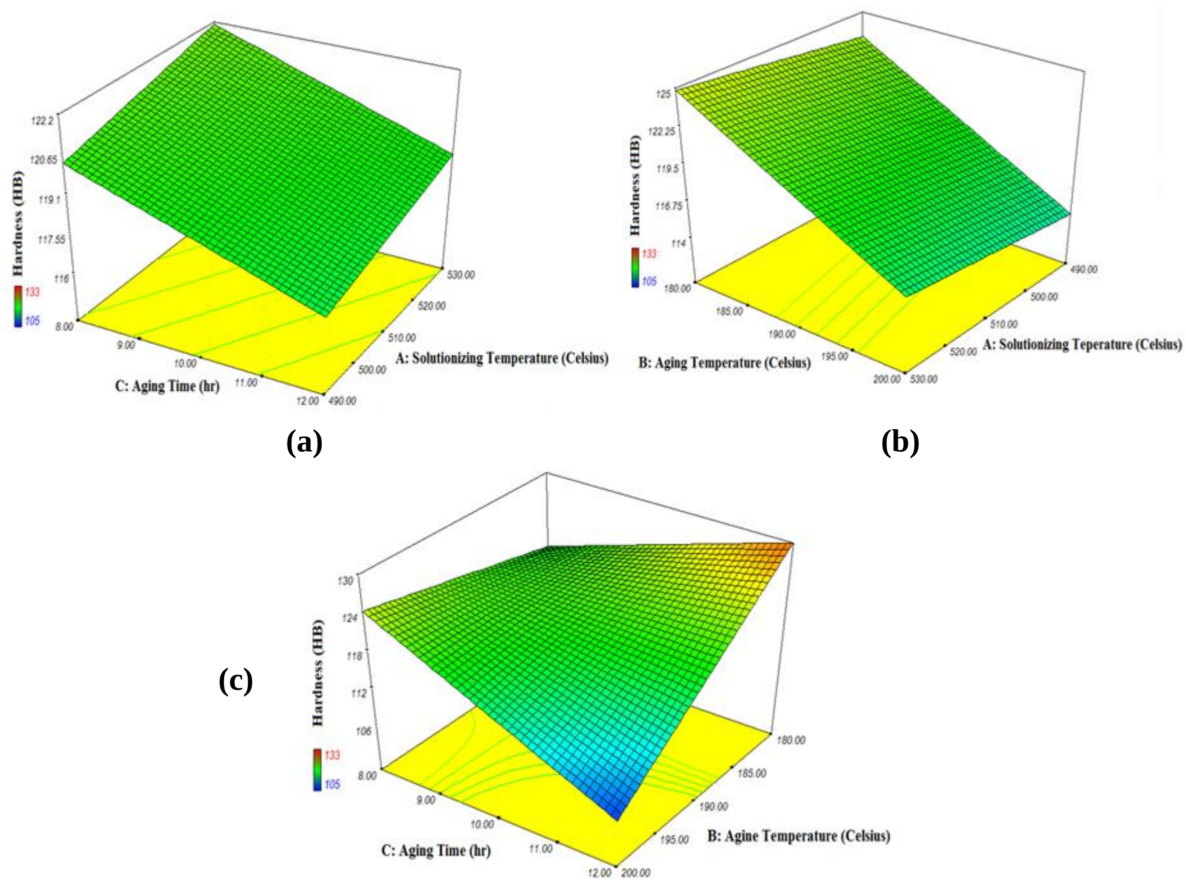

Fig. 4. Three dimensional response surface plots for the hardness of the heat treated Al2024 alloy, (a) effects of the solution treatment temperature and aging time at the center level of the aging temperature, (b) effects of the solution treatment temperature and the aging temperature at the center level of the aging time, and (c) effects of the aging temperature and the aging time at the center level of the solution treatment temperature

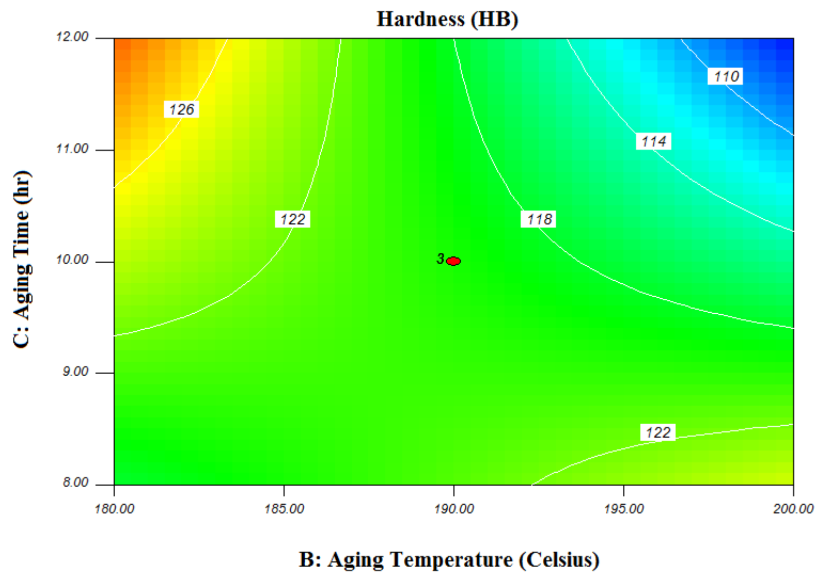

Fig. 5. Contour plot showing the interaction effect of time and temperature of aging on the hardness of the heat treated Al2024 alloy

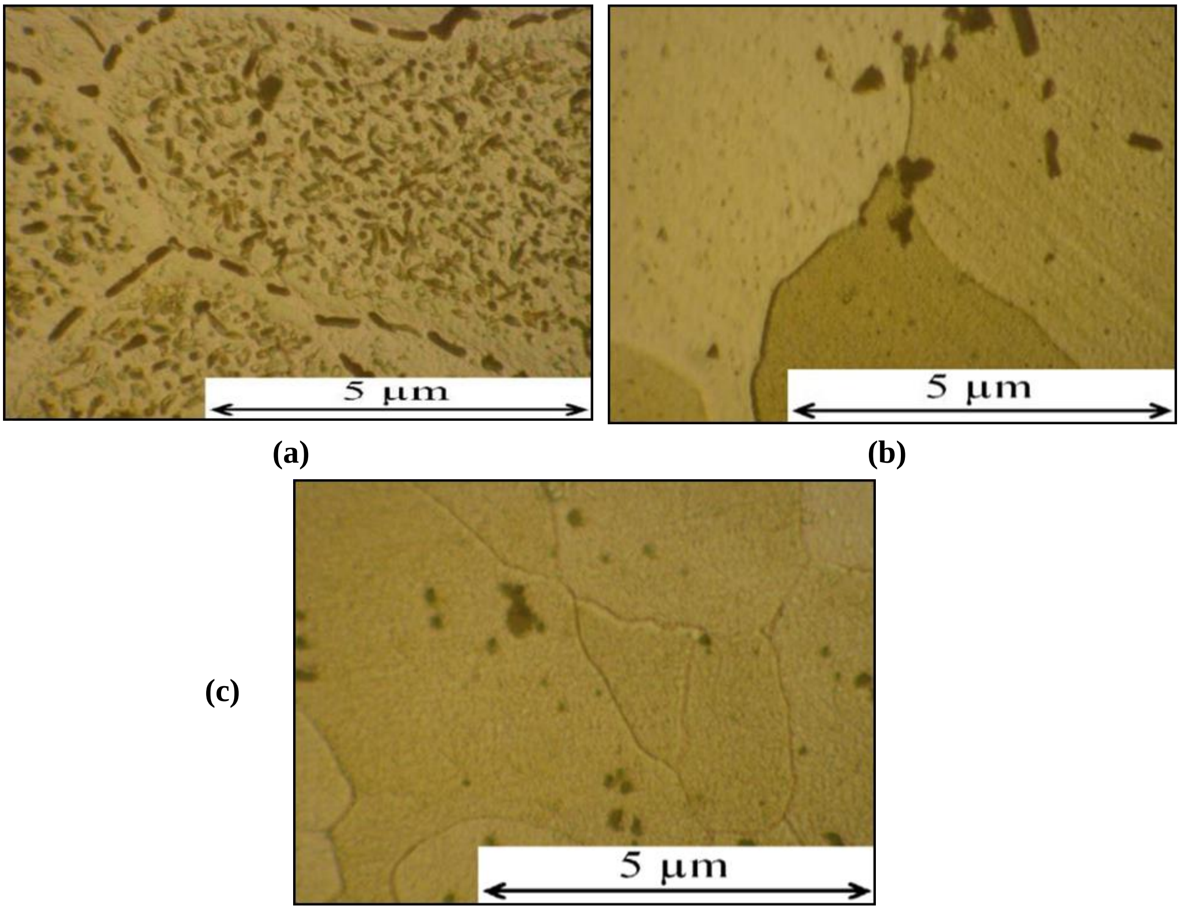

Fig.6. Optical micrographs of : (a) the as-received Al2024 alloy, (b) the heat treated specimen including solution treatment at 530°C for 60min and (c)) the solution treated specimen and aging in 180°C for 12h

due to overaging of the specimen. In addition, the Al_2CuMg phase exists in the grain boundaries of the alloy. This problem results in low hardness value of 70 Brinell. Fig. 6b shows microstructure of the solution treated specimen at 530 °C. Comparing Fig. 6a and b clearly illustrates that precipitates are dissolved.According to previous researches, solutionizing for 1 h is required to completely solve allthe precipitates and limit thekinetics of grain growth. Also, Fig.6b shows that the time of solution treatment is appropriate to solve almost all of the precipitates even those present in the grain boundaries. The hardness of the specimen is measured about 62 Brinell.

Fig.6c shows the microstructure of the solution treated sample which is aged at 180°C for 12 h. It can be seen that the precipitates are not formed in the microstructure. By measuring the hardness of the specimen, it is concluded that the fine participates which are not observed by optical microscope causethe mean hardness value of the specimen to reach about 131.5 Brinell.

4. Conclusions

A two-level full factorial design was applied to modeling and process optimization for heat treating of Al2024 alloy. Using the method, eleven experiments were needed to determine the percent contribution of each process parameter, including solution treatment temperature, aging temperature and aging time on the hardness value and to find the optimum condition of the process. It was found that hardness increases with a decrease in the aging time and the aging temperature and that the solution treatment temperature does not significantly affect the hardness. The contribution percent of the aging temperature and the interaction of the aging temperature and time to the enhancement of the hardness were found to be 22.40 and 68.04%, respectively. Optimizing all factors is performed and reported in this study and the results are solution treatment temperature of 530°C, aging temperature 180°C and aging time of 12h. The obtained result of hardness testing in the optimum condition showed a good agreement with the predicted value by the model.

References

1. J. L. Brandt, “Properties of pure aluminum”, ASM international, 1984.
2. J. R. Davis. “Aluminum and aluminum alloys”, ASM Specialty Handbook, 1957.
3. S. P. Ringer, K. Hono, I. J. Polmear, T. Sakurai, “Precipitation processes during the early stages of ageing in Al-Cu-Mg Alloys”, Appl. Surf. Sci. Vol. 94, 1996, pp. 253–260.
4. Y. Gefen, M. Rosen, A. Rosen, “Aging phenomena in Duraluminum 2024 studied by resistometry and hardness”, Mater. Sci. Eng. Vol. 8, 1971, pp. 181-188.
5. A. Charai, T. Walther, C. Alfonso, A. M. Zahra, C. Y. Zahra, “Coexistence of clusters, GPB zones, S″-, S′- and S-phases in an Al–0.9% Cu–1.4% Mg alloy”, Acta Mater. Vol. 48, 2000, pp. 2751–2764.
6. S. P. Ringer, T. Sakurai, I. J. Polmear, “Origins of hardening in aged Al-Gu-Mg-(Ag) alloys”, Acta Mater. Vol. 45, 1997, pp. 3731–3744.
7. M. N. Cavalli, V. Mandava, “Effect of temperature and aging time on 2024 aluminum behavior”, Proc. the 6th International Congress and Exposition, Orlando, Florida-USA, 2008, pp. 674-687.
8. T. Lundstedt, E. Seifert, L. Abramo, B. Thelin, A. Nystrom, J. Pettersen, R. Bergman, “Experimental design and optimization”,Chemom.Intell. Lab. Syst. Vol. 42, 1998, pp. 3–40.
9. A. C. Tamhane, “Statistical analysis of designed experiments: theory and applications”, 1th ed, Wiley series in probability and statistics, 2009.
10. S. A. Sayyad, B. P. Panda, S. Javad, M. Ali, “Optimization of nutrient parameters for lovastain production by Monascus MTCC 369 under submerged fermentation using response surface methodology”, Appl. Microbiol. Biotechnol. Vol. 73, 2006, pp. 1054–1058.
11. R. Gottipati, S. Mishra, “Process optimization of adsorption of Cr (VI) on activated carbons prepared from plant precursors by a two-level full factorial design”, Chem. Eng. J. 160, 2010, pp. 99–107.
12. T. Golshani, E. Jorjani, S. ChehrehChelgani, S. Z. Shafaei, Y. HeidariNafechi. “Modeling and process optimization for microbial desulfurization of coal by using a two-level full factorial design”. Int. J. Mining Sci. Technol. Vol. 23, 2013, pp. 261-265.
13. H. Jafaria, M. H. Drisa, A. R. Shayganpour, “Evaluation of significant manufacturing parameters in lost foam casting of thin-wall Al–Si–Cu alloy using full factorial design of experiment”, T. Nonferr. Metal Soc. Vol. 23, 2013, pp. 2843-2851.
14. R. Fontanaa, G. Pistone, M. P. Rogantin, “Classification of two-level factorial fractions”, J. Stat. Plan. Infer. Vol. 87, 2000, pp. 149-172.
15. D. C. Mongomery, “Design and analysis of experiments”, 5thed, John Wiley and Sons. INC, 1997.