Istraživanja i projektovanja za privredu

ISSN 1451-4117 DOI:10.5937/jaes0-38109 www.engineeringscience.rs



Journal of Applied Engineering Science Vol. 20, No. 4, 2022 Original Scientific Paper Paper number: 20(2022)4, 1026, 1214-1225

OPTIMIZATION AND PREDICTION OF THE HARDNESS BEHAVIOUR OF LM4 + SI3N4 COMPOSITES USING RSM AND ANN - A COMPARATIVE STUDY

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In the present work, LM4 + Si₃N₄ (1, 2, and 3 wt.%) composites were fabricated using the two-stage stir casting method. Precipitation hardening treatment was carried out on the cast composites and hardness results were compared with as-cast specimens. Microstructural analysis was performed using Scanning Electron Microscope (SEM) images to validate the existence and homogenous distribution of reinforcement in the matrix. LM4 + 3 wt.% Si₃N₄ composite with multistage solution heat treatment (MSHT) and aging at 100°C showed higher hardness viz., 124% improvement when compared to as-cast LM4 due to the uniform distribution of Si₃N₄ and precipitation of metastable phases during the heat treatment process. The microhardness values of the fabricated composites was investigated using Artificial Neural Network (ANN) and Response Surface Methodology (RSM). Both RSM and ANN models predicted hardness values close to experimental values with minimum error, and the prominence of aging temperature in the improvement of hardness values within the constraints of the factors under consideration. Based on the error values it can be concluded that the ANN model can deliver results with higher accuracy than the RSM model.

Keywords: Artificial Neural Network (ANN), Response Surface Methodology (RSM), hardness, precipitation hardening treatment, Multistage Solution Heat Treatment (MSHT)

1 INTRODUCTION

1.1 Basic introduction

Aluminum alloys and their composites are widely used in various sectors of large and small-scale industries [1]. They possess a good strength-to-weight ratio and can be used at elevated working temperatures [2], but it is not used for many structural applications in automotive industries because of their lower hardness [3]. This study focuses on cast aluminum alloys, particularly hypoeutectic Al-Si alloy (LM4) which has high demand in the automotive sector. LM4 has good strength and can be used for high-temperature applications [4]. The mechanical properties of these aluminum allovs can be enhanced by the addition of reinforcements. Several research works related to LM4 composites were studied over a period with (TiB₂, WC, and AIN) as reinforcements [5] to [7]. Research work related to silicon nitride (Si₃N₄) as reinforcement is very rare and they have a great potential for improving the mechanical properties of aluminum alloys. Aluminum composites are generally prepared using stir casting and powder metallurgy techniques. Fabrication techniques play an important role in attaining uniform distribution of reinforcement and reducing fabrication defects. Another mode of improving the mechanical properties of aluminum and its composites is to perform precipitation hardening treatment [8]. Particularly for LM4 (cast alloys) solution heat treatment (SHT) followed by artificial aging is done to improve the mechanical properties [9]. A great deal of experimentation is required to acquire optimum process parameters that may produce the best mechanical properties, which require a lot of energy, money, and people. Many parameters must be considered throughout the precipitation hardening process. Another challenge with precipitation hardening modeling is the nonlinear connection between the parameters. To overcome the obstacles and save time, sophisticated optimization tools are required, which may deliver superior optimization outcomes within the study boundary conditions. Both RSM and ANN techniques were used in this work for modeling, optimization, and prediction. The following section provides a brief overview of RSM and ANN.

1.2 Response Surface Methodology (RSM) and Artificial Neural Network (ANN)

RSM is a fully advanced and sophisticated design of experiments (DOE) technique that provides statistical analysis and formulation for developing a model which provides optimized results with the selected input parameters. RSM can be a strong tool for predicting the end properties of aged samples without depending on time-consuming and costly experimentation [10], [11]. This is owing to the RSM's capacity to generate mathematical models as well. As the model is generated it establishes a relation between the input and output parameters and assesses them, this is done with the help of some statistical approaches [12]. Kamran *et al.*, [11] used RSM to optimize aging parameters of AA6056 alloy and from the results, they concluded that RSM can be effectively utilised to optimize the aging

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process, evaluate the relevance of aging parameters, and estimate the combined effect of process factors on the aging behaviour of AA6056. Many researchers used RSM as a statistical tool in their work and concluded that it is an effective tool for optimization and prediction [13] to [15].

To manufacture a certain product in today's world, it is critical to have a thorough understanding of the mechanical properties of materials (under working conditions) that will be utilised in the fabrication process, which must be chosen from the lot and should match the requirements criteria such as hardness and strength; if not, they should be treated to get the desired results. It costs money and energy to test and choose materials. We can predict the properties of materials using ANN as an alternative method by providing precise inputs to the model. When correctly trained, the use of ANN is an efficient, less time-consuming, less expensive, and more reliable method [16]. ANN is favored by many researchers because it can resolve nonlinear relationships between input and output variables extremely effectively [17] to [20]. Hosein *et al.*, [21] explained the operation of ANN in a detailed manner. Bilal *et al.*, [14] used both RSM and ANN methods for optimization of process parameters, from results they concluded that both RSM and ANN could estimate the responses but only RSM could effectively determine the parameters.

The present work focuses on the effect of reinforcement wt.% and aging temperature on the hardness variation of LM4 + Si_3N_4 composites. RSM model was created by considering important parameters in both the casting and precipitation hardening process using MINITAB, which helped in evaluating interactions between input and output variables and optimizing them to provide the best peak hardness values as output. ANN model was developed using MATLAB. Comparison was done between the predictions of ANN and RSM to identify the influence of selected factors on the peak hardness of the composite.

2 METHODOLOGY

Figure 1 depicts the overall methodology used in this investigation. Composites were prepared using a two-stage stir casting process and then subjected to precipitation hardening treatment to obtain peak hardness values. ANN and RSM were used to predict the hardness values of the composites and these results were compared with experimental hardness values.



Fig. 1. Methodology followed for the present investigation





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2.1 Materials and composite preparation

LM4 was used as a base material which is having 5.92 Si, 2.47 Cu, and 0.176 Mg as major alloying elements in wt.%. As reinforcement powder, Si₃N₄ with an average particle size of 26 μ m was used and the SEM and Energy Dispersive X-ray Analysis (EDAX) of the Si₃N₄ powder are shown in figure 2. Figure 2 EDAX validates the existence of Si and N elements, as well as the absence of contaminants and SEM confirms the uniform particle size of Si₃N₄ particles. Since the particle size is homogeneous and there are no contaminants, casting can be performed without any pre-treatments.





Fig. 2. SEM and EDAX of Si₃N₄ reinforcement powder

Coming to composite preparation, the two-stage stir casting method was selected. In this method, LM4 was melted at 780°C in a crucible using an electric furnace, a mechanical stirrer was used to create a vortex of the molten melt, then the melt temperature was brought down to 600°C (semi-solid state), and then preheated reinforcement (350° C for 30 min) was added to the melt for better bonding [22]. Stirring was continued for another 10 min and the composite mixture was poured into the preheated molds. As-cast samples were cut into small specimens accordingly and subjected to fine mirror finish polishing for SEM analysis to confirm the presence and uniform distribution of Si₃N₄ within the matrix. Micro Vickers hardness test was performed on the as-cast LM4 and it is composites.

2.2 Heat treatment procedure

As shown in figure 3, as-cast bar mold samples were machined into small cube specimens for precipitation hardening treatment and hardness test. Specimens of LM4 and its composites were divided into four sets and different precipitation hardening treatment was performed for each set. Set 1 and 2 specimens were subjected to MSHT viz., heating at 495°C/2 h in furnace 1 and 520°C/4 h in furnace 2, followed by warm water quenching (at 60°C). Then set 1 specimens were then subjected to aging at 100°C and set 2 specimens were then subjected to aging at 200°C. Similarly set 3 and 4 specimens were subjected to single-stage solution heat treatment (SSHT) viz., heating at 520°C/2 h in furnace 2, followed by warm water quenching (at 60°C). Then set 3 specimens were then subjected to aging at 100°C and set 4 specimens were then subjected to aging at 200°C. Artificial aging was conducted until peak hardness was attained for each set of LM4 and its composites. After precipitation hardening treatment, specimens are subjected to polishing before hardness testing.





2.3 Hardness testing

Hardness of the heat-treated and as-cast specimens of composites at different aging times was measured using Micro Vickers Hardness Tester, MODEL-MMT X 7A with a load of 200 gmf and dwell time of 15 seconds. In hardness testing, the possible experimental error was below 1%. The tests were carried out at room temperature (21-24°C), five measurements were collected for each sample at various positions, and the mean value was used.

2.4 RSM and ANN model

The central composite design (CCD) approach was used in RSM modeling and the input parameters and levels used were shown in table 1. It contains two factors wt.% and aging temperature with (1, 2, and 3 wt.%) and (0, 100, and 200°C) as levels. Using these input values, matrix of design was tabulated (as shown in table 2) using MINITAB, for which peak hardness values of MSHT specimens were selected as output variables because as per our previous studies [9] MSHT + artificially aged specimens gave better hardness results when compared to as-cast and SSHT + artificially aged specimens.

Factor	Levels	Values
wt.% of Si ₃ N ₄	3	1, 2, and 3
Aging temperature in °C	3	0, 100, and 200

Table 1. Factors used for RSM

StdOrder	RunOrder	PtType	Blocks	wt.%	Aging temperature in °C			
6	1	-1	1	3	100			
9	2	0	1	2	100			
4	3	1	1	3	200			
1	4	1	1	1	0			
11	5	0	1	2	100			
13	6	0	1	2	100			
10	7	0	1	2	100			
12	8	0	1	2	100			
5	9	-1	1	1	100			
3	10	1	1	1	200			
8	11	-1	1	2	200			
7	12	-1	1	2	0			

Table 2. Matrix of design

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StdOrder	RunOrder	PtType	Blocks	wt.%	Aging temperature in °C
2	13	1	1	3	0

In ANN same input and output parameters that are used for RSM were chosen for creating and training the model. 2 input variables, 1 output variable, 1 hidden layer, and 10 hidden neurons were used. The network diagram is shown in figure 4 and the Levenberg-Marquardt training algorithm with 15% testing, 15% validation, and 70% training target values was used as operational parameters.



Fig.4. Network used for ANN model

3 RESULTS AND STATISTICAL ANALYSIS

3.1 SEM analysis

Figure 5 shows the SEM and EDAX of LM4 + 3 wt.% Si_3N_4 cast composite specimen. From this image, we can see that the distribution of reinforcement is uniform throughout the sample and zero porosities were observed. The presence of Si_3N_4 in the matrix is confirmed by the EDAX analysis. Two-stage stir casting method turned out to be the finest method for the preparation of aluminum-based composites as we are adding preheated reinforcements to the semi-solid melt which resulted in better bonding between Si_3N_4 particles and matrix, also this may be due to good wettability between them, definitely preheating the reinforcement at 350°C for 30 minutes aided in this. Moreover, due to preheating of reinforcements the chances of volatile materials absorption inclusions in the castings as oxides are avoided [22].



Fig. 5. SEM and EDAX of LM4 + Si₃N₄ composite

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3.2 Hardness measurement

With the increase in wt.% of Si₃N₄, the hardness values of the composites improved. When compared to as-cast alloy the Vickers Hardness Number (VHN) of 1, 2, and 3 wt.% Si₃N₄ composites improved by 24, 25, and 28% (table 3). When SSHT and MSHT (without artificial aging) were performed on as-cast specimens, the hardness values decreased (table 3). This is because after solutionizing, a homogeneous solid solution is generated, which is transformed into supersaturated solid solution once quenched and this phase is soft and unstable, so, it exhibits lower hardness values than as-cast specimens [23]. MSHT + artificially aged specimens displayed better hardness than as-cast and SSHT + artificially aged specimens because of the presence of more number of precipitates formed, the results of which are in line with the other studies [24], [25]. Specimens aged at 100°C outperformed specimens aged at 200°C when hardness is compared, but the time taken to reach peak hardness is more for specimens aged at 100°C than aged at 200°C, this phenomenon can be explained by aging kinetics [26]. The comparison of hardness values among as-cast, SSHT, MSHT and aged at 100 and 200°C are shown in figure 6 (a-d). With the increase in wt.% of Si₃N₄ composite specimens exposed to MSHT + artificial aging at 100°C displayed the highest hardness which is 124% higher than the as-cast alloy hardness value.



Fig. 6. (a-d) Comparison of hardness values of as-cast alloy and LM4 + Si₃N₄ composites subjected to SSHT, MSHT, and artificial aging at 100 and 200°C

Table 3. Hardness value	s of LM4 and its composites	(as-cast and heat-treated in VHN)
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Hardness values in VHN								
Туре	As-cast	SSHT	MSHT	SSHT+200°C peak aged	MSHT+200°C peak aged	SSHT+100°C peak aged	MSHT+100°C peak aged	
LM4	70	64	56	89	116	95	130	



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Hardness values in VHN								
Туре	As-cast	SSHT	MSHT	SSHT+200°C peak aged	MSHT+200°C peak aged	SSHT+100°C peak aged	MSHT+100°C peak aged	
LM4+1 wt.% Si₃N₄	87	66	59	94	120	99	135	
LM4+2 wt.% Si₃N₄	88	70	61	100	125	105.5	143.5	
LM4+3 wt.% Si₃N₄	90	74	62.5	103	135	112	157	

3.3 RSM and ANN analysis

From the experimental results, it is confirmed that MSHT + artificial aging at 100°C gave the best hardness results. To statistically verify the results, RSM model was developed for prediction of hardness values using MINITAB, for which MSHT specimen readings were considered as input parameters. Estimated regression coefficients for peak hardness of MSHT specimens are shown in table 4. Analysis of Variance (ANOVA) for peak hardness is shown in table 5.

Table 4. Estimated regression coefficients for peak hardness of MSHT specimens

Term	Coef	SE coef	Т	Р
Constant	143.569	1.105	129.906	<0.001
wt.%	9.667	1.087	8.896	<0.001
Aging temperature	31.583	1.087	29.066	<0.001

Table 5. ANOVA for peak hardness							
Source	DF	Seq SS	Adj SS	Adj MS	F	Р	
Regression	5	15569.2	15569.2	3113.84	439.55	<0.001	
Linear	2	6545.7	6545.7	3272.85	462.00	<0.001	
Square	2	8933.3	8933.3	4466.63	630.51	<0.001	
Interaction	1	90.3	90.3	90.25	12.74	0.009	
Residual error	7	49.6	49.6	7.08			
Lack-of-Fit	3	44.6	44.6	14.86	11.89	0.018	
Pure error	4	5.0	5.0	1.25			
Total	12	15618.8					

 $S=2.66161,\,R\text{-}Sq=99.68\%,\,R\text{-}Sq(\text{pred})=97.05\%,\;R\text{-}Sq(\text{adj})=99.46\%$

P-value (<0.001) in tables 4 and 5 indicates that the factors and the model developed for optimizing and predicting the peak hardness values are in a good fit, usually, the p-value should be less than or equal to the significance level (0.05). The R² value is 99.68% which indicates that the factors majorly contribute to achieving peak hardness. R²(adj) is 99.46% which implies that the model is a good fit. It is observed in figure 7 that aging temperature has a major influence on the peak hardness followed by wt.% of reinforcement. This can be compared to experimental results as the specimens aged at 100°C exhibited better hardness compared to those aged at 200°C, also with the increase in wt.% of reinforcement, there is an improvement in hardness.



Fig. 7. 3D surface plot of peak hardness vs wt.% and aging temperature

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Figure 8 depicts the main effects plots for signal-to-noise (S/N) ratio of wt.% and aging temperature. As the option selected here is to display "larger is better", from the graphs we can infer that 3 wt.% and aging temperature of 100°C displayed better results. Table 6 shows the response table of S/N ratios, here significance of each factor is displayed in percentages, we can see that for wt.%, level 3 viz., 3 wt.% is having high significance, similarly for aging temperature, level 2 viz., 100°C has achieved more percentage indicating that it is having high significance on improving hardness values. Ranking for highest contribution in improving hardness is also displayed in which aging temperature secured first place.



Signal-to-noise: Larger is better

Fig. 8. Main effect plot for S/N ratios and mean

Table 6. Response table for S/N ratios

Level	wt.%	Aging temperature in °C
1	39.36	35.46
2	40.15	43.11
3	40.79	41.73
Delta	1.43	7.65
Rank	2	1

From ANN the following results were obtained, figure 9 shows the comparison between testing, validation, and training between projected and experimental data. Results validated that there is a strong relationship between expected and experimental results because of the model's training accuracy. The overall R-value obtained is 0.9182 which is close to 1, so it can be said that the performance is satisfactory, but the error of predicted values after the first training was large so, the model was retrained until minimum error is attained. Finally, the trained model is effective with low prediction error and can be used to predict hardness values for unknown data sets within the boundary limits. As a result, the created model can predict the hardness of heat-treated LM4 composites.

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Fig. 9. Regression graph for the developed network

The predicted values of RSM and ANN based on the regression analysis in comparison to experimental values are shown in table 7 and figure 10. Both the models predicted the values with minimum error viz., RSM with 1.7288 and ANN with 1.5699. Since the data set used is small there is minimal variation between RSM and ANN, with larger data set the results may vary, for this given set of data ANN is proved to be more effective in predicting hardness values with less error than RSM.

Table 7. Comparison o	f experimental and	predicted values	of RSM and ANN
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S. No	wt.%	Aging temperature in °C	Experimental	RSM predicted	ANN predicted
1	3	100	157	152.994	156.2159
2	2	100	143.5	143.569	140.9991
3	3	200	135	136.836	133.9535
4	1	0	56	54.336	54.86857
5	2	100	142	143.569	141.7556
6	2	100	145	143.569	146.8869
7	2	100	144	143.569	142.0994
8	2	100	143	143.569	141.0951
9	1	100	130	133.661	130.388
10	1	200	110	108.003	111.4253
11	2	200	122.5	122.661	120.9759
12	2	0	60	59.494	59.82237
13	3	0	62	64.17	61.57171

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Fig. 10. Comparison of experimental, RSM predicted, and ANN predicted peak hardness values of MSHT specimens

4 CONCLUSIONS

From the experimental results and statistical analysis, the following conclusions are drawn

- Composites were successfully fabricated using the two-stage stir casting method, SEM and EDAX confirm the presence and uniform distribution of reinforcement within the matrix.
- When compared to as-cast alloy the hardness of 1, 2, and 3 wt.% Si₃N₄ composites improved by 24, 25, and 28%, this is due to the addition of hard Si₃N₄ particulates to the soft LM4 matrix and good bonding between them.
- Precipitation hardening improved the hardness values of both as-cast LM4 and its composites. MSHT + artificially aged specimens displayed better hardness than as-cast and SSHT + artificially aged specimens as the number of precipitates formed is more in MSHT specimens.
- Specimens aged at 100°C displayed higher hardness than specimens aged at 200°C. LM4 + 3 wt.% Si₃N₄ composite specimens subjected to MSHT + artificial aging at 100°C displayed the highest hardness which is 124% higher than the as-cast alloy hardness value.
- Both RSM and ANN models predicted the hardness values with a minimum error of 1.7288 and 1.5699, from ANOVA analysis it is seen that the factors contribute effectively to the hardness of the composites, and as per the S/N ratio, the aging temperature is noted to be a major factor in improving hardness followed by wt.% of reinforcement. Aging at 100°C and 3 wt.% of Si₃N₄ addition are the best combination to achieve maximum hardness.
- When compared to RSM, ANN predicts hardness efficiently with the least error.

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Paper submitted: 26.05.2022.

Paper accepted: 28.07.2022.

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