

Optimization of Age Hardening Behaviour of AA2024 Hybrid Composite Reinforced with Red Mud and Fly Ash through RSM



G. Siva karuna, P. Suresh Babu

Abstract: Hybrid AA2024 Metal Matrix composite find explicit application, so important factors like Hardness, lightweight is considered and hybrid composite was synthesized by stir casting route. In this investigation, the effects of age hardening on Hardness of AA2024 reinforced with industrial wastes is measured by using a Vickers hardness test and the influence of different process parameters are studied using Design Expert. A Response Surface Methodology is an effective technique used to observe the impact of age hardening parameters and their interconnection on surface hardness of the prepared hybrid AA2024 composite. A mathematical model was created to optimize the weight percentage of reinforcement and artificial ageing process parameters for maximum Hardness. Adequacy and lack of fit of the developed model were checked using the Analysis of variance Technique. Hardness is taken as the response and optimum parameters are obtained by using the quadratic model. Based on the results, it can be perceived that the weight percentage of Red mud, ageing temperature and ageing time has shown a notable effect on the response. For a specifically optimized parameter, the hardness is improved and it was observed that ageing temperature is the most influencing factor based on the RSM. The optimized parameters for the desired response are Ageing Temperature is 198.87°C, Ageing time is 6.82884 hours, and Red mud wt % is 4.2865 respectively. A confirmation test is carried out with the optimized parameters experimentally and its error is less than the 5 % which shows good results with RSM. Surface morphological studies are carried for the optimized Age Hardened hybrid composite and it showed the uniform distribution of reinforcements in the matrix. Because of the increased hardness, these hybrid composite would perceive real time applications like Orthopaedic braces, aircraft structures and manufacturing crew machine products.

Keywords: Hybrid Composites, Nano Materials, Response Surface Methodology

I. INTRODUCTION

Among all the materials, aluminium metal matrix composites possess unique characteristics such as high thermal stability with augmented thermal conductivity, greater response to age hardening, high specific strength, and stiffness. AA2024 alloy has the inflated hardness. With all unique characteristics, these composites are finding a variety of applications in the automobile and aerospace industries [1].

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Recent years, usage of industrial wastes as the secondary reinforcements in the synthesis of composites is gaining more and more importance. By inclusion of these waste materials in the synthesis of MMC result in a production of low-cost aluminium products due to cheap and readily available of reinforcements [2]. Besides the readily available with low cost and lower densities, most of the researchers have reported the potential and limitations of the use of wastes as reinforcements [3],[4]. With the usage of the fly ash as the reinforcement in the aluminium alloys, there is an augmentation in the properties like compression, tensile and hardness strength. For aluminium composite with more than 15 weight percentage fraction of fly ash particles, the tensile strength was seen to be decreased [5]. Red mud is also used as the reinforcement because it comprises of ceramic constituents so the addition of red mud particles enhances the hardness values [6].

Nanoparticles found potential application in aircraft structural application where enhanced mechanical characteristics are essential. Due to larger milling time, the intensity of the peaks in the XRD pattern was significantly reduced. For 30 hours of milling, the crystallite got reduced to 42nm [7]. Materials with nanocrystalline have found to be advantaged over their microcrystalline parts in hardness, fracture, toughness and ductility [8]. with the increase of the weight percentage of the red mud as reinforcement would lead to higher hardness values in aluminium metal matrix composites. By incorporation of red mud as reinforcement in metal matrix composites will reduce the total weight of the composites as it is lightweight and due to its hardness, it enhances the hardness of the synthesized MMC [9]. Heat treatment is another efficient method, which is associated with increasing the strength of the material. Heat treatment is a proficient process applied to Al-based MMCs to increase the Hardness of the particulate MMCs. Hardness is a very important factor which affects the strength of the material. The factors like Volume Percentage, particle size and method of synthesis will influence the hardness of the prepared MMC [10]. The Artificial age hardening process consists of solutionizing, quenching and ageing at elevated temperature (artificial ageing) will affect the heat treatment process of the aluminium alloys [11]. During the long process of heat treatment at elevated temperature, may lead to modification of the composition of the matrix due to the reactivity between the reinforcement and the matrix [12]. To analyze multiple parameters, recent days factorial design of experiments (DOE) has emerged as an important tool [13],[14].

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For various properties of MMCs, many researchers have used this methodology and developed models [15],[16]. For predicting hardness of an Al/Al₂O₃ composite, a mathematical model was developed using response surface methodology and observed the effects of various volume fractions of the reinforcements [17].

Therefore, the present investigation is to synthesize aluminium metal matrix composite with non-conventional reinforcements and to study the influence of the artificial age hardening process on the AA2024 hybrid composite reinforced with the industrial wastes and the influence of various factors such as ageing time, ageing temperature and weight percentage of Red mud on the hardness of the prepared composite by RSM methodology using the Design of Expert software. The optimized factors for attaining the peak hardness are analyzed using the DOE.

II. MATERIALS AND METHODS

A. Materials Used

An AA2024 was used as the matrix material for the required composite in the form of a cylindrical rod. Industrial wastes such as Fly ash powder with a particle size of 53µm and Red mud Powder with a particle size of 44 nm are used as the reinforcement particulates in the preparation of the Hybrid MMC. Fly ash is the by-product produced during the burning of coal in Thermal Power plant and Red mud is the by-product produced during the synthesis of alumina for the production of alumina. Fly ash was brought from the Thermal Power Plant, Vizag steel plant, Visakhapatnam and Red mud were brought from the NALCO, Odisha. Sieve analysis is carried for the taken reinforcements and the finest size in sieve analysis is considered for the synthesis of the composite which is 53µm. Both reinforcements are heated up to the temperature of 450°C for 30 min to avoid moisture and dissolved gases present in it.

B. Nano Conversion of Red Mud

Nano conversion of micro Red mud is carried out in the High energy ball mill (Emax, Retsch) with a speed of 300 rpm. Milling is performed with Tungsten carbide (WC) balls with a ball to powder ratio of 10:1 for 20 hours. Toluene is used as the surfactant to inhibit agglomeration of powder during ball milling. After milling the Red mud is preheated in furnace up to 50° C to remove moisture and stored in desiccators.

C. Characterisation of Red Mud Nano Particles

Red mud Nanoparticles characterisation after mechanical nano conversion is very important to study their crystallite size, and the lattice strain, because the phase constitution and transformation of reinforcements in the matrix is gravely reliant upon them. Additionally, individual can also characterize the transformation behaviour of prepared nanopowders in any heat treatment process. crystalline size in nanoparticles can be determined by X-ray diffraction which depends upon the peak width. XRD is carried out for the prepared Red mud and the peaks of the x-ray diffraction are shown in the Fig1. The most frequently used equation to determine crystalline size based on the XRD data is the Scherer Equation as shown in the equation 1.

$$D = k \lambda / (\beta \cos\theta) \quad (1)$$

Where ,

D = Crystalline Size

β = Peak full width at half- maximum (FWHM)

θ = Diffraction angle

λ = Wavelength of the X-Rays used.

k = A dimensionless shape factor whose value is close to 0.9.

Based on the above equation, the crystallite size is reduced from 53 µm to 44 nm during 30hours of milling in high energy ball mill. The intensity of peaks in the XRD pattern was observed to small and the peak broadening was considerably large due to larger milling time.

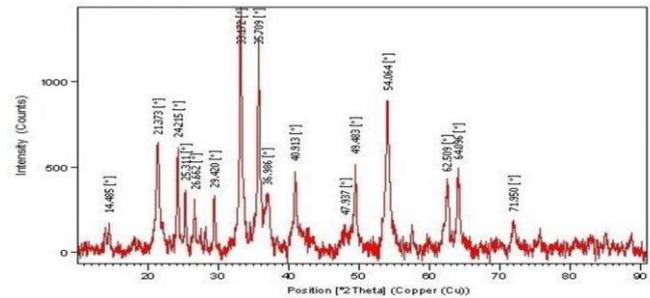


Fig. 1. X-ray diffraction of Red mud

D. Synthesis of Hybrid Composite

The Stir casting process is used for the preparation of hybrid composites. Three different models of composite materials are fabricated by stir casting process with a fixed weight percentage of micro Fly ash of 15% and varying weight percentage of Red mud of 2%,4% and 6%. The stir casting equipment with a stirrer arrangement is shown in Fig 2. The Casting process is carried out at the temperature of 700°C with the stirrer stirring speed of 700 rpm for 10 min [18]. Argon inert gas is used during casting to maintain the inert atmosphere. Casting defects like porosity and inclusion due to clustering of reinforcements particles at some places in composite due to the non-uniform rate of reinforcement feed has been controlled by uniform feed rate of reinforcement.

Reinforcement feed rate used in present work is 0.5 gram per second. Stirring temperature is gradually increased to 700°C. After reaching 700°C the stirring speed is gradually increased from 0 to 700 rpm with aid of the controller. During Stirring, preheated reinforcements are added with a feed rate of 0.5 gram per sec by using conical hopper manually [19]. After complete stirring, the stirrer speed is gradually decreased to zero and without any time lag, the prepared molten slurry is poured into the mould cavity and allowed to cool up to reach room temperature. During pouring, care is taken to keep the crucible at a minimum distance from the mould to avoid gases trapping.



Fig. 2. Stir casting equipment

E. Artificial Age Hardening

AA2024 and the prepared composites were subjected to Solutionized hardening at 450° C in a muffle furnace for 2 hours and were water quenched at room temperature. All specimens were then subjected to Artificial ageing at 150°C, 200°C and 250°C with an ageing duration of 2, 4,6,8 and 10 hours and later allowed to cool up to room temperature. The age-hardening response of the prepared small composites was characterized by measuring hardness using Vickers hardness measuring equipment performed at room temperature with a load of 1 kgf for 15 seconds. To find the peak ageing time and temperature hardness values are determined. The surface is prepared before the hardness test to remove oxide formed during ageing. The hardness test specimens are shown in Fig 3.

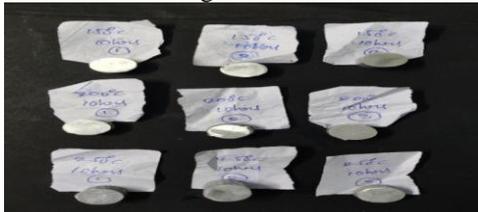


Fig. 3. Age hardened Hybrid composite specimens

III. RESPONSE SURFACE METHODOLOGY

Response surface methods are widely used in many fields [20],[21]. To perform analysis and optimize various parameters which affect the final desired characteristics [22]. In the present investigation, Design expert 11 software is used with 3 factors. The age hardening of composite consists of factors like weight percentage of Red mud, ageing time and ageing temperature. The process parameters considered for the analysis are the composition of Red mud (A), ageing temperature(B) and ageing time (C) is chosen at different levels. The experimental factors and levels are given in table 1. Average Hardness values and factors are outlined in Table 2. To find the optimized age hardening parameters, a quadratic mathematical model was developed by using the RSM. It can be used to establish response regression form and used to find out the coefficient of the mathematical model.

Table-I: Process variables used

Factors	Low Limit	High Limit
Composition of Red mud(A)	2%	6%
Ageing Temperature(B)	150°C	250°C
Ageing Time (C)	2 Hrs	10Hrs

Table-II: Experimental results of Vickers Hardness Number for the Prepared Composites

S.No	Composite	Temperature °c	Vickers Hardness Number (VHN)				
			2hrs	4hrs	6hrs	8hrs	10hrs
1	AA2024+2%RM+15%FA	150	136.8	138	140.62	139.14	137.76
2	AA2024+2%RM+15%FA	200	138.11	145.21	167.8	150.21	142
3	AA2024+2%RM+15%FA	250	137.65	140.73	143	144.73	139.65
4	AA2024+4%RM+15%FA	150	139.9	140.63	142.56	145.39	150.65
5	AA2024+4%RM+15%FA	200	145.38	168.88	183.27	170.63	167.86
6	AA2024+4%RM+15%FA	250	142.63	148.54	150.54	155.39	151
7	AA2024+6%RM+15%FA	150	141.65	145.38	143.21	142.6	141
8	AA2024+6%RM+15%FA	200	142.62	148.11	150.21	157.85	150.91
9	AA2024+6%RM+15%FA	250	142.8	144.8	148.78	149.65	148.14

IV. RESULT AND DISCUSSION

RSM is one of the most efficient statistical approaches of which reduces the number of trials of the experiment and also to determine the combined effect of various input parameters on the desired response. In this work, RSM is completely depended on the factorial design of experiments which consists of two factors (weight percentage of Red mud, Ageing temperature) of three levels and one factor (Ageing time) with 5 levels was used.

The experimental results were statistically analysed by RSM technique using Design-Expert software which is extensively used in many interdisciplinary research fields. ANOVA is performed on experimental results to check the

statistical significance of the Quadratic model of the hardness. Process variables such as weight percentage of Red mud(A), Ageing Temperature(B) and Ageing Time (C) on response variable such as Hardness are used experimental data for analysis of variance. The results are analysed with a confidence level of 95% and P-value <0.05 indicates model terms are significant. The model F-values of 8.51 implies that the model is significant. There is only a 0.01% chance that an ' F ' Value this large could occur due to noise. The predicted R² is in reasonable agreement with adjusted R² i.e. the difference is <0.2. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable.

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In the present work, a ratio of 11.028 indicates an adequate signal. This model can be used to navigate the design space. ANOVA table for the Vickers Hardness is given in table 3.

The regression equation is obtained from the design expert software in terms of actual factors is used to predict the Vickers hardness of the age hardened specimens.

$$Vhn = +164.62 + 1.88 * A + 2.42 * B + 3.07 * C + 0.3445 * AB + 0.4643 * AC + 1.40 * BC - 8.25 * A^2 - 11.83 B^2 - 7.71 * C^2$$

A= Weight percentage of Red mud (Comp)

B= Ageing Temperature,

C= Ageing Time

The normal probability plots of residuals versus the predicted response for hardness are shown in Fig 4. It disclosed that the residuals mostly fall on a straight line,

which implies that the errors are normally distributed. This implicit that the model proposed is ample and there is no reason to suspect any violation of the independence or constant variance assumption [23]. For the developed RSM based mathematical model, the influence of parameters was examined. Fig 5(a) shows the Predicted versus Actual values of Hardness (VHN) are very useful in validating the experimental data. The values fall near the straight line which indicated that the experimental data can be acceptable. Fig 5(b) shows the residual versus predicted values of Hardness. There is no specific pattern formed in the plot and the residuals are falling near the straight line, which indicates that the errors are normally distributed.

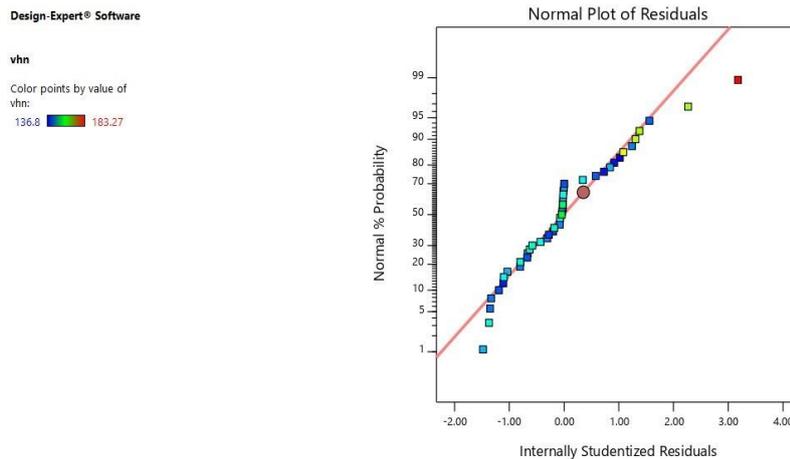


Fig. 4. Normal Plot of Residuals

Table-III: ANOVA Table for Vickers Hardness

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value	
Model	3075.46	9	341.72	8.51	<0.0001	significant
A-comp	105.66	1	105.66	2.63	0.1138	
B-temp	176.34	1	176.34	4.39	0.0434	
C-time	212.13	1	212.13	5.28	0.0276	
AB	2.37	1	2.37	0.0591	0.8093	
AC	3.23	1	3.23	0.0805	0.7783	
BC	29.22	1	29.22	0.7276	0.3995	
A ²	680.02	1	680.02	16.93	0.0002	
B ²	1398.46	1	1398.46	34.82	<0.0001	
C ²	468.06	1	468.03	11.65	0.0016	
Residual	1405.79	35	40.17			
Cor Total	4481.25	44				

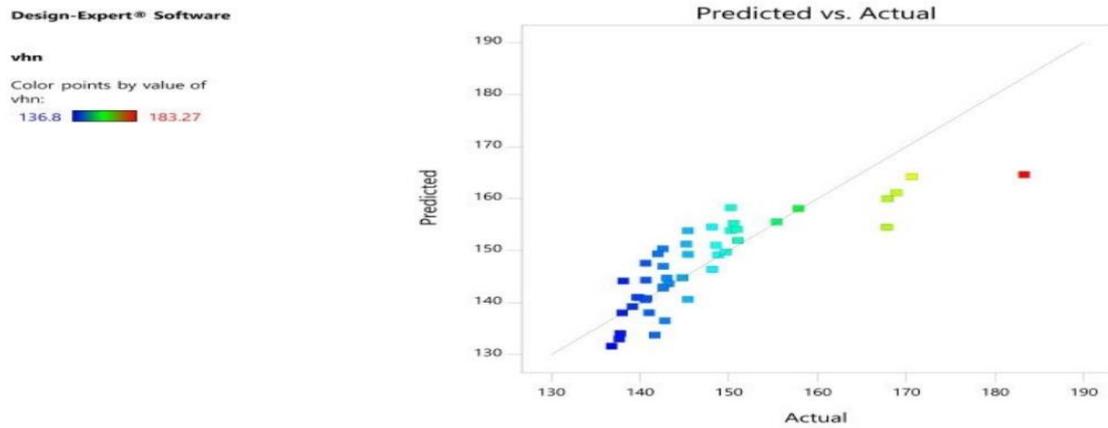


Fig. 5(a). Comparison of Predicted and Actual Values of VHN

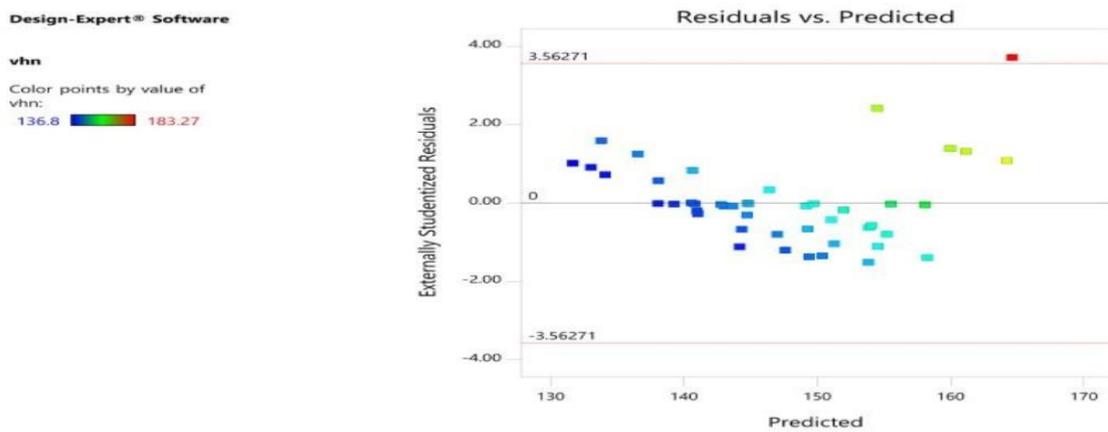


Fig. 5(b). Comparison of Residuals and Predicted Values of VHN

4.1 Effect of factors on the Hardness

The change in Hardness under the influence of weight percentage of Red mud, ageing temperature and ageing time is shown in Fig 6. From the graph, it is observed, as the weight percentage of Red mud increases the hardness of the composite increases up to the optimal value and further decreased. It is noticed that as ageing temperature and ageing time increases the hardness of the prepared composites increases which further decreases. The interaction effects of the variables are represented in Fig (7-9). From Fig 7, the hardness is high at the moderate values of compositions of Red mud and ageing temperature. It is noticed that the

hardness values are low at the lower levels of the ageing temperature and weight percentage of the Red mud.

From Fig 8 and 9, the same effects can be observed in the interaction plots for the response versus the combination of the remaining two factors. It has been observed that ageing time has shown a noticeable effect on the hardness concerning the weight percentage of the Red mud. As the Ageing Temperature increases, the hardness value also increases with the ageing time and decreases after a certain extent.

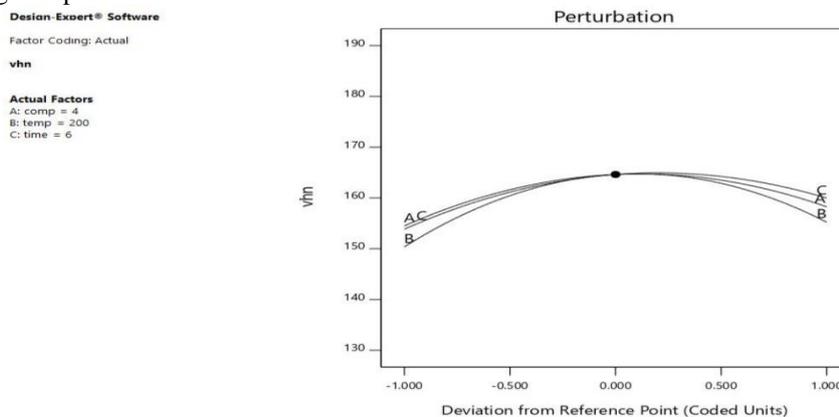


Fig. 6. Variation of Hardness (VHN)

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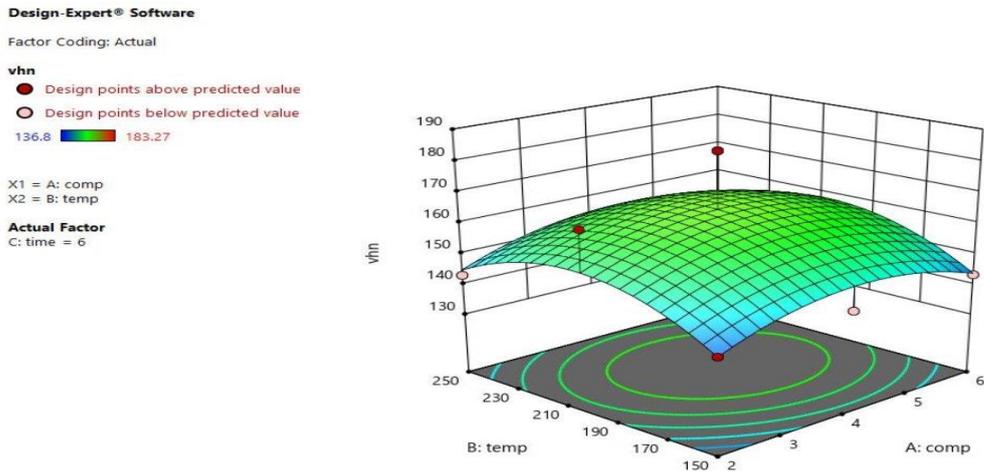


Fig. 7. Effect of composition of Red mud and ageing temperature on hardness

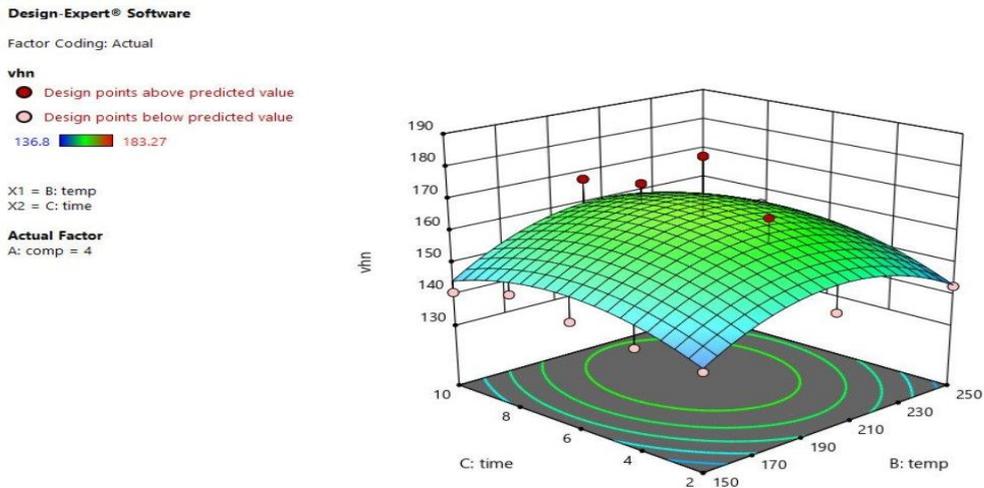


Fig. 8. Effect of ageing temperature and ageing time on hardness

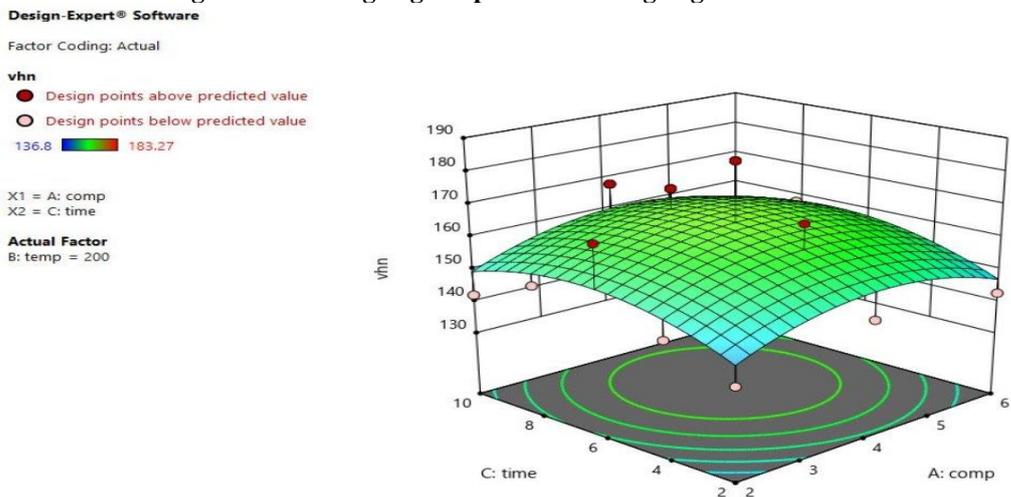
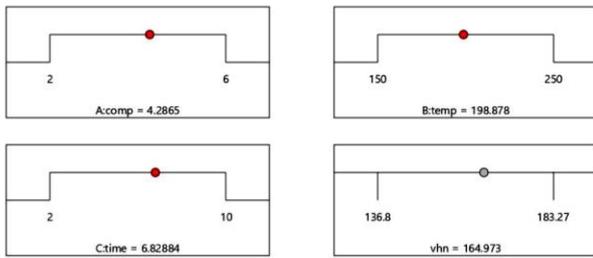


Fig. 9. Effect of composition of Red mud and ageing time on hardness

4.2 Optimization of Hardness factors

The main aim of optimization is to provide the optimum hardness parameters which give the maximum Hardness. In this work, analysis is based on "Larger is better" Concept which means higher hardness is considered as optimum. The Input hardness parameters were selected based on the desirability value. concerning hardness as a response, the values of the most effective parameters were selected based upon the desirability values close to 1 unit. The optimal solution for the hardness is shown in Fig 10. The optimal

solution for the hardness is weight percentage of Red mud is 4.2865%, Ageing Temperature is 198.878°C and Ageing time is 6.82884 hours. From Fig 11, it can be noticed that approximately 100% of desirability is achieved for the output response and shows the overall desirability function of the responses. The bar graph indicates each variable satisfying the criteria, a value close to one is considered to be proficient.



Desirability = 1.000

Fig. 10. Ramp function graph of desirability of Hardness

4.3 Validity of the Hardness Model and confirmation of experiments

Equation of response for the model is derived from quadratic regression. To check the validity, a confirmation test must be conducted. Parameters selected for the

Table-IV: Confirmatory and comparison of Experimental and RSM model

Exp No.	Hardness Parameters			Response parameter		Error (%)
	Composition (A)	Ageing Temperature (B)	Ageing Time (C)	Exp.	RSM	
1	4.28	199	6.8	163.24	164.973	1.05
2	4.28	200	6.8	162.78	164.973	1.4

4.4 Surface Morphological Studies of Optimized Age Hardened Hybrid AA2024 Composite

The surface morphological studies for the optimised Age-hardened hybrid AA2024 composite specimens were studied using field emission scanning electron microscope (FESEM), JEOL/JSM and7100F model under 20Kv accelerating voltage. Fig.12, FESEM image justify the distribution of selected reinforcement particles in the matrix material. Nano red mud particles and fly ash particles are found evenly distributed on the matrix material which influences the hardness of the composite material.

Due to an increase in the ageing time, they have precipitated into larger particles under the area of observation this could be due to coalescence of reinforcement particles. By artificial heat treatment, it has attributed to the formation of intermetallic precipitates due to the chemical composition of reinforcement and matrix materials. By introducing of fly ash and red mud reinforcements into the matrix material assists zone kinetics and phase formation reaction at the time of heat treatment process. The Acceleration of the precipitation in the matrix material is greatly influenced by the percentage of reinforcements in the matrix. If the ageing parameters are increased beyond the optimised parameters then it would result in the decrease of the hardness of prepared composite attributable to a coalescence of the precipitates into larger particles so that they will form few obstacles to the movement of dislocation.

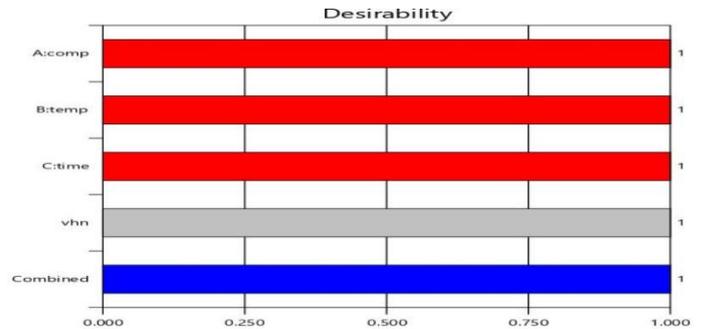


Fig. 11. Desirability bar graph of Hardness

confirmation experiments must lie within range for which the equation was derived. The comparison between the experimental values and predicted values for the hardness are listed in the below table 4.

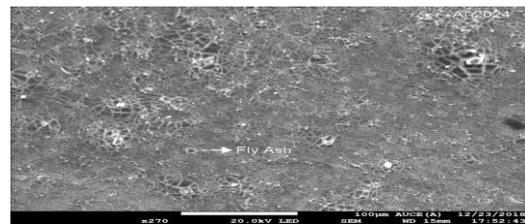


Fig. 12. FESEM image of optimized age hardened composite

4.5 Energy-dispersive X-ray spectroscopy (EDS) Studies of Optimized Age Hardened Hybrid AA2024 Composite

For the optimized age hardened hybrid AA2024 composite, to confirm the presence of elemental composition of both reinforcements and matrix an EDS analysis is carried for the taken spectrum and results of the EDS analysis are given the below Fig 13. It shows the analogues peaks of individual elements to affirm their presence in prepared specimens.

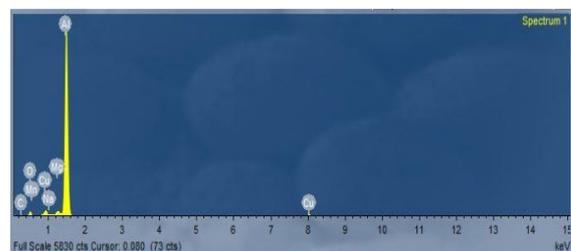


Fig13. EDS Analyses result image of optimized age hardened composite

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V. CONCLUSION

In the present investigation, AA2024 metal matrix composites are synthesized by the stir casting process and prepared composites are undergone artificial age hardening. For the Age hardened composites, the Hardness test was performed and factors which affect the Hardness of composites are optimized by using Design Expert Software and then following conclusions are drawn.

- Successfully prepared the required AA2024 hybrid composite with red mud and fly ash particles as reinforcement using stir casting method.
- Hardness is high at the moderate values of compositions of Red mud and ageing temperature. It is noticed that the Hardness values are low at the lower levels of the ageing temperature and weight percentage of the Red mud.
- As the weight percentage of Red mud increases the hardness of the composite increases up to the optimal value and further decreased. It is noticed that as ageing temperature and ageing time increases the hardness of the prepared composites increases which further decreases after a certain range of Ageing Temperature and Time.
- ANOVA results for the Hardness indicated that the Ageing Temperature is the most influential factor followed by Wt% of Red mud, Ageing time for the Hardness of the Hybrid Composite.
- By RSM method, Optimization for Vickers Hardness through Artificial Ageing for the prepared composites is Maximum Hardness of 164.973 VHN, the optimum value of Ageing Temperature is 198.878°C, Ageing time is 6.82884 hours, and Red mud wt % is 4.2865 respectively.
- The Confirmation experiments showed that the error between the experimental and predicted value of wear rate lies within the range of $\pm 5\%$ for the desired response.
- FESEM analysis exhibit the existence of the particle in reinforcement and their uniform distribution throughout the matrix.

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