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Research Paper

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Optimization of fabrication process parameters of stir-cast Rice Husk Ash (RHA) and Alumina (Al_2O_3) reinforced Aluminium – Silicon hybrid composite

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ABSTRACT

Materials research and development have shifted from monolithic materials to composite materials owing to the benefits of composites such as reduced weight, low cost, high performance, economic and environmental sustainability especially in the automotive industry. Agricultural waste derivatives have shown great potential of been utilized as complimentary reinforcements in the development of metal matrix composites. The present study is aimed at optimizing the fabrication process parameters particle size (S) of RHA and weight percent(wt%) of RHA as a complimentary reinforcement. Two sets of nine Aluminium-Silicon reinforced RHA/ Al_2O_3 hybrid composites were fabricated using two-step stir casting technique. Mean particle size and weight percent of the reinforcement were varied according to Taguchi L9 Design of Experiment (DOE). One set of the cast composite were then solution heat treated by heating to 530°C for 2hrs quenching in water at 60°C and age hardened at 175°C for 4hrs. Mechanical properties were determined for both heat treated and non-heat-treated composites. Regression models were developed for mechanical properties of the heat-treated composites. The fabrication process parameters - mean particle size of RHA (s) and weight percent of RHA (wt.%) were then optimized using Taguchi method using Minitab 18 Software for the multiple performance of mechanical properties of the heat-treated composites. Multiple performance optimization showed the most dominant factor levels are level 3 for ultimate tensile strength (UTS) and hardness (HRB), while for wear rate (WR) dominant factor level was level 1. ANOVA results at 95% confidence level showed that mean particle size(s) of RHA had a highest contribution (61.13% in UTS, 83.70% in HRB and 78.17% in WR). The results of this optimization have shown that particle size of RHA and weight percent of RHA have significant effect on the ultimate tensile stress UTS, hardness HRB and wear rate WR of the hybrid composite.

Keywords - Hybrid composites, Matrix materials, Reinforcements, Heat treatment, Mechanical properties, Taguchi optimization

I.INTRODUCTION

Metal matrix composites have been identified as a reliable solution to present days advanced engineering materials. The motive behind their application in components for the transportation sector is based on the requirement for weight reduction and in pursuit of improved efficiency and performance at a reduced cost. So far, researched works have been carried out by incorporating hard ceramic particles such as Silica dioxide (SiO_2), alumina (Al_2O_3), Silicon Carbide (SiC), Fly Ash (FA), graphite (Gr) and a host of synthetic materials to soft light matrix such as Aluminium, magnesium and titanium to mention a few. (Bodurin *et al*, 2015).

This has been limited by high cost, and non-availability of the ceramic and industrial waste material especially in developing countries. Other challenges reported of interest in using ceramic particulates are inferior ductility, low fracture tightness and in ability to predict corrosion behavior (Hpydi *et al*, 2013; Macke *et al*, 2012; Das *et al*, 2014).

Several approaches have been adopted to resolve these problems. One such approach is finding alternative cheaper reinforcements which are aimed at providing solution to problems posed by high cost and limited availability of ceramic particulates. Industrial waste and agricultural waste derivatives are some of the alternatives reinforcing materials that have been investigated. (Bodurin *et al*, 2015; Madakson *et al*, 2012; Anilkumar, *et al*, 2012; Loh *et al*, 2013).

Results obtained from investigations carried using these alternative reinforcements have been promising as they showed significant improvement in the properties of the composite developed over the unreinforced metal or alloy. Among the several categories of metal matrix composites, aluminum-based composites are used in spread acceptance especially in applications where weight and strength are of prime concern. Lakhvir *et al* (2013), noted that the though the application scope of metal matrix composites is expanding, the major hindrance is in the production. Also, mechanical properties of metal matrix composites are highly influenced by type of metallic phases and fabrication process parameters such as fabrication method adopted, heat treatment conditions, type of reinforcements, particle size of reinforcements and percentage of reinforcements.

Therefore, optimization of fabrication process parameters, conditions of heat treatment, selection of reinforcement and matrix phase are highly essential for improved mechanical properties of metal matrix composites. Rice husk ash has been identified as a possible complimentary reinforcement in the development of metal matrix composites. Its application aims at reducing cost of production of metal matrix composites. (Bodurin *et al*, 2015). This work is geared toward utilizing the properties of RHA as optimization parameters as the reinforcement properties have been identified by many researchers as important factors on the mechanical properties in the development of metal matrix composites

The purpose of optimization is to achieve the best design relative to a set of prioritized criteria or constraints. These include maximizing or minimizing factors such as productivity, mechanical properties, reliability, longevity, efficiency and utilization. Design optimization allows thousands of designs to be experimented thus speeding up the process by trying out designs which are diverse. This promotes broadening of the solution possibilities. (Manikandan, 2017).

Arulraj *et al*, (2017) Optimized process parameters in stir casting of hybrid metal matrix (LM25/SiC/B₄C) composite using Taguchi method. The effect of three major stir casting parameters (stir speed, stir duration and preheated temperature of reinforcement materials) was investigated. Analysis of the result using the conceptional signal to noise (S/N) ratio approach showed that the optimum level of casting parameters to obtain good tensile strength for stir casting of LM25/Si C/B₄C hybrid composite are stir speed of 180 revolutions per minute, stir duration of 55minutes and preheated temperature of reinforcement particle of 400°C. Radhika *et al*, (2011) investigated the tribological behavior of Aluminium/Alumina/Graphite hybrid metal matrix composite using Taguchi technique. Investigation to find the influence of applied load, sliding speed and sliding distance on wear rate as well as the coefficient of friction during wearing process was carried out. Results showed that sliding distance had the highest (46.8%) influence followed by applied load 31.5%) and sliding speed (14.1%). Singh *et al*, (2013) performed optimization of process parameters for stir casted Aluminium matrix composite using Taguchi method. The investigation was done to study the effect of different input process parameters namely: Particle size of alumina (75µm, 105µm and 150µm), weight percent of alumina (3%, 6% and 9%) and stirring time (15, 20 and 25minutes) on the hardness, impact strength and tensile strength. An L₉ orthogonal array table was used to make different specimens. The contribution of each process parameter on hardness, impact strength and tensile strength was analyzed using ANOVA. Result showed that weight percent of alumina, stirring time had significant effect on the hardness, impact strength and tensile strength. Baradeswaran *et al*, (2013) performed statistical analysis and optimization of wear behavior of Al- Al₂ O₃ composite using Taguchi technique. The composites were prepared by conventional liquid casting method with varying Al₂O₃ content. Wear test was

conducted with pin-on-disc apparatus. Controlling parameters were applied load of 10, 20 and 40N and sliding distance 200mm, 600mm and 1200mm. Statistical analysis was done using response surface methodology and Taguchi technique over design of experiment with regression equation using MINITAB software. Graphical and analytical results of Taguchi showed the optimum combination of parameters of 10N load and sliding distance of 400mm for minimum wear loss.

Going by the literatures reviewed, it can be gainfully accepted that mechanical properties of metal matrix composites are highly influenced by the selection of effective and controlled fabrication process parameters which include heat treatment conditions, type of matrix, particle size, percentage and type of reinforcement. Therefore, optimization of fabrication process parameters, is highly essential for strength-based applications of composite materials. This work presents a comparative study of mechanical properties of an Al – Si alloy reinforced with Al_2O_3 and RHA as a complimentary reinforcement to produce a hybrid composite both in as – cast and heat-treated condition, fabricated through two – stir casting method. Keeping a view on the industrial demand for materials with optimal combination of strength - based properties. Multiple response optimization of processing parameters is conducted and linear regression models are developed for three mechanical properties of the heat-treated metal matrix composites namely: Ultimate tensile strength (UTS), Hardness and Wear rate.

II. MATERIALS AND METHODS

2.1 Design of experiment

For the fabrication of the hybrid composite mean particle size (S) and weight percent (wt. %) of RHA reinforcement were considered as process parameters. Three levels for each are as presented in Table 1. The weight percent of alumina was kept constant at 5%. The hybrid composites were fabricated as per Taguchi L9 orthogonal array (OA) design of experiment (DOE) as presented in Table 2. Two sets of the nine different hybrid composites samples were produced and numbered accordingly as performed by Das *et al*, (2016) and Singh *et al*, (2013).

Investigation of the experimental outcome used signal to noise (S/N) ratio to support the determination of the finest process design and as a performance characteristic to measure the process robustness and to evaluate deviation from desired values. (Sylajakumari *et al*, 2018). The S/N ratio, a logarithmic function is computed by assessing the proportion of signal (mean) to the noise (standard deviation). In order to diminish noise and the effects of uncontrollable factors, high values of S/N ratios are preferred. High S/N ratios indicate improved quality of the product. There exist three types of S/N ratios, namely; higher-the-better, nominal-the-best and smaller-the-better as shown in equations (1) – (3). (Sylajakumari *et al*, 2018).

$$\left(\frac{S}{N}\right) = -10 * \log_{10} \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right) \dots \dots \dots (1)$$

$$\left(\frac{S}{N}\right) = 10 * \log_{10} \left(\frac{y^2}{S^2} \right) \dots \dots \dots (2)$$

$$\left(\frac{S}{N}\right) = -10 * \log_{10} \left(\frac{1}{n} \sum_{i=1}^n y_i^2 \right) \dots \dots \dots (3)$$

Where n is the number of experiments, y_i represents the response value of the i th experiment in the OA, y^2 the mean and S^2 the variance of observed data.

Table 1. Fabrication Process Parameters and their Levels.

Process parameters	Levels of Parameters		
	1	2	3
Mean particle size, S(μm)	125	250	355
Weight percent (Wt.%) RHA	2.5	5.0	7.5

2.2 Methodology.

Two-step stir casting technique was used for the fabrication of the hybrid composite as performed by Alaneme and Aluko, (2012) and Alaneme *et al*, (2013). The quantities of aluminum alloy, alumina (A1203), RHA and 0.1wt% magnesium were evaluated using charge calculations.

Table 2. Taguchi L9 Orthogonal Array (OA).

Sample number	Sample composition	Particle size S(μm)	Weight percent (wt.%) RHA
A1	2.5 wt. % RHA + 5.0 wt.% Al_2O_3	125	2.5
A2	5.0 wt. % RHA + 5.0 wt.% Al_2O_3	125	5.0
A3	7.5 wt. % RHA + 5.0 wt.% Al_2O_3	125	7.5
B1	2.5 wt. % RHA + 5.0 wt.% Al_2O_3	250	2.5
B2	5.0 wt. % RHA + 5.0 wt.% Al_2O_3	250	5.0
B3	7.5 wt. % RHA + 5.0 wt.% Al_2O_3	250	7.5
C1	2.5 wt. % RHA + 5.0 wt.% Al_2O_3	355	2.5
C2	5.0 wt. % RHA + 5.0 wt.% Al_2O_3	355	5.0
C3	7.5 wt. % RHA + 5.0 wt.% Al_2O_3	355	7.5

The alumina particles and RHA were initially preheated to a temperature of 250°C for 5 minutes to help improve wettability with the Aluminum alloy melt. Equally, the crucible pot was preheated to red-hot to ensure that the crucible pot was free from moisture and any volatile matter that could lead to contamination.

They aluminum alloy ingots were charged into a charcoal fired crucible furnace and heated to a temperature of $750 \pm 30^\circ\text{C}$ (above the liquidus temperature of the alloy) which is the casting temperature of aluminium. At this temperature, the slag was scooped and removed from the surface of the molten alloy. The alloy was then allowed to cool on the furnace to a temperature of 600°C. The preheated alumina, RHA and 0.1 wt. % magnesium was then added at this temperature and stirring of the slurry was performed manually for 5 to 10 minutes. The composite slurry was then superheated to 780°C and a second stirring operation was performed using a mechanical stirrer at a speed of 200r.p.m for 10 minutes to help improve distribution of the alumina and RHA particles. The molten composite was then cast into prepared sand molds and allowed to cool. After cooling, hybrid composite samples were then obtained. Samples were then prepared for density measurement tensile strength test, hardness test and wear test. Temperature measurements were performed using a hand-held double laser infrared thermometer model 201506011240.

2.2.1 Heat Treatment

One set of the developed hybrid composite samples were then subjected to T6 heat treatment condition. This involved solution heat treatment at 530°C for 1 hour, quenching in water at room temperature and age hardening at 175°C for 4 hours as performed by Sridhar Bhat *et al*, (2014). This was performed using an electric resistance furnace model 5 - x - 12.

2.2.2 Mechanical Properties Test

Tensile test was carried out according to ASTM standard D3039: Standard code for tensile test of composites using a Hounsfield Tensiometer. Hardness was determined in accordance to ASTM Standard E18 - 79: Rockwell Hardness and superficial hardness of metallic materials using Indentec universal hardness testing machine model 8189.5LKV(B) while the wear rate test was carried in accordance to ASTM G99- 9C: Standard test method for wear test with a Pin - on - Disc tribometer TRB of Anton Paar.

2.2.3 Microstructural Examination.

Crystallographic specimens were prepared as per ASTM E3 - 95 standard for optical microscopy. SEM analysis of the composite showed needle like primary and acicular hyper - eutectic Si at the interdendritic regions (A), coarse primary α - Al dendrites (B) and acicular eutectic Si phase with stacked Fe rich γ phase having a skeletal shape as shown in Figure 1. (Mangai *et al*, 2023)

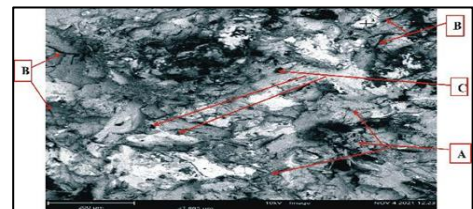


Figure 1. SEM micrograph of a representative sample.

III. RESULTS AND DISCUSSION

3.1 Chemical composition of samples

The chemical composition of both the aluminium alloy is presented in Table 3 while that of RHA is presented in 4.

Table 3 Chemical Composition of the Aluminum Alloy.

Element	Na	Mg	Si	P	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Zn	Al
%	0.98	0.97	18.79	0.02	0.02	0.49	0.03	0.14	0.05	0.02	0.14	1.36	0.78	rest

Table 4 Chemical Composition of RHA

Oxide	SiO ₂	CaO	K ₂ O	Na ₂ O	MgO	Al ₂ O ₃	P ₂ O ₅	SO ₃	Cl	TiO ₂	Cr ₂ O ₃	Mn ₂ O ₃	Fe ₂ O ₃	ZnO
%	93.79	0.78	0.68	0.07	1.01	1.36	1.53	0.36	0.04	0.03	0.09	0.26	0.01	0.01

Experimental results of the mechanical properties tests for both as - cast and heat-treated Al - Si reinforced RHA and Al₂O₃ hybrid composites are presented in Table 5 and Table 6 respectively. Better mechanical properties were observed for the heat-treated samples as presented in table 6 compared to as - cast samples in table. Therefore, for further investigations only heat-treated composites were considered.

Table 5. Mechanical properties of as – Cast Al – Si reinforced RHA/Al₂O₃ composites.

Sample Number	Ultimate Tensile Stress UTS (KN)	Hardness (HRB)	Wear Rate WR (mm ³ /m)
A1	138.2	66.3	0.4929
A2	149.5	74.6	0.5020
A3	142.5	69.2	0.3755
B1	109.6	65.9	0.7393
B2	132.6	61.1	0.2510
B3	142.5	68.1	0.2503
C1	135.4	61.7	0.3696
C2	146.7	61.4	0.3765
C3	143.9	63.9	0.1251

Table 6. Mechanical Properties of Heat - Treated Al – Si reinforced RHA/Al₂O₃ Composite.

Sample Number	Ultimate Tensile Stress UTS (KN)	Hardness (HRB)	Wear Rate WR (mm ³ /m)
A1	115.80	52.0	0.0376
A2	127.3	59.4	0.1252
A3	139.2	61.4	0.1252
B1	147.5	63.7	0.2465
B2	156.4	68.8	0.2465
B3	170.1	68.0	0.2465
C1	140.5	74.2	0.3565
C2	178.5	74.6	0.3568
C3	197.5	81.4	0.3765

3.2 Regression Models

Regression analysis aims to determine the best global representation of experimental data for applications in design and analysis of complex engineering systems. Regression analysis consists in arriving at a relationship that may exist between two or more variables. (Das *et al*, 2016).

Linear regression models were developed using least square method of regression analysis to predict the mechanical properties (UTS, HRB, and WR) of the heat-treated Al – Si reinforced RHA/Al₂O₃ composite for different values of fabrication process parameters (s and w). The regression equations for each of the response are presented in equations (1 – 3).

For all the models, the coefficient of determination (R^2) is 90% and above, which indicate a very good prediction of the responses. Further, the predicted values of R^2 are in reasonable agreement with the adjusted R^2 except for δ_{ut} (86.8%). Therefore, it can be concluded that the models are adequate for representing the process and fit the sample data.

In Taguchi optimization, a normal probability plot of the residuals checks if the experimental errors are normally distributed. Therefore, the normal probability plots of residuals for different responses (Figure 1 - 3) depict that the residuals lie reasonably close to the normal probability lines and the terms mentioned in the regressing models are significant and adequate as agreed by Das *et al.*, (2016).

$$\text{UTS} = 70.49 + 0.196X_1 + 6.867X_2; R^2 = 90\%; \text{Radj} = 86.8\% \dots\dots\dots (1)$$

$$\text{HRB} = 39.464 + 0.082X_1 + 1.393X_2; R^2 = 94.3\%; \text{Radj} = 92.4\% \dots\dots\dots (2)$$

$$\text{WR} = -0.135 + 0.0012X_1 + 0.010152X_2; R^2 = 92.8\%; \text{Radj} = 90.4\% \dots\dots\dots (3)$$

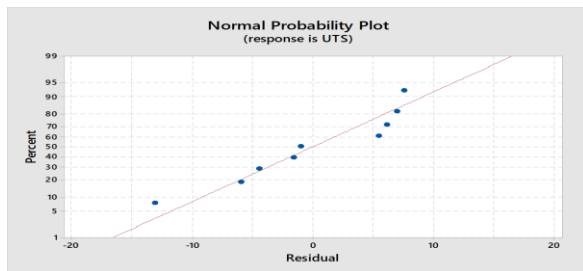


Figure 1a: Normal probability plot of residuals (UTS)

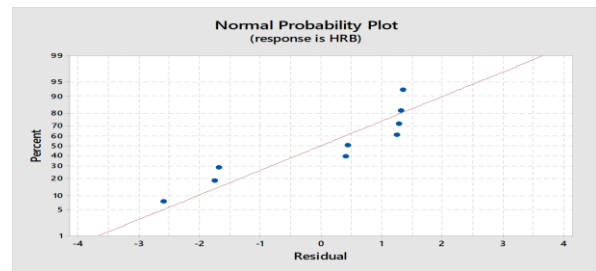


Figure 1b: Normal probability plot of residuals (HRB)

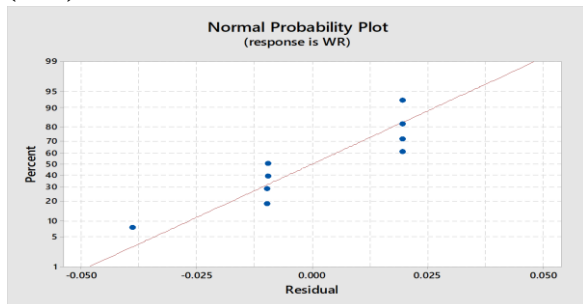


Figure 1c: Normal probability plot of residuals (WR)

3.3 Optimization for multiple performance characteristics

In order to analyze Taguchi L9 OA, the experimental data in Table 6 was converted to S/N ratios using statistical software Minitab 18 (Version 18, Minitab, Ltd. Coventry, UK) in order to measure the robustness of the process. The effect of varying the control factors S and $Wt.\%$ on the multiple responses of δ_{ut} , HRB and WR were analyzed.

As higher values of UTS and HRB are desirable, the Larger – the – better was selected and Smaller – the – better quality characteristics was selected for WR as lower values of WR are desirable. Table 7 presents the Taguchi L9 OA and multi – response results with signal – to – noise (S/N) Ratio.

4.3 Effect of control factors on UTS, HRB and WR.

The dominant control factor was identified from the delta statistics in the response table for the S/N ratios as shown in Tables 8, 9 and 10. The delta statistics were computed based on the difference between the highest and the lowest average value of each factor. Ranks were then assigned according to the delta value. The highest value of delta was assigned the first rank and represents the predominant factor affecting the response. From the response tables, the most dominant factor levels are level 3 for sample size (S) and level 3 for weight percent ($Wt\%$) of UTS and HRB while for WR the dominant factor levels are level 1 for particle size and level 1 for weight

percent (Wt%). This implies that with increase in particle size and increase in volume fraction of RHA, the UTS and HRB increase and better WR at lower values of the particle size and weight percent of RHA as depicted by main effect plots for S/N ratios in figure 2.

Table 7. Taguchi L9 OA and multi – response results with signal to noise (S/N) ratio.

Sample Designation	Control Factors		Response Values			S/N Ratio (db)		
	S (μm)	Wt%	UTS (N/mm ²)	HRB	WR(mm ³ /m)	UTS(N/mm ²)	HRB	WR
A1	125	2.5	115.8	52	0.0376	42.2742	34.3201	28.4962
A2	125	5	127.3	59.4	0.1252	42.0966	35.4757	18.0479
A3	125	7.5	139.2	61.4	0.1252	42.8728	35.7634	18.0479
B1	250	2.5	147.5	63.7	0.2465	83.3758	36.0828	12.1637
B2	250	5	156.4	68.8	0.2465	43.8847	36.7518	12.1637
B3	250	7.5	170.1	68	0.2465	44.6141	36.6502	12.1637
C1	355	2.5	140.5	74.2	0.3565	42.9535	37.4081	8.9588
C2	355	5	178.5	74.6	0.3568	45.0328	37.4548	8.9515
C3	355	7.5	197.5	81.4	0.3765	45.9113	38.2125	8.4847

Tables 8, 9 and 10 show that the particle size S is the most influential factor with delta values of 2.55, 2.51 and 12.732 respectively.

Table 8. Response Table for S/N Ratios of UTS.

Level	S	Wt%
1	42.08	42.53
2	43.96	43.67
3	44.63 ¹	44.47 ¹
Delta	2.55	1.93
Rank	1	2

¹Desired factor levels

Table 9. Response Table for S/N Ratios of HRB

Level	S	Wt%
1	35.19	35.94
2	36.49	36.56
3	37.69 ¹	36.88 ¹
Delta	2.51	0.94
Rank	1	2

¹Desired factor levels

Table 10. Response Table for S/N Ratios of WR

Level	S	Wt%
1	21.53 ¹	16.540 ¹
2	12.164	13.054
3	8.798	12.899
Delta	12.732	3.641
Rank	1	2

¹Desired factor levels

Main effects plots show the significance of each parameter which can be judged by the inclination of the plot. Main effects plots for the mechanical properties are presented below. This shows that particle size of RHA is the dominant parameter in the development of the hybrid composite. In main effect plot the significance of each parameter can be judged by the inclination of the plot. (Das et al,2014)

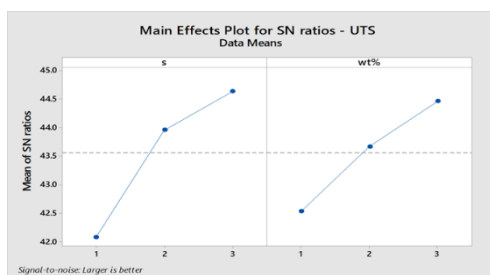


Figure 2a: Main effects plots for S/N ratios of UTS

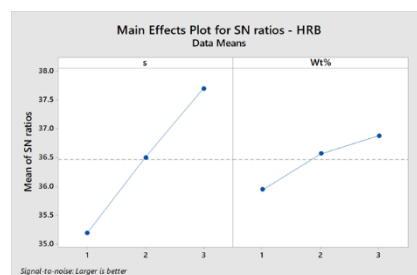


Figure 2b: Main effects plots for S/N ratios of HRB

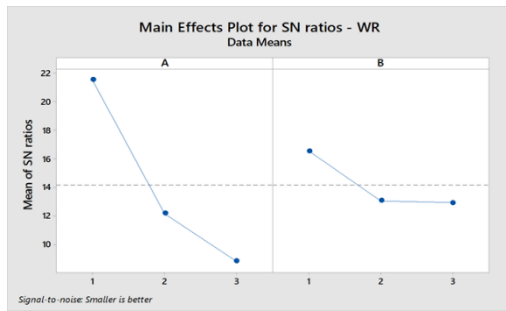


Figure 2c: Main effects plots for S/N ratios of WR

for HRB of the variability can be explained by the model and hence affirms the validity of the model. (Sylajakumari *et al*, 2018).

Similarly, for the WR the most influential parameter is the particle size of the RHA. In this case the contribution percentage is less compared to that of UTS and HRB. This is in agreement with the P value and the R^2 value.

Table 11. ANOVA for S/N Ratio of UTS

Source	DF	SS	MS	F	P	Contribution (%)	Remarks
S	2	10.487	5.2436	20.71	0.008	61.13	Significant
Wt%	2	5.655	2.8275	11.17	0.023	32.97	Significant
Error	4	1.013	0.2531			5.9	
Total	8	17.155		S = 0.5031	$R^2 = 94.10\%$	Radj= 88.19%	

Table 12. ANOVA for S/N Ratio of HRB

Source	DF	SS	MS	F	P	Contribution (%)	Remarks
S	2	9.4218	4.7109	40.42	0.002	83.7	Significant
Wt%	2	1.3686	0.6843	5.87	0.065	12.16	Not Significant
Error	4	0.4662	0.1166			4.14	
Total	8	11.2566		S = 0.3414	$R^2 = 95.86\%$	Radj= 91.72%	

Table 13. ANOVA for S/N Ratio of WR

Source	DF	SS	MS	F	P	Contribution (%)	Remarks
S	2	261.18	130.59	11	0.024	78.17	Significant
Wt.%	2	25.43	12.71	1.07	0.424	7.61	Not Significant
Error	4	47.5	11.87			14.22	
Total	8	334.1		S = 3.4460	$R^2 = 85.78\%$	Radj = 71.57%	

IV. CONCLUSION

This work optimizes the effect of volume fraction of RHA and Wt.% on the mechanical properties and microstructure of an Al – Si hybrid composite produced via stir casting technique using Taguchi optimization. Regression models were developed using least square technique to determine the adequacy of the data. For all the models, the coefficient of determination R^2 was above 90% and above which indicates a good prediction of the responses. An L_9 orthogonal array was developed to optimize the process parameters using the conceptual S/N ratio approach. The optimum casting parameters to obtain good ultimate tensile strength and hardness was at 355 μ m particle size and 7.5wt.% of RHA while for good wear rate at 250 μ m particle size and 2.5wt.% of RHA. Results of analysis of variance (ANOVA) showed that particle size of the RHA had the highest contribution of 61.13% for ultimate tensile strength (UTS), 83.70% for hardness (HB) and 78.17% for the wear rate (WR).

AUTHOR(S) CONTRIBUTIONS

Mangai M. Morgan conceptualized the study, designed the methodology, conducted the initial analysis, wrote the first draft and partook in review/editing of the final manuscript. Ede E. Godwin supervised the experimental process, wrote the final draft of the manuscript and partook in the review/editing of the final manuscript. Nicodemus Datau partook in the final draft and headed the review and editing of the final manuscript.

AI DISCLOSURE

No AI tools were utilized at any stage of the research work

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DECLARATION OF CONFLICT OF INTEREST

Authors have no conflict of interest to declare

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