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Evaluation of Vickers Micro Hardness of Solution Heat Treated (SHT) Al6061/2%Gr/0-6%Al_2O_3 Composites for Piston Applications

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ABSTRACT

This research seeks to evaluate the Vickers micro hardness of Solution Heat Treated (SHT) Al6061/2%Gr/0-6% Al_2O_3 composites, and delve into their prospect for piston applications. Al_2O_3 was used as reinforcement in Al6061/Gr composites to improve mechanical properties. Solution heat treatment was carried out at different temperatures (400°C, 500°C, 600°C) and durations (1, 1.5, 2 hours), followed by ageing at varying temperatures (150°C, 175°C, 200°C) and durations (2, 4, 6 hours). The factors and optimal levels were identified using Minitab Results. The Taguchi L9 Orthogonal Array in Minitab was run using the factors and levels. The hardness properties were evaluated through Vickers micro hardness tester. After completing the experiments, the measured values for each trial of the hardness were entered into Minitab. Then the S/N ratios were analyzed in Minitab via Analyze Taguchi Design sub-menu and selecting the Signal-to-Noise Ratio (S/N) response option for the desired outcome of Vickers hardness. The S/N ratio type was set to "Larger-the-Better" for hardness. The Minitab was used to output the mean S/N ratios for each level of each factor and the highest S/N ratio for each factor, which represents the optimal level for that factor, was identified and interpreted. The results reveal significant improvements in hardness with higher Al_2O_3 content, and thus offer insights into their suitability for high-stress applications, such as pistons.

Keywords: Al6061/ Gr/Al_2O_3, Composites, SHT, hardness, automobile.

INTRODUCTION

The need for high-performance materials in automotive and aerospace industries entails continuous improvement of alloy composites for critical components such as pistons (Miladinovic *et al.,* 2022 and Paul *et al.,* 2022). Materials for making piston include but not limited to cast iron, steel and aluminum alloys (Aliemeke and Oladeinde, 2020). The weight of cast iron will hinder high speed piston and result to more fuel consumption and lead to increase in emissions (Aniekan, 2020). As a result, relevant researchers have developed different alternative methods of minimizing vehicle weight using lightweight materials (Umar *et al*., 2019; Deshmukh *et al*., 2023 in press; and Kosa and Goksenli, 2023). Aluminum alloys, especially Al6061, are generally used in these applications due to their valuable blend of strength, lightweight, and corrosion resistance but are may fail

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under very high thermal conditions (Salonitis *et al*., 2021, Chelladurai *et al*., 2021 and Kareem *et al*., 2021). Hence the addition of reinforcements such as graphite (Gr) and alumina (Al_2O_3) to further enhance the mechanical properties, mostly under high thermal and stress conditions (Chelladurai *et al*., 2021). Furthermore, age hardening as a heat treatment method can be employed on heat treatable aluminum alloys in order to increase some the mechanical characteristics (Hussein *et al*., 2020 and Ashwath *et al*., 2018). Hence varying compositions produce different properties.

This study focuses on examining the effect of varying amounts of Al_2O_3 (0%, 2%, 4%, 6%) on the hardness of Al6061/2%Gr composites, processed through solution heat treatment (SHT). The research inspects how different SHT conditions (temperature and duration) and ageing treatments affect the mechanical performance of these composites, providing valuable awareness into their prospective usage in pistons. Since heat treatment still remains the major way of improving the properties of aluminum alloy, the use of Taguchi approach in optimizing the hardness properties of this alloy was proposed in this study.

OPTIMIZATION OF SOLUTION HEAT TREATMENT PARAMETERS USING TAGUCHI METHOD

Sathish and Karthick (2020) reported that the Taguchi approach is a controlling tool for designing a process based on the OA-Orthogonal Array.

Orthogonal array

Taguchi employs an orthogonal array from the design of experiment theory when a smaller number of experiments are being used to investigate large number of variables (Sathish and Karthick, 2020). The conclusions reached in the small number of experiments turn out to be effective over the whole span of the experiment carried out via establishing the control factors (Umar ***et al***., 2019). Hence, the use of orthogonal array in Taguchi method helps to make the research easier and their resultant linear graphs to fit specific conditions of the study (Sathish and Karthick, 2020). In this research, the orthogonal arrays used was a L9 (3 4) where 9 represent the number of experiments, 3 is used for the number of levels, while 4 represent the number of factors used.

Furthermore the degree of freedom is usually computed in order to select an orthogonal array that will be used in the experiment. The degree of freedom is known as the number of comparisons between the process parameters which help to determine the better level and specify how much better it will be (Umar ***et al***., 2019).

Ratios Analysis

The use of a signal to noise (S/N) ratio distinguishes Taguchi from other optimal processes, as it tend to maximize the mean (signal) and as well minimize the process variations (noise) (Ashwath ***et al***., 2018, Umar ***et al***., 2019 and Sathish and Karthick, 2020). The S/N ratio of each factor is usually computed to determine the effect of each factor on the responses. The signals show that the sensitivity of the experiment output to the noise factors were measured from its effect on the average responses and the noise.

The required S/N ratio depends on the objective of analyzing repeated results (Sathish and Karthick, 2020). The S/N ratios are summarized below:

$$\frac{S}{N} =- 10 \log \frac{1}{n} \sum_{i=1}^{n} (y_n - \bar{y})^2$$

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……………...For nominal is better (1)

For minimization objective function,

$$\frac{S}{N} = -10 \log \frac{1}{n}\left(y_1^2 + y_2^2 + \dots + y_n^2\right)$$ ……

…………...For smaller is better (2)

For maximization objective function,

$$\frac{S}{N} = -10 \log \frac{1}{n}\left(\frac{1}{y_1^2} + \frac{1}{y_2^2} + \dots + \frac{1}{y_n^2}\right)$$

…………………For larger is better (3)

Where:

y = Observed data

$\bar{y}$ = Average of the observed data

n = Number of replications

Analysis of Variance (ANOVA)

ANOVA could be used to minimize the number of experiments and also efficiently improve the processes (Sathish and Karthick, 2020). ANOVA is commonly adopted in investigating the interactions between the statistics obtained from experiments conducted (Umar ***et al***., 2019). It facilitates the process of analyzing the difference between two or more sample means. The ANOVA is achieved by subdividing the total sum of squares. One way ANOVA is the simplest case. Basically, ANOVA is analogous to regression as it is employed in evaluating and showing the association prevailing between a response variable and the independent variables (Ashwath ***et al***., 2018, and Umar ***et al***., 2019). In this investigation, the researchers intend to consider the response value of hardness and the heat treatment parameters at Solution heating temperature (SHT), Solution heating duration (SHD), aging temperature (AT) and aging duration (AD) and wt. % reinforcements (graphite and Al_2O_3). This study will consider the design of experiments of L9 orthogonal array.

Prediction of Optimal Value

From the S/N analysis and mean response characteristics, the optimum levels of the control factors can be computed (Umar, 2019). Therefore, the predicted mean of quality characteristics was evaluated using Equation 4.

$$\eta_{opt} = \eta_{mean} + (A_1 - \eta_{mean}) + (B_1 - \eta_{mean}) + (C_1 - \eta_{mean}) + (D_1 - \eta_{mean})$$ ……….Equation 4

Where

η_{mean} = Total mean of performance characteristics (in this case, corresponding to all the 27 (9x3) readings.

A_1, B_1, C_1 and D_1 are the average tensile and impact strength, hardness and wear or coefficient of friction values of the materials

$_{opt}$ = Predicted mean of the $Al6061/2\%Gr/0\text{-}6\%Al_2O_3$ tensile and impact strength, hardness and wear or coefficient of friction of the materials at optimum conditions

The optimization can be easily employed for industrial applications with negligible error where the optimal condition and SHT process parameters can be repeatedly used for achieving the desired properties. The optimization searches for a combination of factor levels that can satisfy the conditions of the individual factors and responses.

Validation Experiment

The final step involves a confirmatory test using the optimal conditions of the SHT process parameters for the solution heat treatment. The validated values will then be compared with the predicted values to determine if there was any improvement in terms of responses (Sathish and Karthick, 2020; Singh *et al*., 2023 in Press; Ashwath *et al*., 2018 and Umar *et al*., 2019).

PRODUCTION AND SOLUTION HEAT TREATMENT PROCESS OF Al6061/2%Gr/0-6%Al_2O_3 COMPOSITE

Production of the Al6061/2%Gr/0-6%Al_2O_3 As cast composite

A 2- Step Stir casting technique was employed in the fabrication of the required composite as shown by the Stir casting flow process in Figure 1. In the first step, recycled Aluminum 6061 ingot alloy was melted in a graphite crucible to a temperature of 680°C inside a muffler furnace that was set up. 1 wt. % Magnesium Chloride ($MgCl_2$) was added inside the molten alloy to improve the wettability and act as de-fluxing agent to remove unwanted bubbles and impurities. The melted aluminum 6061 was stirred with a graphite stirrer to form vortex at constant speed of 400 rpm and for 3 minutes. The Gr reinforcement was preheated at 350°C and added to vortex so as to ensure a uniform distribution of particles in sample A (Control specimen) and then 2-6 wt. % Al_2O_3 reinforcement in steps of 2 wt. % was preheated at 350°C and added to vortex so as to ensure a uniform distribution of particles in sample B, C & D (Deshmukh *et al*., 2023 in press and Bhat *et al*., 2021).

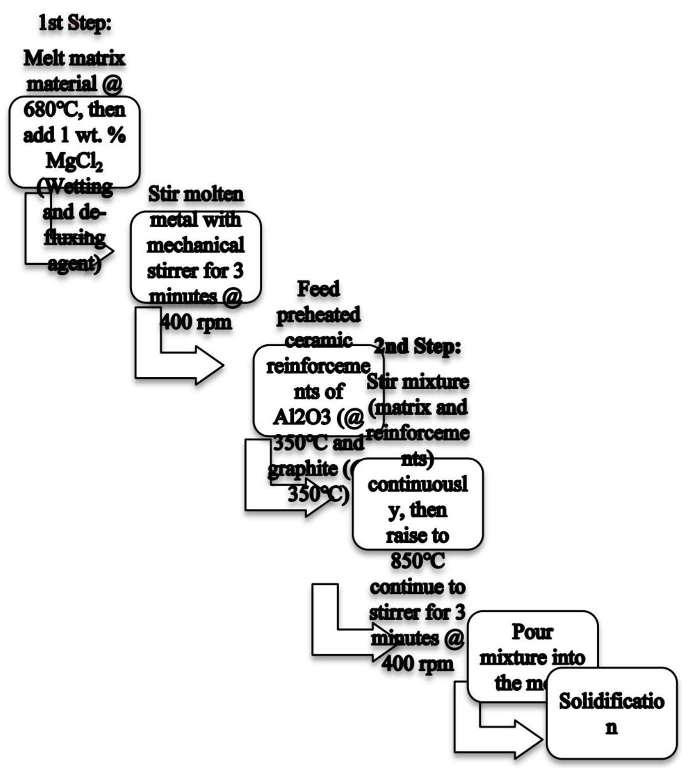

Figure 1: Stir Casting Flow Process.

In the second step, temperature of molten mixture in the muffler furnace was raised to 850°C to superheat and the stirring continued at same speed of 300 rpm and for

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3 minutes. The molten mixture was thereafter be poured into the metallic moulds (A, B, C and D) as presented in Table 1.

Table 1: Percentage Compositions of the Samples produced

S/N	Mould	Composition by Wt.
1	A	Al6061 + 2% Gr (dual matrix as Control)
2	B	Al6061 + 2% Gr + 2% Al_2O_3
3	C	Al6061 + 2% Gr + 4% Al_2O_3
4	D	Al6061 + 2% Gr + 6% Al_2O_3

The castings were then machined to form the test specimens in preparation for the heat treatment process of the Al 6061/graphite/Al_2O_3 Composite.

Heat treatment of the Al6061/2%Gr/0-6%Al_2O_3 composite

Solution heat treatment (SHT) is a key factor adopted in order to increase some of the mechanical characteristics of heat-treatable Al6061/2%Gr/0-6%Al_2O_3 composite, where the solution and ageing treatments specified to these composites were optimized (Hussein *et al*., 2020, Ashwath *et al*., 2018, and Singh *et al*., 2023 in Press). A characteristic heat treatment process for Al6061/2%Gr/0-6%Al_2O_3 composite is the T6-temper condition, which consists of a solution heat treatment, quenching and ageing at an elevated temperature (Hussein *et al*., (2020).

Principally the solution treatment under goes a double stage process, in which the first step involving a single phase supersaturated solid solution is formed by heating the material at a temperature where the phase diagram reveals a maximum solubility, usually at a eutectic temperature, followed by rapid quenching at room temperature (Umar *et al*., 2019). This step is then followed by an ageing procedure consisting in maintaining the sample at a higher temperature generally around 200°C (artificial ageing or T6) where a hardness peak is observed in Figure 2.

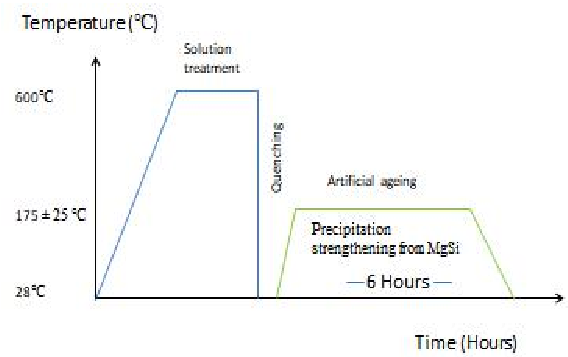

Figure 2: A Graph of temperature against time during ageing heat treatment.

This process produces precipitates evolving from the Guinier Preston (GP) zones to coarse incoherent precipitates (β) when the equilibrium is reached. Usually, the improved properties of the material are due to the presence of fine intermediate (coherent/semi-coherent) precipitates (β^1/β^{11}) which harden the matrix upon a subsequent deformation process (Umar ***et al***., 2019).

Solution of fabricated Al6061/2%Gr/0-6%Al_2O_3 composite was performed by homogenizing the structure and properties. In the heat treatment process the composite was heated at solution heating temperature (SHT) of 400, 500 and 600°C for 1, 1.5 and 2 hours in in an electric muffler furnace. After heating, the composite was quickly quenched in water. Aging of the prepared samples was then carried out in an electric oven at 150, 175 and 200°C for duration of 2, 4 and 6 hours (Singh *et al*., 2023 in Press; Hussein *et al*., 2020; Ashwath *et al*., 2018 and Umar *et al*., 2019).

TAGUCHI EXPERIMENTAL DESIGN OF THE HEAT TREATMENT PROCESS

L9-Taguchi design was employed to optimize the chosen key factors namely: solution heat treatment (SHT) temperature and duration (SHD), and ageing temperature (AT) and duration (AD), in relation to the response which is hardness (Singh *et al*., 2023 in Press; Hussein *et al*., 2020; Ashwath *et al*., 2018 and Umar *et al*., 2019). Table 2 presents the levels of the factors used in the experiment.

Table 2: Levels of Factors used in the Experiment.

Control factor	**Unit**	**Level**		
		1	**2**	**3**
A: Solution heating temperature (SHT)	(°C)	400	500	600
B: Solution heat treatment duration (SHD)	(Hours)	1	1.5	2
C: Ageing temperature(AT)	(°C)	150	175	200
D: Ageing duration (AD)	(Hours)	2	4	6

According to Taguchi design of experiment, 4 factors of 3 levels each enabled the researchers to carry out nine experimental runs as presented in Table 3. In each case, three trial runs were replicated in order to obtain an average value (Sathish and Karthick, 2020; Hussein *et al*., 2020).

Table 3: L9 Taguchi Matrix of the Solution heat treatment

Expt. No.	**Factors**			
	SHT (°C)	**SHD (Hours)**	**AT (°C)**	**AD (Hours)**
1	400	1	150	2
2	400	1.5	175	4
3	400	2	200	6
4	500	1	175	6
5	500	1.5	200	3
6	500	2	150	4
7	600	1	200	4
8	600	1.5	150	6
9	600	2	175	2

Heat Treatment of Al 6061/Graphite/Al_2O_3 Composite

The produced Al6061/graphite/Al_2O_3 composite was solution heat treated at temperatures (400, 500 and 600°C) for solution heating durations of (1, 1.5 and 2 hours), quenched in water at room temperature (°C) and aged treated at temperatures of (150, 175 and 200°C) for ageing durations of (2, 4 and 6 hours) as illustrated in Table 3.

Micro-hardness Measurement of the Heat Treated Al 6061/Graphite/Al_2O_3 Composite

The Micro hardness property of the solution treated Al 6061/Graphite/Al_2O_3 composite was evaluated using an FIE Vickers hardness tester of MV1-PC model in accordance with ASTM E384 and ASTM E92 standards (Umar ***et al***., 2019). The measurements were performed under a load of 3.0*kgf*. Using an L9 orthogonal array design for the four three- level parameters (control factors), a total of 9 runs were performed as presented in Table 4. Each run was replicated for at least three times and the average value was recorded. The signal-to-noise (S/N) ratios were also calculated using the condition been larger is the better.

With the aid of Minitab, the S/N analysis was employed to determine the experimental results.. In S/N analysis, the experimental results tend to be better when the S/N value is greater.

In Tables 4 the highest value of S/N (38.1947) was obtained corresponding to a higher hardness profile, with SHT at 600°C for SHD duration of 2 hours, and aged treated at temperature of 175°C and ageing duration of 2 hours.

Table 4: Taguchi L9 Orthogonal Array, Experimental measured values of average hardness and their SNR for Al6061/2%Gr/0-6%Al_2O_3 composites.

Run	**Control Factors**				**Mean hardness value**	
	A	**B**	**C**	**D**	**Mean (HV)**	**SNR**
1	400	1	150	2	59.9	35.5473
2	400	1.5	175	4	73.97	37.3799
3	400	2	200	6	62.77	34.9546
4	500	1	175	6	65.20	36.2850
5	500	1.5	200	3	57.57	35.2034
6	500	2	150	4	71.87	37.1306
7	600	1	200	4	52.27	34.3645
8	600	1.5	150	6	44.90	33.0449
9	600	2	175	2	81.23	**38.1947**

In S/N analysis, the experimental results tend to be better when the S/N value is greater. In Tables 4 the highest value of S/N (38.1947) was obtained corresponding to a higher hardness profile, with SHT at 600°C for SHD duration of 2 hours, and aged treated at temperature of 175°C and ageing duration of 2 hours.

Established on this experimental process, it can be inferred that the S/N ratio and the hardness profile of this composite under the ageing conditions are directly proportional. Furthermore, it is also expected that at higher ageing temperature, a lower ageing time is expected to precipitate the solute atoms. This is in line with the work of Umar *et al*. (2019) and Singh *et al*. (2023 in Press). In other to maximize the robustness of the process and to calculate the Delta or "main effect" of each parameter, the S/N ratio was employed, which had some advantages of measuring the relative quality and are independent of the mean value. More so, it reflects the variability in the response of a system caused by noise factors and does not introduce unnecessary complications (Umar *et al*., 2019 and Singh *et al*. (2023 in Press).

It was possible to separate the effect of each control factor at different levels by averaging the responses or their S/N ratios at each level, as Taguchi experimental design is orthogonal (Umar *et al*., 2019 and Singh *et al*. (2023 in Press). The mean S/N ratio for hardness, using the average control factor ratio for hardness (ACRH) at levels 1-3 of control factor A (SHT), was calculated

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by averaging the S/N ratios for the runs 1-3, 4-6 and 7-9 respectively. The summarized S/N ratio mean results for hardness in ACRH and the calculated main effect i.e. difference between the maximum and minimum value. Using the larger the better criterion, the Taguchi analysis of average Vickers micro hardness (VH) against the solution heat treatment parameters of SHT, SHD, AT and AD was presented in the response table for signal to noise ratios and the response table of means in Table 5 and 6 respectively. Tables 5 and 6 reveal the optimal combination factors was obtained in the following level of each factor: A_1, B_3, C_2 and D_1, indicating that the optimal value of the Vickers micro hardness was obtained from the 1st level of SHT factor at 400°C, 3rd level of SHD factor for duration of 2 hours, 2nd level AT factor at 175°C and 1st level of AD factor for duration of 2 hours.

Table 5: Vickers hardness Response Table for Signal to Noise Ratios (Larger is better)

Level	SHT	SHD	AT	AD
1	**36.29**	35.40	35.24	**36.32**
2	36.21	35.21	**37.29**	36.29
3	35.20	**37.09**	35.17	35.09
Delta	1.09	1.88	2.11	1.22
Rank	4	2	1	3

Table 6: Vickers hardness Response Table for Means

Level	SHT	SHD	AT	AD
1	**65.54**	59.12	58.89	**66.23**
2	64.88	58.81	**73.47**	66.03
3	59.47	**71.96**	57.53	57.62
Delta	6.08	13.14	15.93	8.61
Rank	4	2	1	3

Also in Table 5, the main effects of SNR response (dB) table for Vickers hardness in ACRH suggests that the control factors of the solution heat treatment parameters of $Al6061/Gr/Al_2O_3$ of AT ranked first with 2.11 dB, followed by SHD with 1.88 dB, then by AD with 1.22 dB and lastly SHT factor with 1.09 dB. This can be concluded that the ageing temperature AT factor is significant. Similarly the main effects from the response table for means in Table 7 reveal that in the mean of means of the solution heat treatment parameters of $Al6061/Gr/Al_2O_3$, composite, the control factor AT was ranked 1st with 15.93 follow by SHT, AD, and then SHT with 13.14, 8.6 and 6.08 dB respectively.

The result of hardness computed in Table 4 illustrate that the largest effect on the hardness properties of the $Al6061/Gr/Al_2O_3$ composite was due to the ageing temperature (AT) factor. On the other hand, solution heat treatment time (AT) has the lowest effect. This may be because at the ageing temperature, precipitation of solute atoms are formed which serve as obstacle to the movement of dislocation, hence higher hardness value obtained. An increase in hardness of the composite will depend on the amount of obstacles that is available to hamper the dislocation movement. The other selected factors have almost the same effects on the hardness properties of this composite. Thus, it can be concluded that the ageing temperature has the largest effect on the maximum hardness properties of the $Al6061/Gr/Al_2O_3$ piston material in the ACRH using the factors in the optimization process. The results obtained from the current analysis agree with other reports (Singh *et al*., 2023 in press, Pederson and Arnberg, 2014 and Umar *et al*., 2019).

Figures 3 and 4 show the effect of control factors on the heat treatment properties. It can be seen that the influence of ageing temperature is very high, while that of SHT time is less important. A fact which can also

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be concluded from the main effect values presented in Table 6.

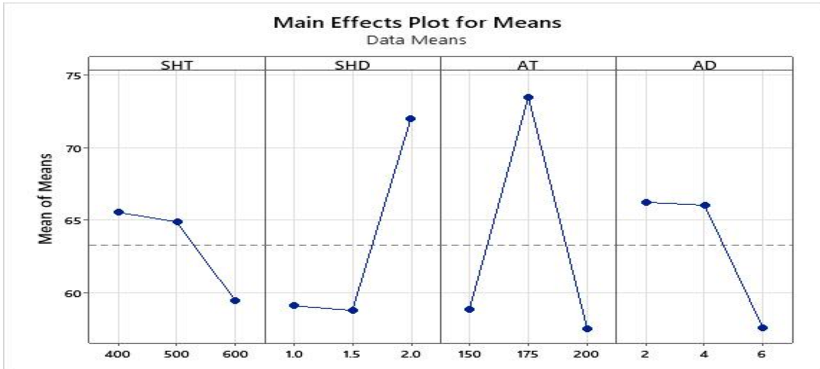

Figure 3: Effect of solution heat treatment parameters on mean response characteristics of Al6061/Gr/Al_2O_3 composite.

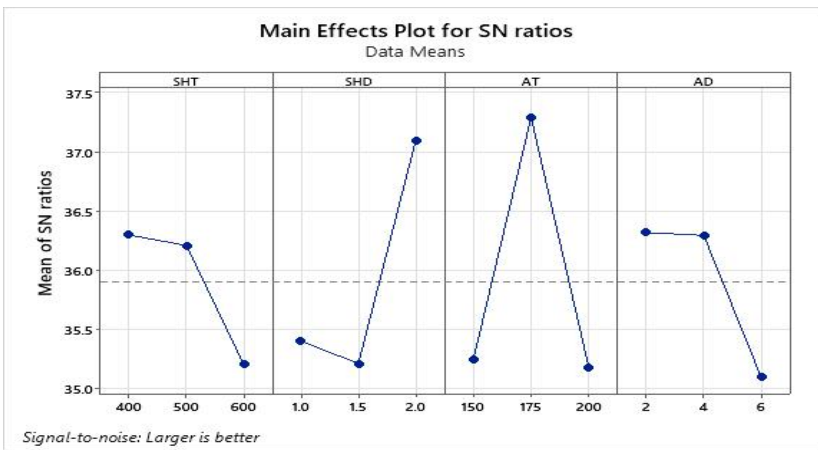

Figure 4: Effect of solution heat treatment parameters on average S/N ratio of $Al6061/Gr/Al_2O_3$ Composite.

At this stage, it is possible to predict the appropriate heat treatment conditions based on the mean and the S/N ratio values of $Al6061/Gr/Al_2O_3$ composite shown in Figures 3 and 4.

Prediction for Optimized Hardness Value $Al6061/Gr/Al_2O_3$ composite

It is now possible to predict the appropriate heat treatment conditions based on the mean of mean and Signal to ratio values of hardness shown in Figures 4 and 5 (Umar *et al*., 2019). Analysis of the the result indicates high hardness values were obtained at levels of A_1 (SHT at 400°C), B_3 (SHD of 2 hours), C_2 (AT of 175°C) and D_1 (AD of 2 hours). To measure the validity of this result, a statistical analysis was established on the developed model and a set of investigation were performed.

Developing Model for Predicting the Optimal Hardness of $Al6061/Gr/Al_2O_3$ composite

Equations 4 and 5 can be used to predict the optimal mean and S/N values for hardness (Umar *et al*., 2019).

$H_{mh} = \bar{h}_{mh} + (A_1 - \bar{h}_{mh}) + (B_1 - \bar{h}_{mh}) + (C_1 - \bar{h}_{mh})$............Equation (4)

$H_{S/Nh} = \bar{h}_{S/Nh} + (A_1 - \bar{h}_{S/Nh}) + (B_1 - \bar{h}_{S/Nh}) + (C_1 - \bar{h}_{S/Nh})$...Equation (5)

It is however be noted that in this study assumptions are made on the basis that there is no interaction between any of the factors A, B, C and D.

Let

H_{mh} = Predicted mean hardness

$H_{S/Nh}$ = Predicted S/N ratios

h_{mh} = Total average of hardness performance characteristics [i.e. corresponding to all the 36 (9x4) readings in Table 4]

$h_{S/Nh}$ = Total average of S/N ratio performance characteristics [i.e. corresponding to all the 36 (9x4) readings in Table 4]

A_1, B_3, C_2 and D_1 = the average values of hardness with process parameters at their respective optimal levels.

Confirmation of the Predicted Hardness Value of $Al6061/Gr/Al_2O_3$ composite

To measure the validity of the analysis, a statistical analysis based on the predicted S/N ratio and a set of experiments were conducted. Equations 6, 7 and 8 were employed to calculate the percentage error, improvement and percentage improvement (Umar *et al*., 2019). Table 7 shows the comparisons between the predicted hardness values and experimental results.

The optimal value was a combination of A_1, B_3, C_2 and D_1. This suggests the optimal Solution heating temperature SHT at 400°C, SHD for a duration of 2 hours, then ageing temperature at 175°C and duration of 2 hours.

The percentage error in mean hardness is 6.96 which is however less than the maximum recommended 10% error reported in literature (Singh ***et al***., 2023 in Press and Umar ***et al***., 2019). The percentage improvement in hardness of 7.48 showed that the hardness of the $Al6061/Gr/Al_2O_3$ composite can be further increased by the use of Taguchi design of experiment.

Table 7: Comparison between the Predicted Hardness Values and Experimental Results Obtained.

Response (HV)	Level	Predictive values		Experimental values		Percentage error		Improvement	
		Mean (VH)	SNR (dB)	Mean (VH)	SNR (dB)	Mean (VH)	SNR (dB)	Value	%
Hardness	A_1,B_3,C_2,D_1	81.2333	38.1947	87.3111	39.2876	6.96	1.0929	6.0778	7.48

CONCLUSION

In this work, Al6061/Gr/Al_2O_3 composite was produced via 2-step stir casting, the solution heat treatment parameters were optimized using Taguchi L9 OA and the Vickers micro hardness were evaluated to obtain the optimal hardness properties. The optimal hardness was calculated using the larger the better quality characteristics. Experimental results have shown that the use of Taguchi design of experiment for optimization have greatly improved the hardness properties of the composite under investigation. Evaluation of Tensile, Impact Strength and Wear Behaviour of Solution Heat Treated (SHT) Al6061/2%Gr/0-6%Al_2O_3 composites for Piston Applications

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