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Characterization and tribological analysis on AA 6061 reinforced with AlN and ZrB$_2$ in situ composites

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ABSTRACT

This research paper deals with AA 6061 composite material with various amounts of weight percentage (wt%) of reinforcements of aluminium nitride (AlN) and zirconium boride (ZrB$_2$). The various amounts of reinforcements like 0 wt%, 3 wt%, 6 wt%, 9 wt% and 12 wt% were mixed with matrix by in situ method of stir casting. Then, the mechanical properties like hardness, tensile and compressive strengths were analyzed as per ASTM standards. The microstructure shows the particle distribution and dispersion within the composites and X-ray diffraction (XRD), energy dispersive spectrograph (EDS) analyzed the constituents of AA 6061 composites. The wear resistance is examined by pin on disc method with various input parameters like composites (wt%), Load (N) and Velocity (m/s). The wear rate ($mm^2/m$) was calculated for each wt% of reinforcements by mass loss ($g$). The SEM examination was conducted before and after the wearing of AA composites to study the consequence worn surfaces and defects. The optimization techniques like Taguchi and ANOVA were then applied to obtain the best processing parameter and most significant parameter of the AA 6061 composites. The theoretical and optimization results were compared for identifying the best results with GA.

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1. Introduction

In the current industrial scenario across the world, aluminium metal matrix composites play an extremely momentous role according their properties, weight and strength ratio. Aluminium is the second most used material which is highly in demand in industries after iron because of their advantageous properties such as light weight, low density, malleability, good thermal conductivity and corrosion resistance [1–4], to name a few. Numerous varieties of ceramic reinforcement particles are used to make AMCs more effective...
and powerful with their properties, some of which are ZrB$_2$, AlN, Si$_3$N$_4$, TiB$_2$, TiN, B$_2$C, TiC, TiO$_2$ and SiO$_2$ [5–13].

The properties of AMCs are improved by manufacturing them in various processes like squeeze casting, stir casting, friction stir processing and powder metallurgy [14–16]. The mechanical properties of AMCs are mainly dependent upon their production methods. The supreme factor among these is mainly the influence of reinforcement particles’ distribution in the matrix material. There are two major categories of production methods by which composite materials are produced, viz. solid state method and liquid state method. Among these two alternatives, the solid state method incurs high production cost and therefore, it is not recommended for large scale production. When compared to the solid state method, the liquid state method incurs lesser production cost, is highly feasible and ensures good dispersion of reinforcements in the matrix material [16–20]. With reference to various literature surveys, it is observed that the stir casting method is a very simple and economical method for producing AMCs with in situ reaction. From the development of aluminium matrix composites, AA 6061 has more satisfying physical properties [21]. Aluminium nitride (AlN) is selected as the reinforcement material taking into regard its superior properties like good thermal expansion coefficient, fair thermal stability, high strength and also its good dispersion with aluminium matrix [22]. Zirconium diboride is also chosen as the reinforcement ceramic due to its favourable properties like electrical stability, high thermal stability as well as good erosion and corrosion resistance [23]. Generally, the tribological tests are taken by pin-on-disc test apparatus. The main control factors of AMCs are wear rate and coefficient of friction. The friction will be created in the specimen and steel or cast iron abrasive discs [24]. Design of experiments (DOE) is used to study and analyze the processing parameters of tribological behaviour of the composites. Taguchi and analysis of variance (ANOVA) methods are used to find the influence of process parameters and percentage of each processing parameters with minimal error [25,26]. Genetic algorithm is the non-traditional technique that is used to find the best and mean values of the output parameters and to predict accurately [27].

In this investigation, Al 6061 composites has been prepared by using aluminium nitride (AlN) and zirconium diboride (ZrB$_2$) reinforcement in various weight percentages (wt%) like 0, 3, 6, 9, and 12. The mechanical behaviour of the AA 6061 composites have been examined by hardness test, tensile test and compression test and the microstructure has also been analyzed, with various wt%. Wear test has been performed by pin-on-disc method with various input parameters. The worn surfaces have been analyzed before and after the wear test of AA 6061 composites by scanning electron microscope (SEM). The optimization techniques like Taguchi, ANOVA and GA have been applied to find out the percentage of each processing parameters as well as the best and mean values of AA 6061 composites.

2. Materials and methodologies

2.1. Sample preparation

In this research, AA 6061 is used as the matrix material and AlN and ZrB$_2$ particles are used as the reinforcement. The composites have been synthesized by using stir casting method. AA 6061 was melted in a preheated furnace at the temperature of 720°C. The elements of AA 6061 are given in Table 1. The reinforcement particles were added with the molten 6061 alloy along with mechanical stirring. The ZrB$_2$ was synthesized by means of two powders viz. KBF$_4$ and K$_2$Z$_2$F$_6$. The same procedure was continued with the reinforcement weight percentages are 3, 6, 9 and 12. According to the William Hume Rothery’s rule, which element can dissolve in the metal, for forming the solid solution. The good solute distribution can be achieved in the solution, if solute atoms are to be less than 15%. The reference of William Hume Rothery’s rule, the reinforcement particles AlN and ZrB$_2$ taken for mixing with AA 6061 matrix less than 15% to achieving good bonding and even distribution in the composites. From the theory reinforcement mixing has less than 15% the one point segregation has been avoided in the matrix material. The mould was already prepared by then, with the dimensions of 10 mm diameter and 20 mm long pins. The well-mixed matrix and reinforcements are then poured in to the mould cavity and cooled down slowly.

2.2. Mechanical properties

The mechanical properties were analyzed with each wt% of AA 6061 composites with ASTM standards. The Micro Vickers’s Hardness machine was used to identify the hardness of AA 6061 composites with various weight percentages as per ASTM E10-07. A total of five samples were involved in this examination, with the indentation made at various places on each sample. The readings were recorded leading to the identification of the average value of the hardness, along with an indentation on the right region of the reinforcement particles.

The tensile and compression strengths were found by universal testing machine with standards of ASTM E08-8 and ASTM E 09-9 respectively. The samples were prepared according to the aforementioned dimensions with each wt% of AA 6061 composites. The samples were well cleaned for removing scratches with the help of SiC 1200 grid paper. The tensile test was done with cross head speed of 2.5 m/min and load of 10 KN. In compression test, the forces were calculated with the deformation of AA 6061 samples, with each wt% of the reinforcements.

| Table 1 – Elements of AA 6061. |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|
| Elements | Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Zn  | Al   |
| Percentage (%) | 0.4–0.8 | 0.7 | 0.15–0.40 | 0.15 | 0.8–1.2 | 0.04–0.35 | 0.25 | remaining |
2.3. Microstructure and SEM examination

In this investigation, optical microscopy was used to analyze the microstructure of AA 6061 composites with various weight percentages of reinforcements. The cold settled samples were prepared for holding the samples in microscope. The cold setting is made by using cold setting liquid and powder. Before setting up this arrangement, the samples were cut down into equal shapes in all the wt% of the composites and the scratches were completely removed from these samples using the dry belt and disc polishing machines. The combination of chemicals namely HNO₃ – 5 ml, HCl – 3 ml, distilled water – 190 ml, HF – 2 ml, popularly known as Keller’s etchant was used to project the surfaces crystal clear. In total, five samples were examined in each wt% of reinforcements with the help of microscope.

The scanning electron microscope (SEM) was utilized for the structural evaluation of AA 6061 composites with various amounts of reinforcements. The worn surfaces were analyzed by SEM examination with various magnifications.

2.4. Wear test

The wear test was taken by means of the Pin-on-Disc test apparatus. The test was taken as per the standards of ATM GG9 GG95a at room temperature. On the whole, 25 samples were examined in this test. The samples were of the dimensions of 10 mm diameter and 25 mm length. The electronic weighing scale was used to find the mass loss of the samples before and after wear. The mass loss was brought from friction between the samples and abrasive disc. The composites (wt%), load (N) and velocity (m/s) would be the input parameters. The input parameters range is given in the Table 2. The sliding distance was fixed at a constant 3000 m.

2.5. Optimization techniques

In this research, Taguchi and analysis of variance are used as the optimization techniques. The input parameters are designed for performing the tests by Taguchi method because it is a very simple, powerful and economical method. The signal-to-noise ratio is normally analyzed under various conditions like ‘smaller is better’, ‘nominal is better’ and ‘larger is better’. In this research, ‘smaller is the better’ condition was selected to achieve the minimum wear rate. The influenced input process parameters also identify with this condition. The L₂₅ orthogonal array was presented in the Table 3.

The ANOVA method is the numerical optimization method used to identify the significant input process parameters here. The MINITAB software was used to derive the design matrix and also to find the most influenced process parameters of the composites. The orders of the influenced parameters are tabulated in ANOVA table, which has influenced the wear rate of AA 6061 composites with various amounts of reinforcements. The mean value of the parameters was also identified with ANOVA technique.

The MATLAB software is used for implementing Genetic Algorithm which is a non-traditional technique with the genetic mechanics. The mechanisms are deployed with the employment of chromosomes with fitness functions. Reproduction, crossover and mutation are the three functional mechanisms. The best and mean values are identified by this technique and it will be compared with the experimental values as well.

3. Results and discussions

3.1. Analysis of mechanical properties

3.1.1. Analysis of hardness test

From the formation of AA 6061 with the various wt% like 0, 3, 6, 9 and 12 of AlN and ZrB₂ by following the in situ method, the Micro Vickers Hardness Test was performed with the constant load of 0.5 kg. The hardness test results were presented with the various amounts of reinforcements in Fig. 1. The addition of reinforcements with matrix, the grains is bonded by the in situ formation increases to oppose the external load nothing but an indentation. It is an evidence of grain refinement. There are no defects that have occurred during the indentation on the composite surfaces, in comparison with unreinforced AA 6061. The reinforced particles are evenly distributed with the matrix material, according to the Orowan strengthening mechanism [28]. The particle movements and dislocations are avoided by the homogeneous particle distribution. The processes of dislocations are obstructed around
Mechanism, ple generally acts unevenly divided into the matrix. The hardness of the AA 6061 composites gradually improves with the addition of reinforcement particles.

3.1.2 Analysis of tensile and compressive strength
The universal testing machine is used to identify the tensile strength of AA 6061 composites with various amounts of reinforcements like 0%, 3%, 6%, 9% and 12%. Each sample is prepared with the specified dimensions and the load acts on its edges equally distributed to the reinforcement particles by the matrix. As a result of the uniform distribution and good bonding between the grains, the composites will get strengthened. According to the Orowan strengthening mechanism, the dislocations are restricted and bow shaped arrest occur among the particles. The bow shaped arrest acts as the barrier of dislocation of grains. If the dislocations are arrested, then the bonding is very high. So, the system strength is increased by increasing the reinforcement wt% of composites. In due course, outside the grains, dislocations cause the continuous gliding. The tensile results are shown in Fig. 2.

From the test results, the compression strength was gradually increased by adding the reinforcements. The test results are shown in Fig. 3. The closeness between the matrix and reinforcements has been achieved by in situ formation. With a good dispersion of ceramic particles, the crushing load is transferred and evenly distributed across the reinforcements. Small cracks are formed. When the stress is evenly distributed through the matrix, the grains are slightly dislocated. The arrangement brings about a slight elasticity to avoid the breakage of shapes of the composites.

According to the William Hume Rothery’s rule, the even reinforcement particles distribution has been achieved in the AA 6061 composites. The SEM analysis has shown the evidence of particle distribution in the matrix material and marked in Fig. 7. When the external load is applying on the composite material, the load transferred and distributed evenly the matrix material through reinforcement particles. From that the higher wt% (12%) has higher particle distribution with good bonding in the AA 6061 composites. So the mechanical properties can be increased with increasing the reinforcement wt%.

3.2 Microstructural evaluation
Fig. 4 illustrates the microstructure of AA 6061 composites with various amounts of the reinforcements, each presented with individual wt% of reinforcements. The grain structures and the bonding between the grains are showed by the microstructure pictures. With the increment of reinforcements, the grain sizes are reduced by the evidence of microstructure, in each wt% reinforcements. The in situ formation confirms the grain refinement in AA 6061 composites. The reinforced particles like aluminium nitride (AlN) and zirconium boride (ZrB2) when dispersed in the molten metal (AA 6061) approaches the growing grains of Al at the time of solidification. The reinforcement particles play the role of grain positioning agents at the time of AA 6061 grains transforming to the solid and while cooling the mixture of matrix and reinforcements, the particles act as the agent of grain nucleation. The microstructure explains that the dispersion of reinforced particles is well mixed with the matrix material and has a good bonding. It is also suggestive that the distribution of reinforcements is intergranular. According to the velocity, before solidification, the particles are submerged in the matrix. Because of the intergranular sites, the particles have a homogeneous distribution, according to which, the loads are evenly distributed by the matrix to the reinforcements. The
From mechanical Fig. there below reinforcements.

3.3. XRD evaluation

From the X-ray diffraction analysis different peaks are identified in the AA 6061 in situ composites in various wt% of the reinforcements in Fig. 5. The XRD confirms the presence of reinforcements in the matrix material. The presence of reinforcements AlN and ZrB$_2$ are evident to successful reaction with AA 6061 matrix. The different peaks of intensity are increased with reinforcements. From this reaction some smaller peaks are identified inter metallic compounds like aluminium oxide. During sintering process oxide may be associated with the aluminium matrix to green compact. The mixture of aluminium and nitriles are having the level of below and each. The intensity of the ZrB$_2$ signals increasing the intensity signals of ZrB$_2$ mass fraction, showing that a feasible restrictions of the mass fraction. In this XRD evaluation there was no evident of any reflections of compounds.

3.4. EDS analysis

Fig. 6 shows the EDS analysis of the AA 6061 composites with various wt% of reinforcements. It is an analysis of qualitative and quantitative analysis of the elements present in the AA 6061 matrix composites. The EDS spectrograph confirms the mixture of matrix and reinforcements. After conducting the wear test EDS was analyzed the highest peak of aluminium is observed. At wearing surfaces some oxidation may be occurred due to heat at time. The reinforcement’s peaks are also identified. From the wearing surface iron is transmitted to mechanically alloying with the reinforcements. The iron layer is to resist the transforms of the atoms during wear, the resistance is also increased. During wear the nitriles are associated with aluminium is evident by the spectrography.

3.5. Analysis of worn surfaces before wear

The worn surfaces are analyzed before and after wear by SEM examination. Fig. 7 represents the images of each wt%
reinforcements of AA 6061 composites. From the SEM images, it is observed that reinforcements and matrix are mixed successfully. From the stir casting method, the reinforcement particles are distributed and dispersed evenly in each wt% of AA 6061 composites. From the SEM images, neither porosity nor cracks have been identified, because of the good refinement of reinforcements. The porosities were not observed because of good thermal expansion coefficient of matrix.

From the morphologies, the wear mechanism can be understood clearly in Fig. 8. From the direction of sliding, some cracks and minor damages are detected. Towards the sliding direction of the wear track, the wear particles formation is continued by formation of ploughing and work hardening with the groove formation. From the morphologies, it is observed that there are some small bubbles and oxide formation on the layer. Due to the high temperature of sliding, the local contacts have been broken. At the same time, the delamination was scattered with oxide layer takes that place. Due to better load withstanding capacity of the composites, there was no evidence of crack formation and wear scar. In the morphology of 0% of composites, there are bad damages due to sliding wear.

From Fig. 6, it is perceived that the wear debris is deposited and some micro fragmentation is also seen on the wear surface. This micro fragmentation causes the better bonding with the layer and grains. At the time of abrasion, the fragmentation has closed the small holes that had formed at the time of casting. The increasing of reinforcements due to good bonding with the matrix and grain refinements has strengthened the composites at the time of applying the loads and induced to wearing action or friction.

4. Investigations on optimization results

4.1 Taguchi optimization technique

In this research, the detailing of the metal matrix had been optimized by the use of L25 orthogonal array (OA). Almost 25 experimental runs with various input constrains had been iterated, till the manufacturing and machine errors became ideal.

AA 6061 was the major part of the material that had been mixed with other impurities for better result by taking 3 factors such as composite weight percentage, velocity and load. Wear rate was mainly kept as the responsive factor. Totally, 25 experimental runs were conducted carrying 5 experimental runs each for the processing factors. Generally, capital value for wear rate must be minimal. On equating, these 25 experimental runs were collectively calculated under the iterated form $-10 \times \log_{10} \left( \text{sum(Y)}^2 / n \right)$. From the observations, it is noted that the increment of the weight percentage results in the decrement of the wear rate. After several iterations, minimum wear rate was recorded as 0.000173 mm$^3$/m produced by 12% weight percentage of AA 2618, measured by 20N load given by the pin-on-disc apparatus and 1 m/s velocity of disc. Optimized results are exhibited in Table 4.

4.2 Signals to noise ratio

Design of experiments gives the physique measurement to find out the authority factors to trim the process by reducing the noise factors. Such factors are reduced by...
Table 4 – Input and output values of 25 experimental runs.

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Composites (wt%)</th>
<th>Load (N)</th>
<th>Velocity (m/s)</th>
<th>Wear rate (mm³/m)</th>
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In both these cases, the composite wt% leads to the metal matrix strength and produces minimum wear rate. Graphical plots for means, signal to noise ratio and wear rate have been articulated in detail in Figs. 9–11. Wear rate graphical plot has been analyzed by keeping data mean value on Y axis whereas the other two graphical plots have been analyzed by keeping wear rate on Y axis.
By the graph plot, the wear rate at the condition of 12% of composite weight, 20N load and 1 m/s velocity of disc. Meanwhile, graph plot of signal to noise ratio tends the above condition which produces the same.

4.3. Analysis of variance

Analysis of variance (ANOVA) is a statistical analysis associated with the variation among groups which create perfect partitions among attributes. Statistical test between mean values are conducted which is used to find out the contribution of process factors that produces better results. Table 7 gives the percentage of inheritance of all the individual parameters. Interaction plot which is shown in Fig. 12 illustrates the detailed graph plot of each factor that contributes to complete a full cycle. Fig. 13 gives the graphical explanation of inheritance percentage.

By the inheritance percentage of process factors, it can be clearly concluded that the wt% of AA 6061 aids to lift the wear rate.

**Fig. 9** – Graph plot of signal to noise ratio – keeping wear rate on Y axis.

**Fig. 10** – Graph plot of means – keeping wear rate on Y axis.
resistance by matrix with other impurities. Almost 86% of contribution was being given by the wt%. To add to it, the other two factors viz. velocity (4%) and load (10%) contribute to the rest of the percentage. Table 8 explains the comparison statements between the experimental investigation and statistical analysis of design of experiments. Variation between the two methods propagates with slight changes. With this, it is concluded that the method of research was fruitful being on the right track and the input parameters of 12 wt% of composite, 20N load and 1 m/s velocity segregates better.
wear resistance in comparison with other cycle runs of the experiment.

4.4. GA for wear rate

Genetic algorithm was a non-traditional algorithm which predicts the input and output parameters to infinity even after the code were installed to train the MATLAB software. The fitness function was derived by various mathematical models shown in Eq. (1). By Eq. (1), this clear that the derivation of mathematical models forms fitness function. The results were optimized by the input parameters given as variables shown in Fig. 14. The observation clears that the minimum wear rate was achieved by the means of 12% composite weight percentage, 20N load and 1 m/s velocity was about 0.000154 MPa and the data mean value have adjusted up to 0.000262 MPa. The input parameters and the fitness function which helps to train the software was shown in Fig. 15. The comparison between the experimental value and software predicted value was shown in Table 9. The slight deviation between the software predicted values and the experimentally obtained values of the process parameters ensure the high standards of GA.

Fitness function for compression strength : \( @ (x) \)

\[ + (0.005224 - (0.000423 \times (1)) - (0.000005 \times x(2))) \]

\[ + (0.000106 \times x(3)). \]

(1)

5. Conclusion

- AA 6061 composites were successfully fabricated by stir casting method with varied amounts of reinforcements like 0, 3, 6, 9 and 12 wt% through in situ formation. From this method, it was ensured that the particle distribution and dispersion was done evenly in the matrix.
- The mechanical properties revealed the strength of AA 6061 composites, the particles dislocated were avoided in the matrix and the load was evenly distributed to the reinforcements from the matrix. The Orowan strengthening mechanism was successfully proved in the mechanical tests. The mechanical properties increase when the corresponding reinforcements are increased.
- From the microscope images, it was perceived that the particles are successfully dispersed and are transforming at the time of cooling. The bonding with the matrix and reinforced particles are good, and therefore the grain refinement is achieved and defects are avoided. The XRD and EDS evaluation reveals the conformation of fillers and constituents of AA 6061 composites.

- The before and after SEM examination has presented the reinforcement particles inside the composites and are observed to be dispersed evenly. There was no evidence of segregation of reinforcement particles in particular areas of the matrix. The worn surfaces of after wear along the sliding direction were clearly observed and some defects are noticed in SEM images. At the time of friction, where the wear debris made a contact with the sample surfaces, ploughing and micro cracks were observed.
- \( L_{25} \) orthogonal array was used to shuffle the parameters. The highest reinforcement wt% has the lowest wear rate. From the optimization results of Wear rate \((\text{mm}^3/\text{m})\) and the ranking from signal to noise ratio, the composites (wt%)
was the of the first rank to influence the wear. Velocity (m/s) influences the second most and load (N) acts as the third rank of wear. From the ANOVA results, the maximum percentages of contribution are of composites (wt%) with 86%, and Velocity (m/s) with 10% and load (N) 4%.

- The comparison of Taguchi and experimental results elaborates that the composite 12 wt% of reinforcement has the lower wear rate 0.000173 mm/m and experimentally it is 0.0003 mm/m.
- The GA and experimental values are compared. The best and mean wear rate was identified. The GA result was 0.000154 mm/m and the experimental value was 0.000173 mm/m. The comparison of GA and experimental is very closer.

Conflicts of interest

The authors declare no conflicts of interest.

REFERENCES


Fig. 15 – The input parameters of minimum wear rate obtained by GA.

Table 9 – Comparison of experimental and software approached wear rate.

<table>
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<th>Methods</th>
<th>Composites (wt%)</th>
<th>Load (N)</th>
<th>Velocity (m/s)</th>
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