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MICROSTRUCTURE STUDIES AND MECHANICAL CHARACTERISATION OF T6 HEAT TREATED ALUMINIUM AND COPPER BASED ALLOY REINFORCED WITH ZIRCON AND GRAPHITE COMPOSITE

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Abstract

The global requirement of high performance, superiority, sustainability, long life, light in weight, low-cost material are the reasons to shift in research from conventional materials to composite materials. Aluminium metal matrix composites are widely used as structural materials in aerospace, sports and automobile applications due to their excellent characteristics such as low cost, high stiffness, low thermal expansion coefficient, high strength to weight ratio, improved resistance to wear, etc. The present research work is focussed on microstructure studies and mechanical properties of composites made of aluminium and copper alloy matrix reinforced with zircon sand by varying weight percentages from 2% to 8% with an increment of 2% by using the stir-casting method. Also studied the effect of adding 2% by weight of graphite for all the above composites. To know the distribution of particles and bonding between reinforcement and matrix, the microstructure of the composites and base alloy were studied using optical and scanning electron microscopy. Hardness and tensile tests were conducted and mechanical properties such as microhardness, ultimate tensile strength, yield strength and percentage of elongation were evaluated for all samples. To enhance the mechanical properties of the composites, the above tests were carried out with heat treatment for all the samples and the results were compared with cast composites.

Keywords: Microhardness, Scanning electron microscope, Stir casting, Weight fraction.

1. Introduction

In the industrialised world, Aluminium Metal Matrix Composites (AMMCs) are the proficient materials widely used in automobiles, aerospace, marine, etc., due to its admirable mechanical properties [1-3]. Different shape of reinforcements such as particulates, fibres and whiskers combined with Al alloys to produce AMMCs. Mcdanels [4] explained that easy accessibility, low cost and autonomy of mechanical properties from particulate orientation, etc., are the reasons for the wide usage of particulate composites.

Different particulate reinforced with Aluminium composites have been tried out using different particulate reinforcements such as Graphite, Silicon Carbide (SiC), Titanium Carbide (TiC), Tungsten (W), Boron (B), Alumina (Al_2O_3), Fly ash, Zirconium (Zr), Titanium Diboride (TiB_2), etc. According to Natarajan et al. [5], due to the effect of reinforcement particles improves the thermal and mechanical properties of AMMCs. The production of particulate reinforced Al alloy composites by using stir-casting method is the most promising method and practised commercially due to its ease and the capability of producing different shapes of castings [6]. The stir casting technique process parameters effects homogeneity and even distribution of reinforcement particles in the metal matrix [7].

High hardness and high modulus of elasticity characteristics possessed by $ZrSiO_4$ made it as a promising reinforcement in Al alloy metal matrix composites. It is observed that the addition of alumina and zircon particles into the Al-4.5 wt% Cu alloy matrix improves the hardness significantly. Further, noticed that the addition smaller size reinforcement particles enhance the hardness of the composite [8]. Akhlaghi and Zare-Bidaki [9] proposed that to reduce friction, wear, thermal expansion and damping capacity self-lubricating composites are prepared with the addition of graphite particulate reinforcement. Heat treatment process strengthens the material system both structurally and physically fit for engineering applications [10, 11].

Addition of fly ash and SiC with Al-4.5 wt% Cu alloy by stir casting route improves the hardness and Ultimate tensile strength of the composite [12]. Mechanical properties of the Al6061 alloy such as tensile strength and hardness improved by addition of zircon and graphite further improved with heat treatment [13]. Study of Aluminium-7075 reinforced with varying percentage graphite and bagasse-ash composites prepared using stir casting technique, Hardness ultimate tensile strength and yielding strength increased gradually with increase in percentage of reinforcements [14]. Singh [15] reported that stir formed Al 6063 with Alumina/Graphite reinforced composites is clearly superior to base Al alloy in the comparison of tensile strength and hardness. It is observed that not much work has been done in characterising the properties of Al/4.5 Cu alloy with zircon and graphite reinforcement until date. Hence, the present research aimed to study the microstructure and evaluate mechanical properties of Al/4.5 Cu alloy reinforced with zircon and graphite by varying weight percentages with and without heat treatment.

2. Material Selection

In this investigation, materials used for the preparation of composite are aluminium and copper powder as base matrix materials, hardener, Zircon ($ZrSiO_4$) with an average particle size of 40 microns and graphite of size 35 microns as reinforcement materials, Hexachloroethane (C_2Cl_6) and scum powder. Table 1 shows the weight percentage of the chemical composition of Al-4.5% Cu matrix alloy.

Table 1. Chemical composition of Al-4.5% Cu.

Element	Weight%	Element	Weight%
Cu	4.51	**Pb**	0.03
Mg	0.061	**Sn**	0.02
Si	0.52	**Ti**	0.012
Fe	0.59	**Zn**	0.12
Mn	0.13	**Al**	Balance
Ni	0.06		

3. Experimental Procedure

The present investigation involves the fabrication of composites by the stir casting technique, which is one of the most effective and simplest methods of manufacturing the composite. Composites fabricated by this method involve melting of the matrix material followed by the addition of reinforcement particles to the melt with simultaneous stirring, followed by casting in a mould.

Aluminium with 98.5% purity is fed into the graphite crucible and heated in an electrical resistance furnace. Aluminium starts melting when furnace temperature is raised to 750 ^{O}C, at that time 4.5 wt% of Cu was introduced into the pure molten Al along with hardener (50% Al and 50% Cu). Hexachloroethane tablets were added to remove impurities and gases from the surface of the molten metal at around 750 °C and scum powder was used as a slag removing agent. The mixture was further heated-up to 800 °C to ensure proper melting of the matrix alloy.

Table 2 shows the test samples of the aluminium alloy matrix and composites with different weight percentages of $ZrSiO_4$ and graphite. $ZrSiO_4$ particles of an average size of 40 microns and graphite size of 35 microns were selected and preheated to a temperature of 200 °C in order to provide a fine bond between the alloy matrix and the reinforcement.

Table 2. Composite test samples considered.

Sample no.	Composition of test samples
Z_1	Al -4.5% Cu matrix
Z_2	Al -4.5% Cu + 2% $ZrSiO_4$
Z_3	Al -4.5% Cu + 4% $ZrSiO_4$
Z_4	Al -4.5% Cu + 6% $ZrSiO_4$
Z_5	Al -4.5% Cu + 8% $ZrSiO_4$
Z_6	Al -4.5% Cu + 2% $ZrSiO_4$+ 2% Gr
Z_7	Al -4.5% Cu + 4% $ZrSiO_4$+ 2% Gr
Z_8	Al -4.5% Cu + 6% $ZrSiO_4$+ 2% Gr
Z_9	Al -4.5% Cu + 8% $ZrSiO_4$+ 2% Gr

Initially, four samples (Z_2-Z_5) of casting were obtained by adding $ZrSiO_4$ into the molten matrix alloy with varying percentage of reinforcement from 0% up to 8% in steps 2%. To ensure uniformity of mixing and complete insertion of particles, the mixture was stirred for 3-5 minutes thoroughly by using a mechanical stirrer. The temperature of the furnace was controlled between 760 °C and 780 °C in the final stirring stage and the pouring temperature was controlled at 750 °C. After thorough mixing, slags and oxides were removed and then the mixture was poured into the preheated finger-shaped mould die to produce the required shape of castings. The same procedure was repeated to prepare another set of four cast samples (Z_6-Z_9) with the constant addition of 2% by weight of graphite by varying $ZrSiO_4$ in steps of 2% up to 8%.

In order to improve the grain structure and mechanical properties between the matrix and reinforcement, T6 heat treatment was carried out for all the composite samples including matrix alloy [16-24]. At a temperature of about 530 °C for a period of 2 hours solutionising heat treatment followed by quenching in water to room temperature then artificial ageing treatment at a temperature of 175 °C for a period of 5 hours were carried out using Muffle furnace. The composite samples were metallographically polished and microstructural characterization studies were conducted to examine the distribution of reinforcement throughout the matrix alloy. This is accomplished by using a Nikon Microscope LV150 with Clemex Image Analyser. The prepared composite samples were etched using Keller's reagent to study the microstructure characterization. Figure 1 shows the microstructure of selected samples of heat-treated composite specimens of 2% zircon and 8% zircon with and without the addition of graphite into the matrix alloy.

As per ASTM-E08 standards, tensile tests were conducted on each composition for six trials on round specimens and average values of ultimate tensile strength, yield strength and percentage elongation for all the unheat-treated and heat-treated composite test samples were calculated. Fully automated MTS/C64.106, innovates servo-hydraulic mechanical testing machine was used to carry out the tensile tests. Microhardness tests were carried out at room temperature conditions for all the composites at a load of 0.5 kg for 30 seconds using an automatic digital Innovatest/4303 Rockwell hardness tester. The hardness values were measured at five different places on each test sample and the average value was calculated for both unheat-treated and heat-treated polished composite specimens.

4. Results and Discussion

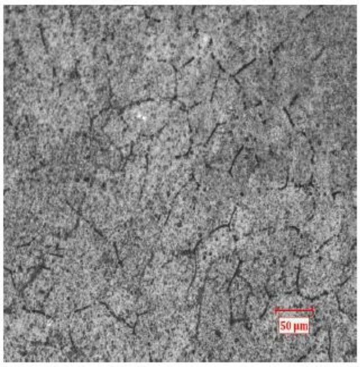
4.1. Surface morphology studies

The micrographs of the specimens shown in Fig. 1 ensures uniform mixing of reinforcement with matrix due to vertex produced in the stirring process, which breaks solid dendrites between particles by friction. The optical micrograph in Figs. 1(a) and (c) show the uniform distribution of zircon particles in the heat-treated composites. The presence of graphite particles can be seen in the form of small clusters in Figs. 1(b) and (d). Due to heat treatment, both zircon and graphite particles established an excellent bond between the matrix and reinforcement results in fine coarsening of grains, decreasing agglomeration, minimal porosity and less formation of cracks in the castings.

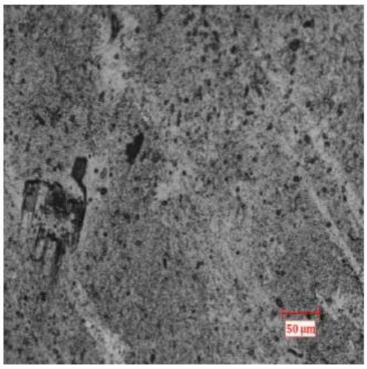
4.2. EDAX analysis

The results of the EDAX analysis of Al-4.5 % Cu +4% $ZrSiO_4$+2% Gr composite specimen sample is as shown in Fig. 2. EDAX spectrum shows the presence of major elements on the surface of the composite. These elements are Aluminium (Al), Copper (Cu), Zircon Sand ($ZrSiO_4$) and Graphite (C).

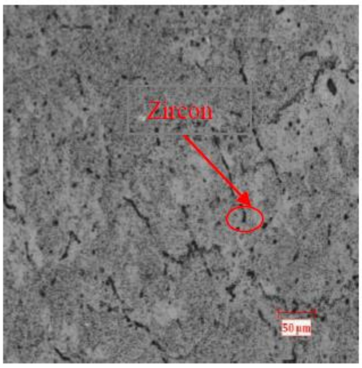
The appearance of a high aluminium peak confirms the presence of Al in the maximum percentage in the composite. The absence of iron indicates that the material's transition did not occur.



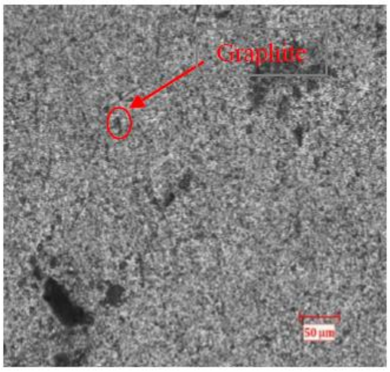
(a) 6% zircon and 0% graphite.



(b) 6% zircon and 2% graphite.



(c) 8% zircon and 0% graphite.



(d) 8% zircon and 2% graphite.

Fig. 1. Optical micrograph images of heat-treated composite specimens.

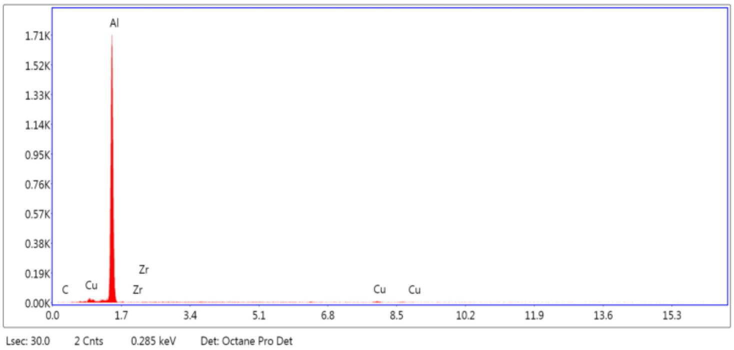


Fig. 2. EDAX of Al -4.5% Cu + 4% $ZrSiO_4$ + 2% Gr composite.

4.3. Hardness test results

The results from the Vickers hardness test for matrix alloy and composites with different weight fractions of zircon and graphite before and after heat treatment are as shown in Fig. 3. It is observed that the hardness of unheat-treated composite increases with the increase of reinforcement of $ZrSiO_4$ to 6% weight fractions of reinforcement. Beyond 6%, the hardness decreases due to increased porosity and brittle nature of the zircon sand.

Based on studies by Saheb [25], although the graphite is soft material, a steep increase in hardness can be observed due to the addition of graphite along with the increase of $ZrSiO_4$ for the unheat-treated composite, since zircon act as a diffusion barrier for *C* diffusion, which decreases the reaction rate between carbon, and aluminium, which leads to the elimination of Al_4C_3 cluster formation.

Also observed from Fig. 3 that, hardness values of the heat-treated composites exhibit high hardness compared to unheat-treated condition, this is due to homogenization of the grain structure and builds bondage between the grains, which enhances the hardness of the composite [17-25]. With the addition of graphite, the hardness of the composite increased further and reaches a maximum value of 139.1 VHN at 6 % zircon and 2% graphite for heat-treated composite. The similar hardness test results observed by different researchers [8, 12-15].

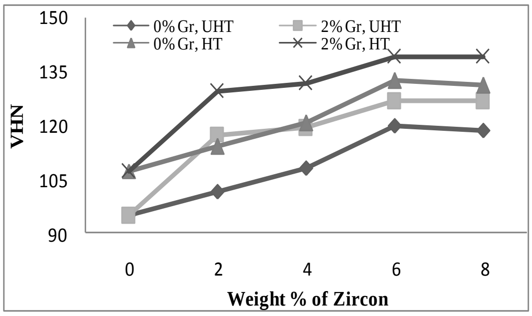


Fig. 3. Effect of weight % of zircon on microhardness for unheat-treated (UHT) and heat-treated (HT) Al-4.5% Cu composite samples.

4.4. Tensile test results

Figures 4 to 6 shows the variation of ultimate tensile strength, yield strength and percentage of elongation respectively for the matrix alloy and composites prior to and after heat treatment. It is observed that, by the increase of $ZrSiO_4$ up to 6%, the ultimate tensile strength and yield strength increases and then decreases in case of both treated and untreated composites by heating. After adding graphite, both ultimate tensile strength and yield strength increases gradually for all the weight fractions of zircon.

The matrix particles are well surrounded, at the interface, the formation of aluminium carbide clusters interface is clean and sharp without any evidence. The carbon solubility decreases in Al by addition of ZrO_4 due to a zircon layer or segregated zircon may act as a diffusion barrier for carbon diffusion, which reduces the reaction rate between carbon and aluminium, which finally removes the formation of Aluminium Carbide (Al_4C_3) [25] clusters. The presence of graphite softens the composite material, which results in increasing ductility, consequently increases the composites fracture load bearing capacity. It is clear from the investigation that, the presence of graphite is resisting dropping UTS and yielding stress even after 6% weight fraction of $ZrSiO_4$ in both treated and untreated composites.

The maximum increase of 46.6 % UTS and 48.9% of yield strength is recorded at 6% zircon and 2% graphite for heat-treated composite. It is observed from Fig. 6 that, there is a rigorous reduction in the percentage of elongation with the increase of reinforcement. There is a steep reduction in the percentage of elongation at the initial stage and then decreases steadily by the increase of weight % of zircon and with the addition of 2% graphite for both heat-treated and unheat-treated composites. The maximum value of reduction in the percentage of elongation is 57.9% at 8% zircon and 2% graphite for the heat-treated composite. The similar tensile test results observed by different researchers [12-15].

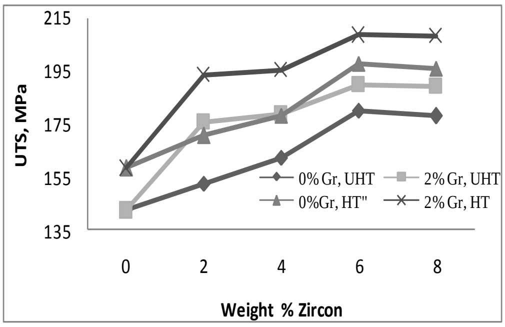


Fig. 4. Effect of weight % of zircon on UTS for unheat-treated (UHT) and heat-treated (HT) Al-4.5% Cu composite samples.

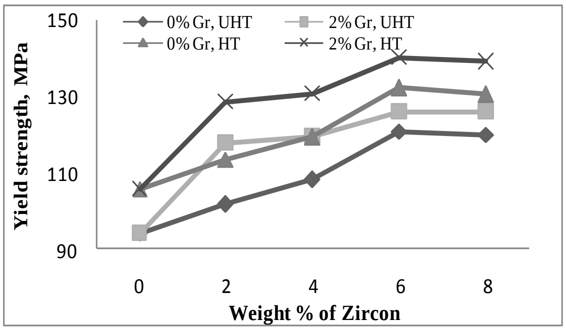


Fig. 5. Effect of weight % of zircon on yield strength for unheat-treated (UHT) and heat-treated (HT) Al-4.5% Cu composite samples.

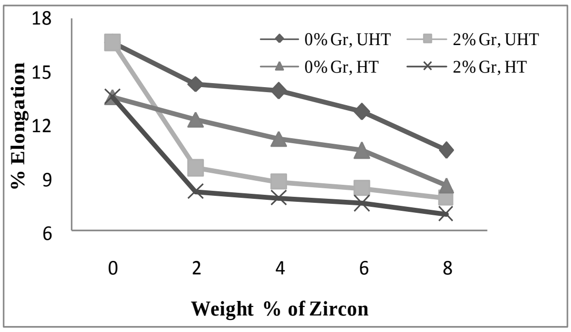


Fig. 6. Effect of weight % of zircon on % elongation for unheat-treated (UHT) and heat-treated (HT) Al-4.5% Cu composite samples.

4.5. Fracture morphology

Figure 7 shows the test specimen SEM images of the matrix alloy Figs. 7(a) and (d), composite with zircon Figs. 7(b) and (e) and hybrid composite Figs. 7(c) and (f) for both unheat-treated and heat-treated samples. The fracture morphology shows evenly distributed large size voids, which lead to ductile fracture. The fractured surface presents relatively flat appearance, which indicates microscopically ductile and macroscopically brittle fracture for the reinforcement of $ZrSiO_4$ particles to matrix alloy Figs. 7(b) and (e). Finer grain size can be observed in the heat-treated composite specimens, which results in reduction of ductility. It is further observed that $ZrSiO_4$ particles remain unbroken in several places on the fracture surface, which shows confirmation of fine bond existing between Al-4.5% Cu matrix and reinforced particles.

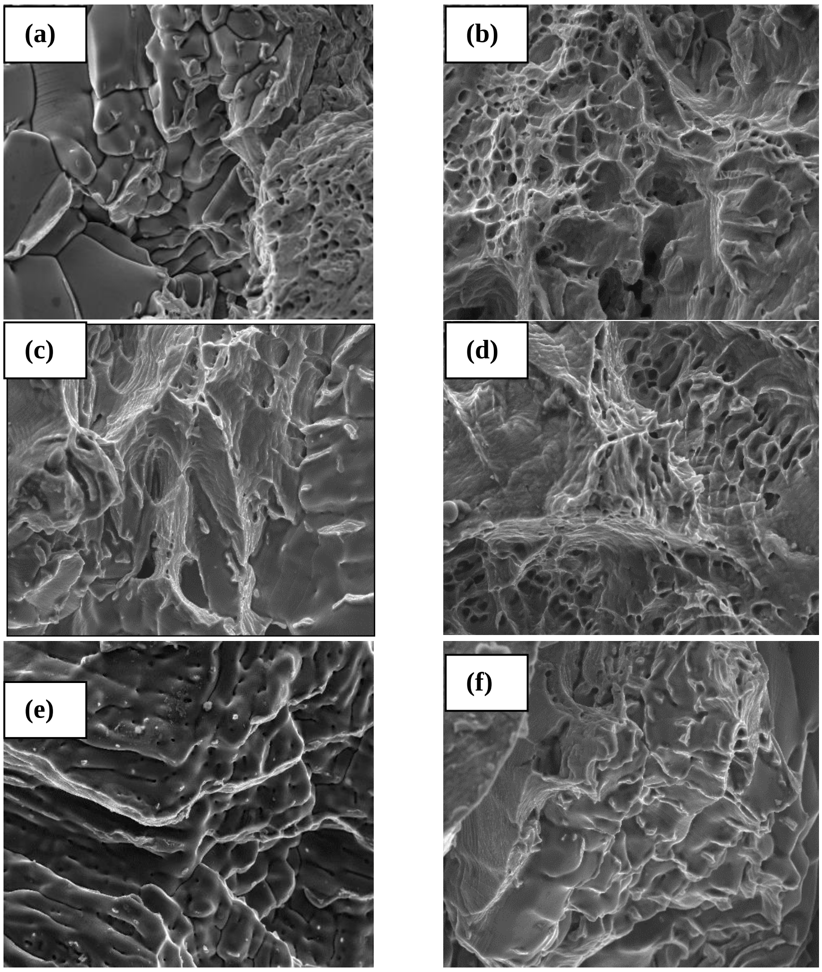


Fig. 7. Fracture morphology of unheat-treated tensile tested samples with: (a) 0% zircon and 0% graphite, (b) 8% zircon and 0% graphite, (c) 8% zircon and 2% graphite and heat-treated tensile tested samples, (d) 0% zircon and 0% graphite, (e) 8% zircon and 0% graphite and (f) 8% zircon and 2% graphite.

5. Conclusions

By using the stir-casting method the Al-4.5% Cu, $ZrSiO_4$ & Gr composites were successfully produced. The microstructure studies, hardness and tensile tests were conducted for all the composites including base matrix (Al-4.5% Cu) and the following conclusions were made:

- The optical, SEM and EDAX studies discovered the existence of $ZrSiO_4$ and graphite in the composite with uniform dispersion.
- The microhardness, ultimate tensile strength and yield strength of the composite is increased with the increase of zircon reinforcement up to 6% and then decreases. With the addition of 2% graphite, the hardness of the composite increased further.
- Percentage of elongation of the composite was decreased with the increase of zircon.
- It is concluded that, 6% zircon and 2% graphite reinforcement with Al-4.5% Cu alloy yields optimal mechanical properties in both unheat-treated and heat-treated condition.

Abbreviations

AMMCs	Aluminium Metal Matrix Composites
ASTM	American Society for Testing and Materials
EDAX	Energy Dispersive Analysis of X Rays
Gr	Graphite
HT	Heat Treated
MPa	Mega Pascal
SEM	Scanning Electron Microscope
UHT	Un Heat Treated
UTS	Ultimate Tensile Strength
VHN	Vickers Hardness Number
$ZrSiO_4$	Zircon

References

1. Hemanth, L. (2009). Quartz (SiO_2P) reinforced chilled metal matrix composite (CMMC) for automobile applications. *Materials and Design*, 30(2), 323-329.
2. Feng, Y.C.; Geng, L.; Zheng, P.Q.; Zheng, Z.Z.; and Wang, G.S. (2008). Fabrication and characteristic of Al-based hybrid composite reinforced with tungsten oxide particle and aluminium borate whisker by squeeze casting. *Materials and Design*, 29(10), 2023-2026.
3. Ramesh, C.S.; Keshavamurthy, R.; Channabasappa, B.H.; and Abrar Ahmed. (2009). Microstructure and mechanical properties of NiP coated Si_3N_4 reinforced Al6061 composites. *Material Science Engineering A*, 502, 99-106.
4. Mcdanels, D.L. (1985). Analysis of stress-strain fracture and ductility behaviour of aluminium matrix composites containing discontinuous silicon carbide reinforcement. *Metallurgical Transactions A*, 16(6), 1105-1115.

5. Natarajan, S.; Vijayarangan; and Rajendran, I. (2006). Wear behaviour of A356/25SiCp aluminium matrix composites sliding against automobile friction material. *Wear*, 261(7-8), 812-22.
6. Singla, M.; Dwivedi, D.D.; Singh, L.; and Chawla, V. (2009). Development of aluminium based silicon carbide particulate metal matrix composite. *Journal of Minerals and Materials Characterization and Engineering*, 8(6), 455-467.
7. Prabu, S.B.; Karunamoorthy, L.; Kathiresan, S.; and Mohan, B. (2006). Influence of stirring speed and stirring time on distribution of particles in cast metal matrix composite. *Journal of Materials Processing Technology*, 171(2), 268-273.
8. Das, S.; Das, S.; and Das, K. (2007). Abrasive wear of zircon sand and alumina reinforced Al-4.5 wt% Cu alloy matrix composites - A comparative study. *Composites Science and Technology*, 67(3-4), 746-751.
9. Akhlaghi, F.; and Zare-Bidaki, A. (2009). Influence of graphite content on the dry sliding and oil impregnated sliding wear behaviour of Al 2024- graphite composites produced by in situ powder metallurgy method. *Wear*, 266(1-2), 37-45.
10. Rajan, T.V.; Sharma, C.P.; and Sharma, A. (2010). Heat treatment principles techniques. *PHI Learning Private Limited*, New Delhi, India.
11. Ramesh D.; Swamy, R.P.; and Chandrasekhar, T.K. (2012). Role of heat treatment on Al6061-Frit particulate composites. *Journal of Minerals and Materials Characterization and Engineering*, 11(4), 353-463.
12. Lokesh, G.N.; Ramachandra, M.; Mahendra, K.V.; and Sreenith, T. (2013). Effect of hardness, tensile and wear behavior of Al-4.5 wt% Cu Alloy/Flyash/Sic Metal matrix composites. *International Journal of Modern Engineering Research*, 3(1), 381-385.
13. Gopi, K.R.; Mohandas, K.N.; Reddappa, H.N; and Ramesh, M.R. (2013). Characterization of as cast and heat treated aluminium 6061/Zircon sand/graphite particulate hybrid composites. *International Journal of Engineering and Advanced Technology*, 2(5), 340-344.
14. Imran, M.; Khan, A.R.A.; Megeri, S.; and Sadik, S. (2016). Study of hardness and tensile strength of Aluminium-7075 percentage varying reinforced with graphite and bagasse-ash composites. *Resource-Efficient Technologies*, 2(2), 81-88.
15. Singh, M. (2016). Studying the mechanical properties of aluminum alloy reinforced with graphite and Al_2O_3 by stir casting process. *International Journal of Advances in Engineering and Scientific Research*, 2(3), 36-51.
16. Das, S.; Das, S.; and Das, K. (2006). Ageing behaviour of Al-4.5 wt. % Cu matrix alloy reinforced with Al_2O_3 and $ZrSiO_4$ particulate varying particle size. *Journal of Material Science*, 41(16), 5402-5406.
17. Abarghouie, S.M.R.M.; and Reihani, S.M.S. (2010). Aging behaviour of a 2024 Al alloy-SiCp composite. *Materials and Design*, 31(5), 2368-2374.
18. Kushwaha, R.K.; Siddiqui, S.A.; and Singh, V. (2014). Characterization Al-4.5% Cu alloy and study of wear resistance. *International Journal on Emerging Technologies*, 4(2), 4 pages.

19. Benal, M.M.; and Shivanand, H.K. (2007). Effects of reinforcement's content and ageing durations on wear characteristics of Al (6061) based hybrid composites. *Wear*, 262(5-6), 759-763.
20. Prashanth, G.D.; Karthick, M.C.; Hasan, M.A; and Amarnath, J.K.C. (2015). Characterization of as cast and heat treated aluminium based hybrid metal matrix composites. *International Journal of Scientific and Engineering Research,* 6(8), 740-745.
21. Shiri, N.D.; Bhat, T.; Bhat, S.; and Kumar, G. (2015). Effect of heat treatment variables on the abrasive wear behavior of Al-6061 alloy and Al 6061-SiC_P composites using taguchi technique. *American Journal of Materials Science*, 5(3C), 151-156.
22. Uvaraja, V.C.; Natarajan, N.; Sivakumar, K.; Jegadheeshwaran, S.; and Sudhakar, S. (2015). Tribological behavior of heat treated Al 7075 aluminium matrix composites. *Indian Journal of Engineering and Material Sciences (IJEMS)*, 22, 51-61.
23. Somashekhar, P.H.; Sharma, K.V.; and Girisha, H.N. (2018). Effect of heat treatment on tensile and hardness properties of aluminium-7075 alloy reinforced with graphite and bagasse-ash composites. *IOSR Journal of Engineering,* 8(8), 38-43.
24. Jones, D.R.H.; and Ashby, M.F. (2006). *Engineering materials-2: An introduction to microstructures, processing and design* (3rd ed.). Oxford, United Kingdom: Butterworth-Heinemann.
25. Saheb, D.A. (2011). Aluminum silicon carbide and aluminum graphite particulate composites. *ARPN Journal of Engineering and Applied Sciences*, 6(10), 41-46.