Nat. Volatiles & Essent. Oils, 2021; 8(5): 1704 - 1713

NVEO
Natural Volatiles & Essential Oils

Experimental Studies and Regression analysis on microstructure and wear loss of MMCs based Zinc-Aluminium alloys with graphite particles reinforcement

O. Gurumurthy[a] and S.Venkateswaran[b]

[a]*Assistant Professor, Department of Mechanical Engineering, BMS Institute of Technology and Management, Bangalore – 560 064*

[b] *Formerly Principal & Head, Department of Mechanical Engineering, BMS Institute of Technology and Management, Bangalore 560064*

Abstract

In recent years, zinc-aluminium (ZA) alloys have found increasing uses in several applications because these alloys exhibit a good combination of mechanical, physical and tribological properties along with low cost of fabrication. In order to further enhance some of the properties of non-ferrous alloys, many attempts have been made to produce MMCs with Al alloys, Mg alloys, Cu alloys and Ti alloys as the matrix materials. Literature on aluminum alloy composites suggests the effective use of graphite as a potent reinforcement. The absence of data on ZA alloys with graphite reinforcement was the stimulus for pursuing this systematic work. In this study, the results of the experimental investigation with varying graphite reinforcements on the microstructure and wear loss of the popular gravity die cast ZA base alloys (viz., ZA8, ZA12 and ZA27) is reported.

The wear tests were conducted using pin-on-disc apparatus to assess the wear loss at three different loads (20N, 29N and 39N) and at constant speed of 200rpm. The results showed that the wear loss decreases with the addition of graphite from 2wt.% to 6wt.% in the ZA8, ZA12 and ZA27 base alloys and it increases with increasing loads. The studies indicate that the wear loss of ZA8 and ZA12 were higher than that of ZA27 based composites.

Design of experiments (DOE) denotes the methodology of application of statistical techniques in experimentation. This has been attempted in this work, for the first time for the ZA alloy based composites. Proceeding further, multiple regression models have been worked out for the UTS, YS, %E, and BHN. Confirmation test results are in good agreement with the values predicted.

Key words: MMC, ZA base alloys, graphite reinforcement, SEM, stir casting, Design of experiments, regression models.

1. Introduction:

Composite materials have attracted tremendous attention in recent years. Out of the three major types of composites viz., MMC (Metal matrix composites), CMC (Ceramic matrix composites) and PMC (Polymer matrix composites). MMCs are the most popular and widely used. Basically, MMCs comprise of a metallic matrix in which particles or whiskers or fibres are disbursed uniformly. Such Metal matrix composites exhibit characteristics that are not possible to be realized in the monolithic constituent materials. The properties that are possible to be improved by such combinations are hardness, wear resistance, coefficient of thermal expansion, stiffness and damping capacity. The development of metal matrix composites, specifically cast metal-matrix particulate composites has been extensively covered in literature. It is apparent that India, in particular, should pay special attention to the possible use of MMCs in energy, housing, and transportation sectors, and then on to high priority domains such as solar photo-voltaic, semi-conductor and super-conductor industries.

Matrix and the reinforcement are the important constituents of MMCs. The properties of the MMCs are controlled to a large extent by the bond between the matrix and the reinforcement. In order to realize optimum properties in the MMCs, a judicious combination of the above two (with compatibility to each other) is essential. Substantial amount of R&D work has been completed on MMCs based on aluminium alloys, magnesium alloys, titanium alloys and copper alloys, but only very few on zinc alloys.

Hard ceramic materials such as silicon carbide and boron carbide have been successfully tried as reinforcing materials. Graphite particles too seem to offer several advantages in addition to cost benefits. Scores of processes are available for fabricating MMCs, but the liquid metal route (Stir casting technique) offers unique advantages for processing low melting metals such as zinc and its alloys. Therefore, an extensive experimental investigation was taken up to produce and characterize zinc-aluminum based MMCs with graphite particles as the reinforcement.

The well-known Japanese scientist Taguchi proposed the simple DOE approach in order to drastically lower the number of trials. Taguchi's concept makes use of Orthogonal Array (OA), and this has become extremely popular in many domains of engineering and science Applying this to ZA alloys has not even been thought of in any investigation till now. Normally, experimental investigations involve extensive trials which call for extended durations, extensive facilities and substantial cost of materials and man power. All these can be appreciably reduced by adopting the design of experiments. Moving further, the findings can lead to the development of Regression Models that accurately predict the properties corresponding to any combination of the variables. A successful attempt has been made on the above as reported here.

2. **Experimental details**:

Commercially available pure zinc (99.8 %) and aluminum (99.90 %) were used to produce three ZA alloys (ZA8, ZA12 and ZA27) test castings. The exact composition of each alloy is given in Table.1. (These are the 3 most popular ZA alloys that have found several end-uses)

Table 1: ZA base alloys investigated and their chemical composition

Alloy	Aluminium, wt. %	Copper, wt. %	Zinc, wt. %
ZA8	8.0	1.10	Balance
ZA12	12.0	1.40	Balance
ZA27	27.0	2.25	Balance

Graphite particles ranging from 40 to 50µm were added as reinforcement to the above ZA alloy to produce composites. The ZA alloys were melted in M/s.Furmaco electric resistance furnace shown in Fig. 1.

The liquid melt (of the three ZA alloys, each at the corresponding 150^0C superheat) was cast in preheated mild steel dies and cooled to room temperature. For dispersing the graphite powder into the melt, molten metal was stirred using a custom made in-house stirrer set-up shown in Fig. 2. The speed of the stirrer (based on the initial trials) was kept at 600 ±20 rpm. This rotational speed resulted in a vortex of adequate depth for absorbing the graphite particles but without undue splashing of the liquid metal.

Fig 1. Melting using electric-resistance Furnace

Fig.2 Custom made in-house stirrer

After solidification, the castings were sectioned to evaluate the microstructure and wear loss. SEM samples of composites were prepared for microstructure analysis are shown in Fig.3. Wear test was carried out using Ducom pin-on-disc tribometer. The wear test samples were prepared as per ASTM G99 standard of 5 mm diameter and 10 mm length as shown in Fig.4.

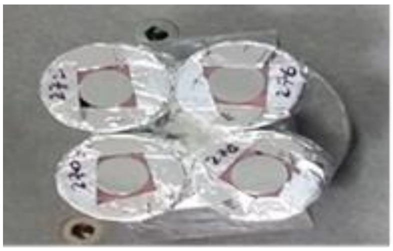


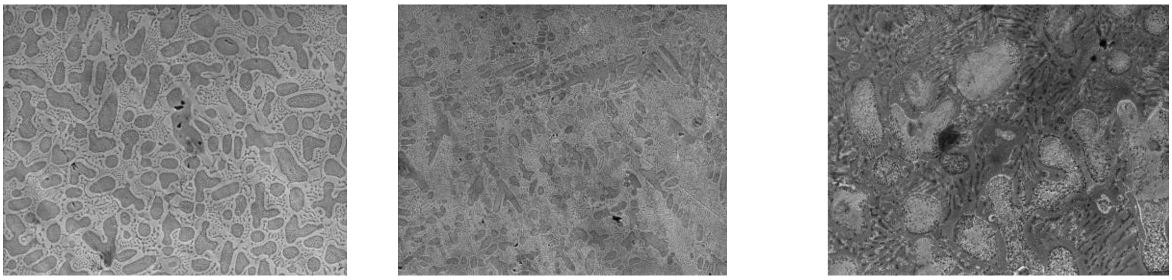
Fig.4 Wear test specimen

3. Results and discussions:

(a) Microstructure studies.

The microstructure of ZA alloys mainly consists of aluminium rich α-phase and zinc rich β-phase; the rich β-phase is higher than that of the α-phase.

SEM microstructural images of ZA8 alloy matrix with 2, 4 and 6 wt. % graphite particles are shown in Figs.5. The microstructural analysis of the ZA8 alloy based composites were carried out using ZEISS computerized scanning High-resolution Electron Microscope FEI-QUANTA 200. Scanning electron microscope (SEM) studies were carried out in the as-polished samples using a large-area backscattered electron detector to produce an image. Images of SEM microprobe analysis are shown in Fig.5. The microstructure of ZA8 alloy exhibited a dendritic structure, wherein primary α-dendrites were surrounded by the eutectic (α+β) and ε-phase respectively. The microstructures show reasonably good solid solubility of graphite in either the aluminium or the zinc-rich phases.

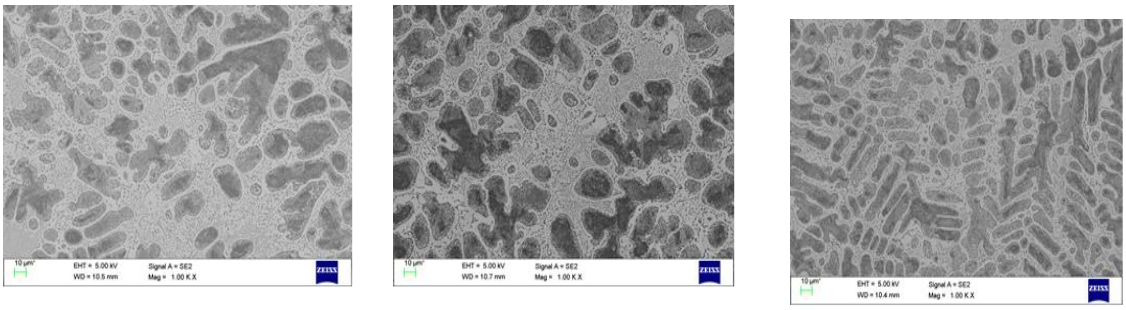


(a). 2wt.%

(b). 4wt.%   (c). 6wt.%

Fig. 5. SEM micrograph of ZA8 alloy reinforced with Gr_p (2/4/6wt.%)

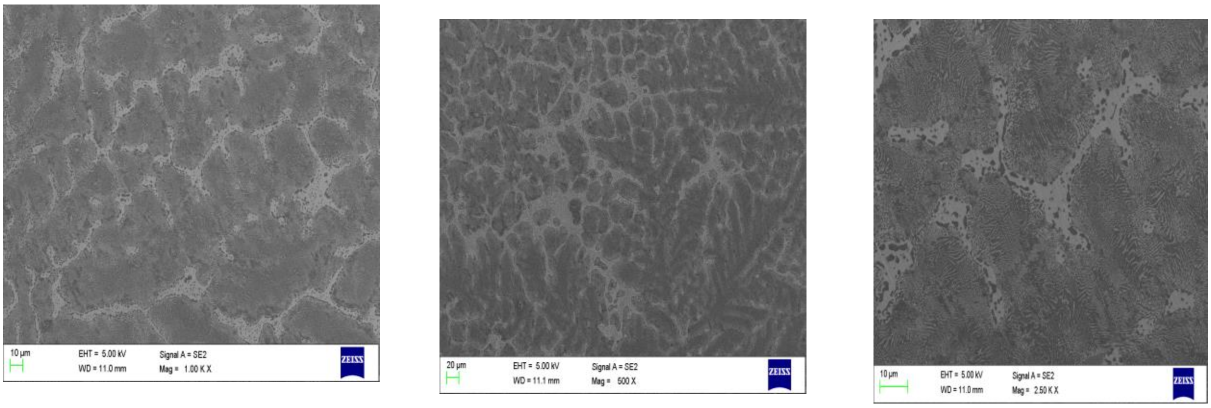
SEM microstructural images of ZA12 alloy matrix with 2, 4 and 6 wt. % graphite particles are shown in Figs.6. The large atomic radii differences between the Al-rich and Zn-rich phases allowed the production of medium-resolution images of good contrast in this alloy which clearly displayed the large scale features of the structure. The microstructures of the composites reveal that both dendritic and eutectic phases are present in the structure. The dendrites are composed of aluminium rich phases (ZA-12), surrounded by a eutectic (containing α and η phases) of varying amount, It can be seen that the graphite particles of varying sizes ranging from 40-50 µm are distributed uniformly throughout. The microstructures show reasonably good solid solubility of graphite in either the aluminium or the zinc-rich phases.



(a). 2wt.%   (b). 4wt.%   (c). 6wt.%

Fig. 6 SEM micrograph of ZA12 alloy reinforced with Gr_p (2/4/6wt.%)

SEM microstructural images of ZA27 alloy matrix with 2, 4 and 6 wt. % graphite particles are shown in Figs.7. From a study of the above micrographs, it can be observed that the addition of reinforcement (graphite) to the molten ZA-27 alloy results in a mixture of α- η rich phase and randomly distributed graphite particles. However, particle segregation occurs during solidification due to their density difference with the melt. Due to the higher aluminium content of the base alloy, there is a greater volume fraction of the dendritic material.



(a). 2wt.% (b). 4wt.% (c). 6wt.%

Fig.7. SEM micrograph of ZA27alloy reinforced with Gr_p (2/4/6wt.%)

(b) Wear loss of ZA base alloy reinforced with graphite particle:

Sliding wear tests were carried out under dry (unlubricated) condition using pin-on-disc apparatus. It can be observed from the Fig.8. that wear loss is high at 2wt.% graphite compared to that of 4wt.% and 6wt.% graphite reinforced ZA8 alloy at a constant speed of 200rpm for different applied loads 20N, 29N and 39N respectively. It can be seen that at a constant speed, the wear loss increases with varying applied loads and the decreasing trend in wear loss is realized as the graphite reinforcement reaches 6 wt.%. Continuous flow of lubricant between two sliding surfaces is very important to achieve excellent tribological properties. This can be easily maintained only when fluid lubricants are used. In order to insure the continuous flow in case of solid lubricant, an excellent innovation is to reinforce graphite as a solid lubricant into ZA base alloys.

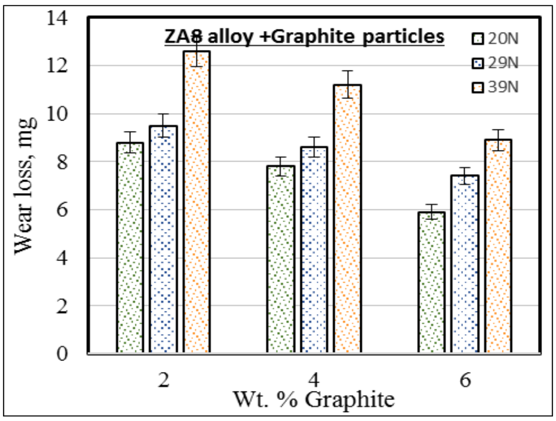


Fig.8: Wear loss vs. ZA8 alloy based composites.

It can be observed from the Fig.9 that wear loss is high at 2wt.% compared to that of 4wt.% graphite and 6wt.% graphite reinforced ZA12 alloy at a constant speed of 200rpm for increasing applied loads 20N, 29N and 39N respectively. It can be seen that at a constant speed, the wear loss increases with varying applied loads and the decreasing trend in wear loss is realized as the graphite reinforcement reaches 6 wt.%.

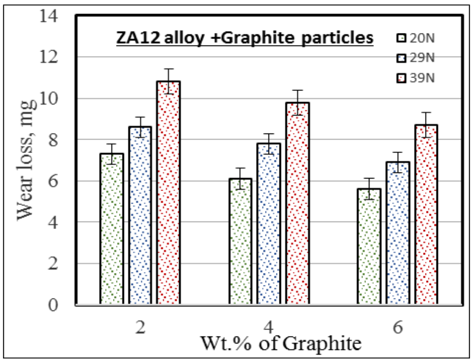


Fig.9: Wear loss vs. ZA12 alloy based composites.

It can be observed from the Fig.10 that wear loss is high at 2wt.% compared to that of 4wt.% and 6wt.% graphite particles reinforced ZA12 alloy at a constant speed of 200rpm for varying applied loads 20N, 29N and 39N respectively. It can be seen that at a constant speed, the wear loss increases with varying applied loads and the decreasing trend in wear loss is realized as the graphite reinforcement reaches 6 wt.%.

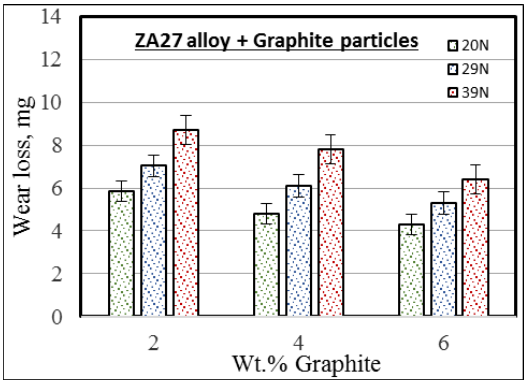


Fig.10: Wear loss vs. ZA27 alloy based composites.

(c) Regression Analysis:

In this section, corresponding to varying additions of graphite particles, the properties of the different ZA alloy based composites are predicted using the Regression models developed. These regression models provide a quick and reliable estimation of the properties of the composites corresponding to any selected combination of base alloy/ amount of addition of reinforcement.

When two or more predictor variables are involved in the trials, multi-linear regression models are the most suitable. A regression model that involves more than one repressor variable is termed as multiple regression model. A multiple linear regression model with two variables can be expressed as:

$y = \beta o + \beta 1x1 + \beta 2x2 + \varepsilon$

y represents is the estimated value of the dependent variable, x1 and x2 are predictor variables.

βo is a constant (called intercept), and β1 & β2 are regression coefficients

In order to verify the suitability of the model, coefficient ***of multiple determinations*** $\boldsymbol{R}^2$ is calculated. Such Regression models were generated for the purpose of accurately predicting wear loss of ZA alloys reinforced with graphite particles.

- Regression model for wear loss of ZA alloys (ZA8, ZA12 and ZA27) with the addition of 2wt. %, 4wt. % and 6wt. % graphite particles.

The relation between the controllable factors and the wear loss was obtained through OA matrix approach. A multiple linear regression model was developed to predict the wear loss.

Wear loss obtained for different combinations of base alloys and reinforcements from the experimental trials were processed using *Minitab 17* software to generate multiple linear regression coefficients. Samples of graphite particle reinforced ZA based composites were tested using the pin-on disc apparatus. Tests were carried out to measure wear loss at a constant speed of 200rpm and at different applied loads 20N, 29N and 39N respectively. The effect of reinforcement content on the wear loss was assessed using the S/N ratio. As the objective of this phase of the investigation was to minimize the wear loss of the composites developed, it is appropriate to consider "smaller-the-better" concept for the S/N ratio in order to arrive at the optimum factor level.

The Equation for calculating the S/N ratio for "smaller-the- better" quality feature is: S/N Ratio = $-10\ \log_{10}(1/n)\sum\left(y_i^2\right)$

- **Typical regression model:**

The experimental results of wear loss due to dry sliding wear behaviour of graphite particle reinforced ZA based composites for factors levels are shown in Table .2.

Table 2. Experimental data-Mean wear loss (mg)

Expt.	x_1: ZA base alloy	x_2: Wt.%Gr.	y: wear loss (mg)		
			Load=20N	Load=29N	Load=39N
1	8	2	8.2	9.5	12.6
2	8	4	7.1	8.6	11.2
3	8	6	5.9	7.4	8.9
4	12	2	7.3	8.6	10.8
5	12	4	6.1	7.8	9.8
6	12	6	5.6	6.9	8.7
7	27	2	5.85	7.05	8.6

8	27	4	4.8	6.1	7.8
9	27	6	4.02	5.3	6.4

Experimental results are computed using Minitab 17 software, and the typically generated regression model for wear loss at constant loads are:

a. Regression Model (Applied load = 20N):
Wear Loss (mg)= 9.756 - 0.10953 X(Al content in the ZA base alloy) - 0.4858X(Wt.%Gr).
R^2 = 0.9749

b. Regression Model (Applied load = 29N):
Wear Loss (mg)= 11.192 - 0.11932X(Al content in the ZA base alloy) - 0.4625X(Wt.%Gr).
R^2 = 0.9877

c. Regression Model (Applied load = 39N):
Wear Loss (mg)= 14.684 - 0.1657x(Al content in the ZA base alloy) - 0.6667x(Wt.%Gr).
R^2 = 0.9528

This shows that all the models are in fairly good fit with experimental data. Results of the confirmations tests on wear are compared with regression models values, and are show in Table.3.

Table 3: Comparison of typical experimental results of wear loss with regression models

Load, N	x_1: ZA base alloy	x_2: Wt.%Gr.	Mean wear loss (mg)	Regression model wear loss	% Error
20	27	6	4.02	3.88	3.48
29	27	6	5.30	5.20	1.97
39	27	6	6.40	6.21	2.97

In multiple regression analysis, the values of coefficient of multiple determination, R^2, values are greater than 0.94 in all the models. The experimental results of weight loss compare well with the weight loss calculated using the regression model.

4. Conclusions:

In ZA alloys based graphite composites, as seen in the microstructure the solidification initiates at the reinforcing particles and the primary dendrites grow around the particles. From a study of the above micrographs, it can be observed that the addition of reinforcement (graphite) to the molten ZA alloys results in a mixture of α- η rich phase and randomly distributed graphite particles. However, particle segregation occurs during solidification due to their density difference with the melt. Due to the higher aluminium content of the base alloy, there is a greater volume fraction of the dendritic material. Results show that the wear loss decreases with the addition of graphite from 2wt.% to 6wt.% in the ZA8, ZA12 and ZA27 base alloys and the wear loss increases with increasing loads. Wear and friction are found to be greatly influenced in graphite reinforced ZA base alloys; the solid graphite particle which consists of carbon atoms arranged in a layer-like structure, displays a very low coefficient of friction while sliding on another clean surface, thus suggesting that it can be used as a solid lubricant.

Multiple regression models were worked out for wear loss of all three ZA alloys. The values of the coefficient multiple determination R^2 are quite high, and in most of the cases it is more than 0.94, indicating that the fit between the experimental and predicted data is very good.

5. REFERENCES

[1]. J. Hashim, L. Looney, M.S.J. Hashmi, Particle distribution in cast metal matrix composites-Part I, Journal of Materials Processing Technology, 123 (2002), pp. 251–257.

[2]. T. J. A. Doel and P. Bowen, Tensile properties of particulate-reinforced metal matrix composites, Composites Part A :27A (1996), pp. 655-665.

[3]. J. Hashim, L. Looney, M.S.J. Hashmi, Metal matrix composites: production by the stir casting method, Journal of Materials Processing Technology 92-93(1999), 1-7

[4]. Jim Birch, New Alloys for Zinc Castings, Materials & Design Vol.11, No.2, 1990.

[5]. R.Lyon, High strength zinc alloys for engineering applications in the motor car, Metals and Materials, Vol.1 (1985), pp.55

[6]. S. Muthukumarasamy, A. Guruprasad , A. Sudhakar & S. Seshan, The Performance of Zinc Alloy Based Metal Matrix Composites Produced Through Squeeze Casting,
Materials and Manufacturing Processes, Vol.:11(3) (1996), pp.351-366.

[7]. S.Muthukumarasamy and S. Seshan, "Structure and properties of fibre reinforced Zn-27% Al alloy based cast MMCs" , Composites Vol. 26 (1994), 387.

[8]. M.A. El-Khair, A. Daoud, A. Ismail, Effect of different Al contents on the microstructure, tensile and wear properties of Zn-based alloy, Materials Letters 58(11) (2004), 1754-1760.

[9]. Shuqing Yan, Jingpei Xie, Zhongxia Liu, Wenyan Wang, Aiqin Wang and Jiwen Li " Influence on different Al contents on microstructure, tensile and wear properties of Zn based alloys" J. Mater. Sci. Technol., 26(7) (2010), 648-652.

[10]. William Mihaichuck, Advanced Technology Improves Zinc Castings, Eastern Alloys Inc. Reprinted from Precision Metal June 1989.

[11]. William Mihaichuck, Zinc Alloy Bearings Challenge the Bronzes, Eastern Alloys Inc. Reprinted from Machine Design December 10, 1981.

[12]. Zeljko Skoko, Stanko Popovic and Goran Stefanic, Microstructure of Al-Zn and Zn-Al alloys, Croat.Chem.Acta 82(2) (2009), pp. 405-420.

[13]. Béla VARGA, *et al.,* Phase Transformations in the Heat Treated and Untreated Zn-Al Alloys, Acta Universitatis Sapientiae, Electrical and Mechanical Engineering, 1(2009), 207-213

[14]. Zhu, Y. H J, Structural Changes of α Phase in Furnace Cooled Eutectoid Zn-Al Based Alloy, J Mater. Sci. Technol., Vol. 23 No.3 (2007), pp. 347-352.

[15]. S C. Sharma, B.M. Girish, Rathnakar Kamath, B.M. Satish, Graphite particles reinforced ZA-27 alloy composite materials for journal bearing applications, Wear 219(1998), 162-168.

[16]. B.M. Girish, K.R. Prakash, B.M. Satish, P.K. Jain, Phani Prabhakar, An investigation into the effects of graphite particles on the damping behaviour of ZA-27 alloy composite material, Materials and Design 32(2011), 1050–1056.

[17]. K.H.W. Seah, S.C. Sharma, B.M. Girish and S.C. Lima, Wear characteristics of as-cast ZA-27/graphite particulate composites,Composites Part A 28A(1997), 251-256

[18]. T. Savaşkan, A.P. Hekimoğlu, Microstructure and mechanical properties of Zn–15Al-based ternary and quaternary alloys, Materials Science and Engineering: A 603 (2014), 52-57.

[19]. A. Türk, M. Durman, E.S. Kayali, The effect of Cu and Al on the mechanical properties of gravity-cast hyper-eutectic Zn–Al-based alloys, Zeitschrift für Metallkunde 94(9)(2003), 1001-1005.

[20]. S. Yan, J. Xie, Z. Liu, W. Wang, A. Wang, J. Li, Influence of different Al contents on microstructure, tensile and wear properties of Zn-based alloy, Journal of Materials Science & Technology 26(7)(2010), 648-652.

[21]. B.K. Prasad, Tensile properties of some zinc-based alloys comprising 27.5% Al: effects of alloy microstructure, composition and test conditions, Materials Science and Engineering: A 245(2)(1998), 257-266.

[22]. Annual book of ASTM standards – test method for tension testing of metallic materials; (1989), p. 147–56.

[23]. S H J Lo, S.Dionne, M. Sahoo, H.M.Hawthore, Mechanical and tribological properties of zinc-aluminium metal-matrix composites, Journal of Material Science 27(1992), pp5681-5691.

[24]. Seun Samuel Owoeye & Davies Oladayo Folorunso & Babatunde Oji & Sunday Gbenga Borisade3, Zinc-aluminum (ZA-27)-based metal matrix composites: a review article of synthesis, reinforcement, microstructural, mechanical, and corrosion characteristics, IntJ.Adv. Manu.Techn, 2018. https://doi.org/10.1007/s00170-018- 2760-9.

[25]. Pulkit Garg, Anbesh Jamwal, Devendra Kumar, Kishor Kumar Sadasivuni, Chaudhery Mustansar Hussain, Pallav Gupta, Advance research progresses in aluminium matrix composites: Manufacturing & Applications, J mater res technol. 2019;8(5):4924–4939.

[26]. Anbesh Jamwal, Prem Prakash Seth, Devendra Kumar, Rajeev Agrawal, Kishor Kumar Sadasivuni, Pallav Gupta, Microstructural, tribological and compression behaviour of Copper matrix reinforced with Graphite-SiC hybrid composites, Materials Chemistry and Physics 251, 2020, 123090
https://doi.org/10.1016/j.matchemphys.2020.123090

[27]. Swati Gangwar, Viresh Payak, Vimal Kumar Pathak, Anbesh Jamwal and Pallav Gupta, Characterization of mechanical and tribological properties of graphite and alumina reinforced zinc alloy (ZA-27) hybrid metal matrix composites, Journal of Composite Materials,0(0), (2020), 1–13: DOI: 10.1177/0021998320938442.

[28]. M.T. Abou El-khair, A. Daoud, A. Ismail, Effect of different aluminium contents on the microstructure, tensile and wear properties of Zn-based alloy, Materials Letters, 19 (2003), pp. 77-81.