A comparative study of solid lubricant types on the microstructure and mechanical behaviour of AA7075-zirconium boride aerospace composites

P Loganathan¹, A Gnanavelbabu*¹, K Rajkumar², S Ayyanar¹
¹Department of Industrial Engineering, CEG Campus, Anna University, Chennai-600025, India.
²Department of Mechanical Engineering, SSN College of Engineering, Chennai-600110, India.

*Corresponding author email: agbabu@annauniv.edu

Abstract. Aluminium alloy is the most favourable material for industrial usage because of outstanding low density, specific strength and resistance to oxidation. In this study, zirconium diboride (5 wt. % ZrB₂) as hard ceramic particles and solid lubricants as hexagonal boron nitride (2.5 wt. % hBN) and tungsten disulfide (2.5 wt. % WS₂) particles were used for fabrication of AA 7075 hybrid composite. The fabrication of hybrid composites is done through liquid metallurgy technique. The uniform distribution particles on matrix material were analysed through Optical Microscope (OM) and X-Ray Diffraction (XRD) was used to identify the phase of alloy and hybrid composite materials. The Micro Vickers hardness and tensile strength were used to evaluate the mechanical strength of alloy and composites. The microstructure of composites reveals fairly distributed ceramic and solid lubricant particles over the base matrix. The XRD peaks reveals presence of AA7075, ZrB₂, hBN and WS₂ with low intensity peaks. AA7075/ZrB₂/WS₂ composite shows an improved hardness and tensile strength as compared to AA7075/ZrB₂/hBN composite. It was observed that the influence of WS₂ on AA7075/ZrB₂ composites have a higher bending strength than hBN composite and hBN particles offers easy grain movement during plastic deformation. Tensile fractured surfaces of alloy and hybrid composites were analysed through Scanning Electron Microscope (SEM). It reveals a ductile fracture in AA7075 alloy whereas ductile-brittle is observed in hybrid composites.

1. Introduction
Zinc based aluminium alloy is used for energy saving applications, due to its greater durability to undergo fatigue loading. AA7075 is mainly used in aerospace structural material as a load bearer [1-3]. Improvement in mechanical properties of an alloy is attained through hard ceramic reinforcement particles [4]. Optimal material properties are attained through hybrid composite for required applications and it’s achieved through the selection of primary and secondary particles which are not affecting base matrix properties [5]. The strengthening of aluminium alloy is normally done through even distribution of ceramic particles in the matrix. Compared to other ceramic particles, boride based particles are stronger, and its refractory nature is able to withstand elevated temperatures [6].
applications include hypersonic rocket nozzle inserts and re-entry vehicles systems in the aerospace industries [7].

Good machinability characteristics and improvement in wear resistance of composites are attained through the addition of a solid lubricant. As compare to graphite, hBN have better frictional heating properties with thermal shock resistance and density of hBN is (2.25 g cm\(^{-3}\)) which is lower than that of pure aluminium [8]. Tungsten disulphide (WS\(_2\)) are well known solid lubricant with lamellar structure with extremely low frictional properties [9-10].

Ashwath and Anthony Xavior [11] studied the mechanical behaviour of SiC and Al\(_2\)O\(_3\) hard ceramic particles reinforced with AA2900 and AA2024 alloy. Al\(_2\)O\(_3\) played a crucial role in improving the strength of the composite. Hamid Reza Ezatpour et al. [12] concluded that the mechanical and microstructural effect on AA6061/ Nano Al\(_2\)O\(_3\) particles was developed through stir casting. The strength and ductility of the composite reduced as increasing in A Al\(_2\)O\(_3\) nano particles due to agglomeration of particles and induced porosity.

Syed Nasimul Alam and Lailesh Kumar [13] carried out detailed analysis on Al matrix reinforced with Natural Flake Graphite (NFG) fabricated through powder metallurgy route. The reduction in tensile strength of composite due to brittle nature of Al\(_4\)C\(_3\) precipitate at the interface of NFG and Al matrix. Agglomeration of graphite particles leads to poor densification of composite, leading to low level of hardness for composite. Ravi Kumar et al. [14] fabricated AA6063/TiC composite with varying wt. %. The addition of TiC particles reduces ductility of the composite, but other way it increases resistance to the plastic deformation.

Ravi Kumar et al. [15] analysed the microstructural behaviour and mechanical property of AA6082/ZrO\(_2\)/Coconut Shell Ash (CSA) hybrid composite. The addition of CSA acts as a soft lubrication medium. VinodKumar et al. [16] studied the microstructure, hardness and tensile behaviour of AA7075/TiB\(_2\) composite processed through ultrasonic assisted stir casting. Agglomerates of TiB\(_2\) particles were broken-down into smaller particles through ultrasonic effect, this provides a fine distribution with a good interfacial bonding between matrix and reinforcement particles which improves the better load bearing capacity. Dinaharan et al. [17] developed AA6061/Al\(_2\)Cu composites through friction stir Welding Process (FSP). Large size Al\(_2\)Cu particles were broken into smaller particles upto 15 times less than the original size due to severe deformation.

Mathan Kumar and Kumaraswamidhas [18] attempted an analysis on the mechanical and wear behaviour of AA6061/ZrB\(_2\)/AlN composite fabricated through stir casting. The mechanical and wear resistance of the composites increases with increasing reinforcement due to strong bonding nature. Kenneth Kanayo Alaneme and Bethel Jeremiah Bamike [19] concluded the hardness and tensile strength decrease with addition of quarry dust (QD) on AA6063/quarry dust (QD)/SiC hybrid composite.

Literatures reveal the application of hBN and WS\(_2\) on AA7075/ZrB\(_2\) composite provides a great opportunity to improve its mechanical and tribological properties. Therefore, this research work investigate the effects of hBN/WS\(_2\) solid lubricant particles on the microstructural characterization and mechanical behaviour of AA7075/ZrB\(_2\) composite.

2. Experimental Setup

The stir casting method was adopted for fabrication of hybrid composites. AA7075 alloy was base matrix with hard ceramic reinforcement particle as ZrB\(_2\) with average particle size is 5.80 µm. WS\(_2\) and hBN were solid lubricants with average particle size of less than 5.00 µm. Base matrix was melted at 800 °C in an electrical resistance furnace. Argon gas was continuously supplied to furnace at a constant flow rate of 2 lpm to avoid any undesirable reactions. Before mixing of reinforcement particles into melt, they were pre-heated at 250°C for improving wettability nature and remove moisture content. The stirring process carried out through coated steel blades to create vortex formation, the pre-heated particles were transferred to molten melt. The stirring speed was maintained at 450 rpm for 5-10 min. Due to smaller size of reinforcement particles, a good bonding and fine
distribution was achieved at lower wt.% which was selected for fabrication of composites. The molten melt was poured into a pre-heated steel die with diameter 20 mm and length of 250 mm. Also composite was fabricated in plate form with dimension of 100 X 100 X 10 mm. The T6 heat treatment was carried out for alloy and fabricated hybrid composites. The fabricated samples are shown in figure 1 and its designation are displayed in table 1.

![Fabricated AA7075 hybrid composites](image)

**Figure 1.** Fabricated AA7075 hybrid composites (a) AA7075+ 5 wt. % ZrB$_2$+ 2.5 wt. % hBN, (b) AA7075+ 5 wt. % ZrB$_2$+ 2.5 wt. % WS$_2$.

### Table 1. Fabricated AA7075 hybrid composites.

| S.No | Composition | Material |
|------|-------------|----------|
| 1    | Ascast      | A0       |
| 2    | AA7075+ ZrB$_2$ (5wt% ) + 2.5 wt.% hBN | A1       |
| 3    | AA7075+ ZrB$_2$ (5wt% ) + 2.5 wt.% WS$_2$ | A2       |

Heat treated samples were subjected to microstructural analysis through an optical microscope. The different grades of SiC abrasive papers were used for polishing sample surface and Keller’s reagent were used as etchant medium to enhance the visibility of the grain boundaries. XRD is used for phase analysis of fabricated hybrid composites and X’Pert high score plus software used to identify the phases in the composites. The ASTM E384-16 standard as used for Micro Vickers hardness of the composites with applied load of 500 gf with a dwell time for 15 second. Seven readings were taken at various locations of composite and average value is calculated. The ASTM E8 standard obtained for tensile strength of composite in a computerized universal testing machine (Make: MTS, 100 KN capacity). Initial strain of 0.01 mm/s is fixed for all experiments. Three replicates were carried out and the average values were recorded. Fracture surface of the tested hybrid composites was analysed through SEM (Scanning Electron Microscope).

### 3. Results and discussions

#### 3.1. Optical Microstructure and XRD Analysis

Figure 2 reveals the optical micrographs and XRD analysis of alloy and hybrid composites. Figure 2a shows the micrograph of A0 alloy. It reveals formation of coarse and fine precipitates of MgZn, AlZn, and Al2MgCu are observed in and around the grain boundaries due to the effect of T6 treatment [20-21].
Figure 2. Optical microstructure and XRD of hybrid composites. 2 (a) A0; 2 (b) A1; 2 (c) A2; 2 (d) XRD analysis of hybrid composites.

The optical micrograph of fabricated hybrid composites is displayed in figure 2(b–c). The micrographs reveals fine distribution of ZrB$_2$/WS$_2$/hBN particles over the base matrix and it was attained through dispersion strengthening mechanism and also suspending of particles for longer time period in the molten melt based on difference in wettability factor among the base matrix and reinforcements, this fine distribution of particles tends to increase the mechanical strength of AA7075 hybrid composites [22]. The reinforcement particles obstructed the grain growth in matrix material and also it refines the grain structure.

The X-ray diffraction for the alloy and hybrid composites are presented in figure 2(d). The analysis confirmed no interfacial reaction among the fabricated composites. The diffraction pattern of the Al and reinforcement particles were observed. The presence of WS$_2$ and hBN is characterized as a solid lubricant showing a lower intensity of peaks exhibiting a binary relationship [23] due to film substrate over the AA7075/ZrB$_2$ composite materials.

3.2. Mechanical behaviour of AA7075/ZrB$_2$ hybrid Composites

The variation in hardness of AA7075 and hybrid composites is presented in figure 3. The presence of hard ceramic ZrB$_2$ particle which act as load bearing and resist the dislocations within the matrix which leads to improve hardness of the composites than base matrix. The effect on hardness of composite materials were associated with fine distribution of reinforcement particles and their bonding interface between the AA7075 [24-25]. The interfacial bonding strength of WS$_2$ was relatively higher than that of hBN, hence hardness of A2 hybrid composite (135.25 VHM) is higher than A1 hybrid composite (127.12 VHM). The Orowan and Hall-Petch strengthening mechanisms is tends to increase the hardness of the composite due to strong interface between the reinforcement and matrix material.
3.3. Tensile Strength

Figure 4 shows the tensile strength of alloy and composites. The tensile strength of composite is increased with addition of reinforcement particles as compared to A0 due to the effective load transferring between the matrix and reinforcement particles through better interfacial bonding and effect of strain gradient strengthening in the composites. The addition of hBN particles increase the brittleness of the composite, hence it decrease the tensile strength as compared to WS$_2$ composite (228 MPa).

Figure 5 (a) shows a fracture surface morphology of A0 alloy which reveals large amount of plastic flow prior to failure and indicating ductile mode of fracture. Figure 5 (b-c) shows the fracture analysis of hybrid composites, which reveals both ductile and brittle fracture mode. ZrB$_2$ particles act as grain refinement and it reduce the ductility of the composite with smaller size of dimples. The reduction of tensile strength of A1 composite is due to low density of hBN particles occupying high weight fraction in the composite and have their low strength when compared to WS$_2$ composite. The uniform distribution of particles reduce the crack propagation and improve the tensile strength.
4. Conclusions

AA7075-ZrB2 hybrid composites were successfully fabricated using the stir casting method with different solid lubricant particles.

- The fine distribution of particles on the matrix material are ensured through optical microscope and phase formation by XRD analysis.
- Hybrid composites shows higher hardness as compared to base alloy. A2 composite shows a higher hardness than A1 due slight improvement in wettability of WS2 with the matrix.
- A2 composite shows higher tensile than A1 due to the smaller grain size of composite which improved strength of the composite.
- Fractography of composites showed both ductile and brittle mode of failure,

Acknowledgments

Authors gratefully acknowledge the support of Department of Science and Technology–Science and Engineering Board (DST-SERB), (Grant No: EEQ/2017/000382) New Delhi, India.

References

[1] Rajmohan T, Palanikumar K, Ranganathan S, “Evaluation of mechanical and wear properties of hybrid aluminium matrix composites”, Transactions of nonferrous metals society of China, Vol. 23(9), pp. 2509-2517, 2013.

Figure 5. Fracture morphology of AA7075/ZrB2 composites. 5 (a) A0; 5 (b) A1; 5 (c) A2.
[2] Xiu Z, Yang W, Dong R, Hussain M, Jiang L, Liu Y and Wu G, “Microstructure and mechanical properties of 45 vol.% SiCp/7075Al composite”, Journal of Materials Science & Technology, Vol. 31(9), pp. 930-934, 2015.

[3] Saravanan L, SenthilvelaN T, “Investigations on the hot workability characteristics and deformation mechanisms of aluminium alloy-Al2O3 nanocomposite”, Materials & Design, Vol. 79, pp. 6-14, 2015.

[4] Sharma P, Sharma S, Khanduja D, “A study on microstructure of aluniniuim matrix composites”, Journal of Asian Ceramic Societies, Vol. 3(3), pp. 240-244, 2015.

[5] Singh J, Chauhan A, “Characterization of hybrid aluniniuim matrix composites for advanced applications–A review”, Journal of Materials Research and Technology, Vol. 5(2), pp. 159-169, 2016.

[6] Deaquino-Lara R, Gutiérrez-Castañeda, E, Estrada-Guel I, Hinojosa-Ruiz G, García-Sánchez E, Herrera-Ramírez JM, Pérez-Bustamante R, Martínez-Sánchez R, “Structural characterization of aluminium alloy 7075–graphite composites fabricated by mechanical alloying and hot extrusion”, Materials & Design, Vol. 53, pp. 1104-1111, 2014.

[7] Meléndez-Martinez JJ, Domínguez-Rodríguez A, Monteverde F, Melandri C, De Portu G, “Characterisation and high temperature mechanical properties of zirconium boride-based materials”, Journal of the European Ceramic Society, Vol. 22(14-15), pp. 2543-2549, 2002.

[8] Chen C, Guo L, Luo J, Hao J, Guo Z, Volinsky AA, “Aluminium powder size and microstructure effects on properties of boron nitride reinforced aluminium matrix composites fabricated by semi-solid powder metallurgy”, Materials Science and Engineering: A, Vol. 646, pp. 306-314, 2015.

[9] Huang SJ., Lin CC., Huang JY, Tenne R, “Mechanical behaviour enhancement of AZ31/WS2 and AZ61/WS2 magnesium metal matrix nanocomposites”, Advances in Mechanical Engineering, Vol. 10(2), pp. 16878140-17753442, 2018.

[10] Xie ZW, Wang LP, Wang XF, Huang L, Yang LU, Yan JC, “Mechanical performance and corrosion behaviour of TiAlSiN/WS2 multilayer deposited by multi-plasma immersion ion implantation and deposition and magnetron sputtering”, Transactions of Nonferrous Metals Society of China, Vol. 21, pp. s470-s475, 2011.

[11] Ashwath P, Xavior MA, “Effect of ceramic reinforcements on microwave sintered metal matrix composites”, Materials and Manufacturing Processes, Vol. 33(1), pp. 7-12, 2018.

[12] Ezatpour HR, Sajjadi SA, Sabzevar MH, Huang Y, “Investigation of microstructure and mechanical properties of Al6061-nanocomposite fabricated by stir casting”, Materials & Design, Vol. 55, pp. 921-928, 2014.

[13] Alam SN, Kumar L, “Mechanical properties of aluminium based metal matrix composites reinforced with graphite nano platelets”, Materials Science and Engineering: A, Vol. 667, pp.16-32, 2016.

[14] Kumar KR, Kiran K, Sreebalaji VS, “Micro structural characteristics and mechanical behaviour of aluminium matrix composites reinforced with titanium carbide”, Journal of Alloys and Compounds, Vol. 723, pp. 795-801, 2017.

[15] Kumar KR, Pridhar T, Balaji VS, “Mechanical properties and characterization of zirconium oxide (ZrO2) and coconut shell ash (CSA) reinforced aluminium (Al 6082) matrix hybrid composite”, Journal of Alloys and Compounds, Vol. 765, pp.171-179, 2018.
[16] Meti VKV, Shirur S, Nampoothiri J, Ravi, K.R., Siddhalingeshwar, I.G., “Synthesis, Characterization and Mechanical Properties of AA7075 Based MMCs Reinforced With TiB$_2$ Particles Processed Through Ultrasound Assisted In-Situ Casting Technique”, Transactions of the Indian Institute of Metals, Vol. 71(4), pp.841-848, 2018.

[17] Dinaharan I., Balakrishnan M, Selvam JDR, Akinlabi ET, “Microstructural characterization and tensile behavior of friction stir processed AA6061/Al$_2$Cu cast aluminum matrix composites”, Journal of Alloys and Compounds, Vol. 781, pp.270-279, 2019.

[18] Kumar NM, Kumaraswamidhas LA, “Characterization and tribological analysis on AA 6061 reinforced with AlN and ZrB$_2$ in-situ composites”, Journal of Materials Research and Technology, Vol. 8(1), pp.969-980, 2019.

[19] Kenneth KA, Bethel JB, “Characterization of mechanical and wear properties of aluminium based composites reinforced with quarry dust and silicon carbide”, Ain Shams Engineering Journal, Vol. 9, pp.2815–2821, 2018.

[20] Kumar PV, Reddy GM, Rao KS, “Microstructure, mechanical and corrosion behaviour of high strength AA7075 aluminium alloy friction stir welds–Effect of post weld heat treatment”. Defence Technology, Vol. 11(4), pp. 362-9, 2015.

[21] Dasgupta R, Meenai H, “SiC particulate dispersed composites of an Al–Zn–Mg–Cu alloy: Property comparison with parent alloy”, Materials Characterization, Vol. 54(4-5), pp.438-445, 2005.

[22] Rajkumar K, Thushal NA, Gnanavelbabu A, Sabarinathan P, “Experimental investigations on the Wire Electrochemical Micro Machining (WECM) integrity of AA6061-TiB$_2$ composite”. Materials Today: Proceedings, Vol. 5(2), pp. 6990-6998, 2018.

[23] Liu, S., Wang, Y., Muthuramalingam, T. and Anbuchezhiyan, G, “Effect of B$_4$C and MOS$_2$ reinforcement on micro structure and wear properties of aluminum hybrid composite for automotive applications”, Composites Part B: Engineering, Vol. 176, pp.107329, 2019.

[24] Manickam D, Santhanam SKV, Sivagnanam K, “Experimental investigation of LM25 alloy reinforced with Sic, Gr and Moa particles”, Materiali in Tehnologije, Vol. 53(3), pp. 395-398, 2019.

[25] Gnanavelbabu A, Muthazhagan C, Rajkumar K, Ayyanar S, Loganathan P, “Experimental Investigation and Reliability Analysis on Wear Performance of AA6061-B$_4$C-Nanographite Hybrid Composites” Materials Today: Proceedings, Vol. 5(2), pp. 8436-8445, 2018.