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## INVESTIGATION OF STRUCTURAL AND MECHANICAL PROPERTIES OF Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> HYBRID COMPOSITES FABRICATED BY POWDER METALLURGY

The present study focuses on the fabrication and characterization of novel hybrid Al matrix composites with a combination of two ceramic reinforcements, i.e. Al<sub>2</sub>O<sub>3</sub>, SiC and one solid lubricant, i.e. WS<sub>2</sub>. This hybrid composite was fabricated by means of the powder metallurgy process. The impact of the hybrid combination of reinforcements in different wt.% on the properties of the hybrid composites was studied. The density of the composites increases from 2.689 to 2.796 g/cm<sup>3</sup> with an increase in wt.% of WS<sub>2</sub>. Uniform distribution of the reinforcing particles in the matrix phase was determined by SEM. The results of, for instance, density measurements and microstructural analysis indicate significant improvement in the physical and mechanical properties with the increase in the wt.% of WS<sub>2</sub>. The microhardness of the as-fabricated composites rises from 98 HV to 119.7 HV with the increase in the wt.% of WS<sub>2</sub> from 0 to 6 wt.%. The novel combination of Al with SiC, Al<sub>2</sub>O<sub>3</sub> and WS<sub>2</sub> can be used to create a suitable and sustainable hybrid metal matrix composite for the automotive industry as a replacement for single ceramic and single solid lubricant composites.

**Keywords:** hybrid metal matrix composites (AMCs), ceramic reinforcements, hardness, solid lubricant, tungsten disulphide

### INTRODUCTION

In recent years, researchers have been trying to produce energy-efficient and environmentally-friendly materials for automotive and aerospace applications. The technological boom in materials engineering tries to fill the gaps by discovering new technologies and materials. Aluminium metal matrix composites are among those with an extraordinary combination of qualities such as low density with high strength and toughness, as well as good lubrication qualities. These characteristics of Al metal matrix composites attract their use for many automotive parts [1-7]. Moreover, several investigations have reported that metal matrix composites with hybrid reinforcements possess superior properties to single-reinforced composites [8-12]. Earlier investigations on the development of aluminium-based hybrid composites with SiC and B<sub>4</sub>C suggest that adding B<sub>4</sub>C significantly influences the mechanical properties. Nonetheless, they exhibit very poor wettability, low wear resistance and are more costly to produce compared to composites with single SiC reinforcement [13]. Kaushik and Rao [14] developed Al6082-SiC-Gr hybrid composites by powder metallurgy and found that with an increase in the SiC content, the wear resistance rose uniformly. Sahoo et al. [15] attempted to fabricate a hybrid composite by adding hBN with Al-SiC using

the stir-casting method. The results show improved physical and mechanical properties with an increase in hBN up to 5 wt.% and SiC up to 4 wt.%. The properties were enhanced by 26% compared to the unreinforced aluminium matrix, which has higher wear resistance. Rahimian et al. [16] fabricated an Al-Al<sub>2</sub>O<sub>3</sub> metal matrix composite with improved wear properties by optimizing the sintering temperature and time. The authors found that the densification of the composites improved at higher sintering temperatures and possessed a low level of porosity. A study was conducted by Umasankar et al. [17] to examine the sintering behaviour an aluminium alloy reinforced with SiC particles produced by the powder metallurgy process. Singh et al. [18] investigated the structural and mechanical properties of a WS<sub>2</sub> reinforced Al-SiC hybrid composite fabricated via the powder metallurgy technique. They experimented with varying contents of the WS<sub>2</sub> reinforcing phase, and discovered that increasing the WS<sub>2</sub> content enhanced the density and hardness of the composites.

From these findings, it is clear that a balance of the tribo-mechanical properties is possible by putting a solid lubricant and ceramic reinforcement together in the aluminium matrix. This helps to expand the use of solid lubricants in various applications such as in the

automotive and aerospace industry, and to become less dependent on liquid lubrication systems. According Cairns et al. [19], MoS<sub>2</sub> and WS<sub>2</sub> are two excellent examples of transition metal dichalcogenides (TMDs), which exhibit comparatively low friction during sliding action at low humidity, vacuum, and moderate temperature settings. TMD components are also added to oils as particle additives to improve their performance and increase the wear life. Researchers [20-26] found that WS<sub>2</sub> provides self-lubrication, which enhances the wear life of the composites. That is why the demand for WS<sub>2</sub> is increasing significantly, which can be used as a reinforcement in the Al matrix. WS<sub>2</sub> is a transition metal that has a hexagonal layered structure (S-W-S). In this layered structure, the atoms within each layer are held by strong covalent bonds. The individual layers have significant distances between one another and are held by weak Van der Waals bonds. Due to weak Van der Waals bonds, the WS<sub>2</sub> structure permits greater compression, different from high pressure, where the force between the S-S planes increases owing to repulsing forces. This ability has made WS<sub>2</sub> one of the most promising lubricating materials [27].

WS<sub>2</sub> has a low coefficient of friction and higher oxidation resistance with thermal stability. WS<sub>2</sub> is widely used because of its lamellar structure and its sustainability in extreme environmental conditions, but there is a huge gap in using the potential of WS<sub>2</sub> to fabricate new-age self-lubricating materials. WS<sub>2</sub> is used as single reinforcement in the Al matrix, which provides enhancement of the structural and mechanical properties of the composites. Therefore, in the present study, an attempt was made to fabricate Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> hybrid composites by means of powder metallurgy by varying the wt.% of WS<sub>2</sub>. Hybrid reinforcement was employed to study the impact of WS<sub>2</sub>. The physical, morphological, and mechanical properties of the newly developed hybrid composites are investigated, providing comprehensive knowledge for its engineering applications.

## MATERIALS AND METHOD

The novel hybrid composite is composed of Al, SiC, Al<sub>2</sub>O<sub>3</sub>, and WS<sub>2</sub> powders, which were purchased from Intelligent Materials Pvt. Ltd., India. The specifications of these materials are listed in Table 1.

TABLE 1. Characteristics of Al, Al<sub>2</sub>O<sub>3</sub>, SiC, and WS<sub>2</sub> powders

Material	Mesh size [μm]	Density [g/cm <sup>3</sup> ]	Purity [%]
Al (Matrix)	40-50	2.71	99
Al <sub>2</sub> O <sub>3</sub> (Reinforcement 1)	40-50	3.95	99
SiC (Reinforcement 2)	40-50	3.21	99
WS <sub>2</sub> (Reinforcement 3)	5-10	7.5	99

Powder metallurgy was employed to fabricate the Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> hybrid composite using a three-step process. This process consisted of powder mixing, compaction, and sintering. The detailed process is explained in the flow diagram in Figure 1. Different weight fractions of the powders, i.e. Al, Al<sub>2</sub>O<sub>3</sub>, SiC, and WS<sub>2</sub>, as specified in Table 1, were weighed using digital scales with a least count of 0.0001 g. The weighed powders were thoroughly mixed with acetone by means of a mechanical stirrer. To obtain a uniform mixture and to avoid agglomeration of the powder particles, the mechanical stirrer was rotated with a speed of 315 rpm at room temperature for 4 h. After adequately mixing the powders, the resulting mixtures were removed and dried at 50°C. Then the dried powder was mixed with polyvinyl acetate, which helps to bind the particles and distilled water was added for blending.

In the next stage, the blended powders were compressed in a uniaxial hydraulic pellet press at 560 MPa using an EN9 mould to make green pellets 10 mm in diameter and 10 mm in length. Zinc stearate was applied to lubricate the mould wall before every compaction for easy removal of the pellets from the mould. The green pellets were sintered at 600°C for 90 min in a muffle furnace in an inert gas atmosphere to avoid oxidation of the aluminium matrix. The pellets were heated and cooled in the furnace at a constant heating and cooling rate, i.e. at 10°C/min. The same procedure was implemented to fabricate Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> hybrid composites with different contents of WS<sub>2</sub>, i.e. 0, 3, and 6 wt.% and keeping the wt.% of SiC and Al<sub>2</sub>O<sub>3</sub> fixed at 5 wt.% for all the composites. The processing parameters considered for fabrication of the Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> composite pellets are 560 MPa compaction pressure in a universal testing machine and 600°C sintering temperature in the muffle furnace, which is well explained by Biswal et al. [28]. In the study, the optimised temperature and pressure were decided by conducting the experiments at variable temperatures and compaction pressures. At 560 MPa compaction pressure and 600°C sintering temperature, the sintered samples are denser with minimum porosity as observed by Biswal et al. [28]. The powder metallurgy process was adopted to maintain uniform distribution of the reinforcing particles in the matrix phase and to avoid the reaction of materials in the molten state.

The characterization process included X-ray diffraction (XRD, Ultima-IV, Rigaku, Japan) for phase analysis and scanning electron microscopy (SEM) for microstructural investigations. The densities and porosities were calculated with the help of Archimedes' principle and the rule of mixtures. The hardness of the fabricated samples was measured using a Vickers microhardness tester (Leco LM248AT) at a 50 gf load for 15 s. The composition of the sample was investigated using energy dispersive spectroscopy (EDS) analysis.

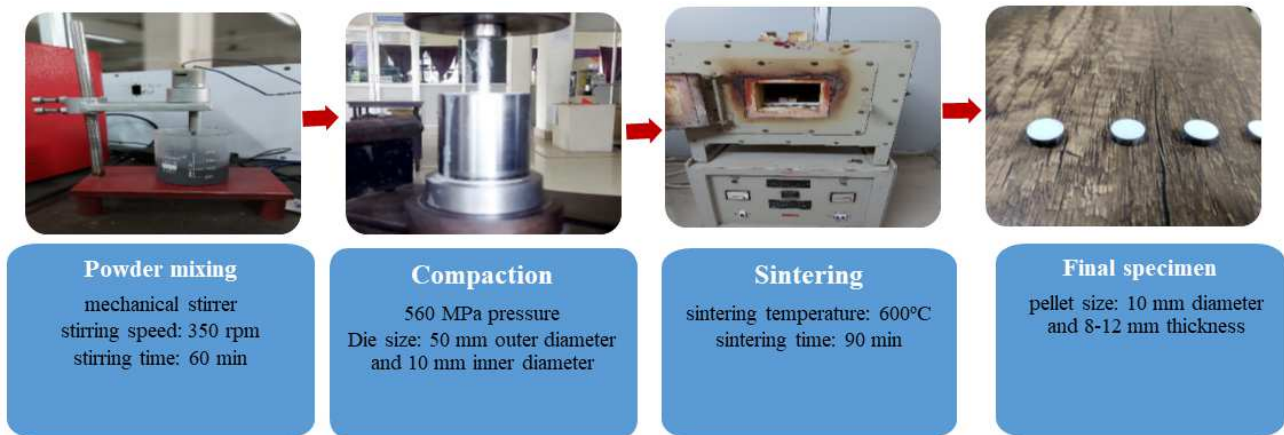


Fig. 1. Schematic representation of experiment with sample preparation

## RESULTS AND DISCUSSION

The physical and mechanical properties of the hybrid composites were evaluated using different testing methods as discussed in the experimental procedure section. The sintered density and theoretical density of the H1, H2, and H3 samples were measured and analysed. The density of the sintered samples was measured employing the Archimedes method, and then compared with the theoretical density, which was calculated using the rule of the mixtures. The percentage of porosity of the samples was calculated by means of both the theoretical and experimentally measured density. The density and porosity results are given in Table 2. From the results, it was found that at a higher wt.% of  $WS_2$ , the porosity is a minimum of 1.2% and the density is a maximum of  $2.83 \text{ g/cm}^3$ . The compactness of the composite with 6 wt.%  $WS_2$  is more than the two other composites. It is due to the presence of micron-size  $WS_2$  which fills the pores. As the amount of  $WS_2$  increased from 0 to 6 wt.%, the porosity decreased from 2.21 to 1.12%. The density of the three composites is enhanced owing to the addition of a higher wt.% of both the ceramic and solid lubricant as mentioned by Biswal et al. [28, 29].

TABLE 2. Physical properties of Al hybrid composites

Properties	Reinforcement content [wt.%]				Sintered density $\rho_s$ [ $\text{g/cm}^3$ ]	Theoretical density $\rho_{th}$ [ $\text{g/cm}^3$ ]	Porosity [%]
	Al	$Al_2O_3$	SiC	$WS_2$			
H1	90	5	5	0	2.689	2.75	2.21
H2	87	5	5	3	2.77	2.81	1.42
H3	84	5	5	6	2.796	2.83	1.20

The XRD patterns of the hybrid composites are presented in Figure 2. From the XRD analysis, it is observed that there is a phase shift of  $1^\circ$  after sintering. The presence of  $WS_2$  is visible at H3 with 6 wt.%  $WS_2$  at  $28.814^\circ$  [0, 0, and 4] and  $33.49^\circ$  [0, 0, and 6]. Al peaks are dominant and visible at  $39^\circ$ ,  $45^\circ$ ,  $65^\circ$ ,  $78^\circ$ , and  $82.5^\circ$  as compared to SiC,  $Al_2O_3$  and  $WS_2$ . There are no other elements present in the sample after sinter-

ing, which is validated by the XRD pattern shown in Figure 2 and the EDS profile given in Figure 3 of all the three composites. The XRD results indicates that the reaction of molten  $WS_2$  at the sintering temperature of  $600^\circ\text{C}$  is controlled as no other compound phase is present in the sample. Figure 3 presents the microstructure of the composites, indicating that there is uniform distribution of the reinforcing particles in the matrix phase. Few pores or voids are visible, which may occur as a consequence of improper sintering. No sign of agglomeration was observed, which enhances the strength of the composites.

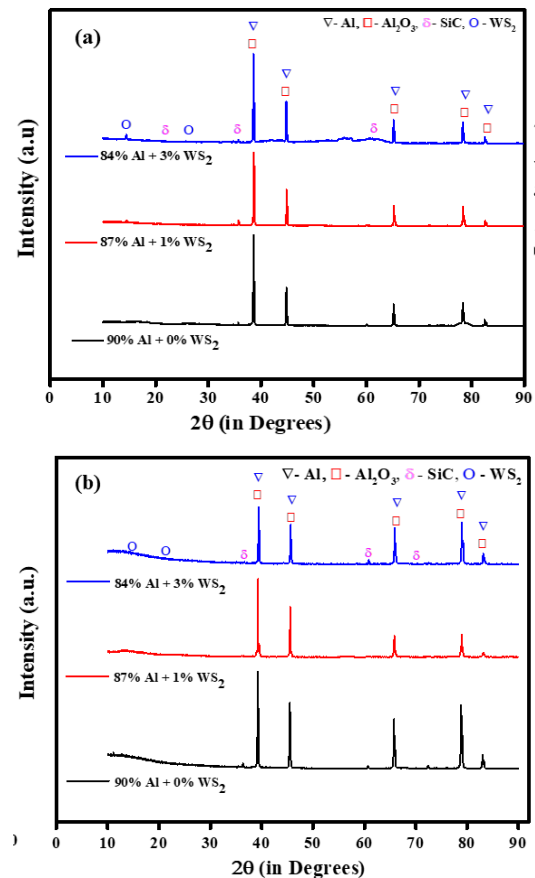


Fig. 2. XRD pattern of H1, H2, and H3 hybrid composites: a) before and b) after sintering

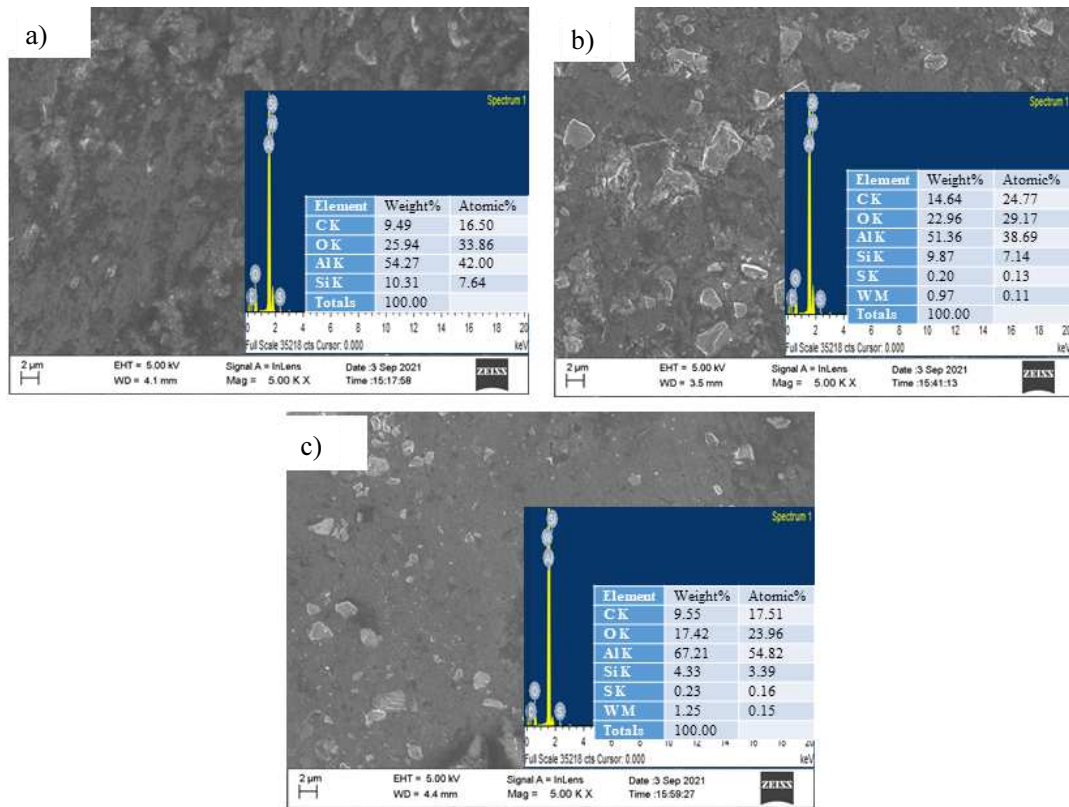


Fig. 3. Microstructure and corresponding EDS of hybrid composites: a) H1, b) H2, c) H3

The microhardness of the fabricated composite samples was determined using the Vickers microhardness tester by making seven indentations at different points on the surface of the sample. Figure 4 shows the variation in microhardness for samples H1, H2, and H3. From the graph, it was found that the hardness grew with respect to the increase in the wt.% of WS<sub>2</sub>. This is due to the addition of the highly dense reinforcing phase, i.e. WS<sub>2</sub>.

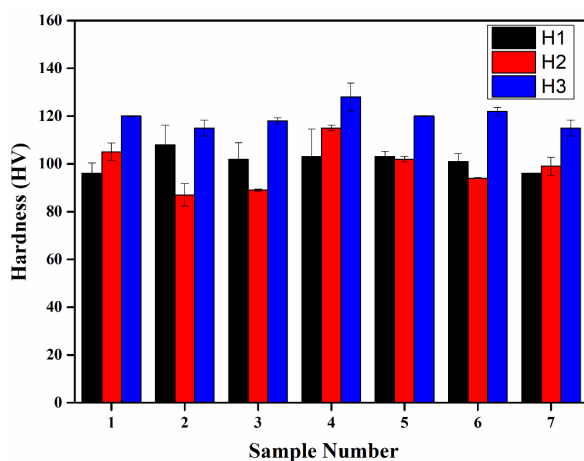


Fig. 4. Comparison of hardness for H1, H2, and H3 hybrid composites

Hard ceramic reinforcements such as SiC and Al<sub>2</sub>O<sub>3</sub> helped to enhance the hardness value for the three composites. The results shown in Figure 4 indicate that the hardness of these composites (H1, H2, and H3) is

greater than the Al-SiC, Al-Al<sub>2</sub>O<sub>3</sub>, Al-WS<sub>2</sub>, Al-SiC-Al<sub>2</sub>O<sub>3</sub>, and Al-Al<sub>2</sub>O<sub>3</sub>-WS<sub>2</sub> composites [30-33]. The highest average hardness value is 119.71 HV for the H3 composite, whereas the H1 composite has the lowest value, i.e. 98 HV.

## CONCLUSIONS

Based on the current situation of the development of hybrid composites, the evolution of material development helps to reduce friction and save energy. In this regard, the present study is a novel approach to reach that goal. The fabrication of multiple reinforced aluminium composites not only creates a demand but is also a sustainable solution for the automotive industry. The development of a novel hybrid composite with the combination of Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> provides notable changes in both the physical and mechanical properties. The Al-Al<sub>2</sub>O<sub>3</sub>-SiC reinforced composite with 6 wt.% WS<sub>2</sub> obtained better results as compared to the other two composites. In the composite with 6 wt.% WS<sub>2</sub>, the density and porosity of the composite are 2.796 g/cm<sup>3</sup> and 1.20%, respectively, which is more than the composites with 0 and 3 wt.% WS<sub>2</sub>. The hardness of the composites increased by 2.06 and 18.5% from 0 to 3 wt.% and 3 to 6 wt.% WS<sub>2</sub>, respectively.

Finally, Al-Al<sub>2</sub>O<sub>3</sub>-SiC-WS<sub>2</sub> is a novel hybrid composite with multiple reinforcements without agglomeration. This composite can be used as a self-lubricating material with high corrosion and wear resistance, which

can perform outstandingly. The findings of the present study provide a guideline to increase the wt.% of reinforcement ( $WS_2$ ) in the matrix phase to control the properties of the hybrid composites as well as to optimize it in order to achieve the best combination of mechanical, tribological and corrosive properties.

## REFERENCES

- [1] Rana V., Kumar H., Kumar A., Fabrication of hybrid metal matrix composites (HMMCs) – A review of comprehensive research studies, *Materials Today: Proceedings* 2022.
- [2] Wozniak J., Kostecki M., Cygan T., Buczek M. Olszyna A., Self-lubricating aluminium matrix composites reinforced with 2D crystals, *Composites Part B: Engineering* 2017, 111, 1-9.
- [3] Rajesh A.M., Kaleemulla M., Doddamani S., Bharath K.N., Material characterization of SiC and  $Al_2O_3$  reinforced hybrid aluminium metal matrix composites on wear behaviour, *Advanced Composites Letters* 2019, 28, 0963693519856356.
- [4] Omrani E., Afsaneh D.M., Pradeep L.M., Pradeep K.R., Influences of graphite reinforcement on the tribological properties of self-lubricating aluminium matrix composites for green tribology, sustainability, and energy efficiency – a review, *The International Journal of Advanced Manufacturing Technology* 2016, 83 (1-4), 325-346.
- [5] Ravishankar B., Nayak S.K., Kader M.A., Hybrid composites for automotive applications – A review, *Journal of Reinforced Plastics and Composites* 2019, 38(18), 835-845.
- [6] Kant S., Verma A.S., Stir casting process in particulate aluminium metal matrix composite: a review, *International Journal of Mechanics and Solids* 2017, 9(1), 61-69.
- [7] Sahoo S., Self-lubricating composites with 2D materials as reinforcement: A new perspective, *Reinf. Plast.* 2021, 65(2), 101-103.
- [8] Mistry J.M., Gohil P.P., An overview of diversified reinforcement on aluminium metal matrix composites: Tribological aspects, *Proceedings of the Institution of Mechanical Engineers, Part J, Journal of Engineering Tribology* 2017, 231(3), 399-421.
- [9] Niste V.B., Ratoi M., Tanaka H., Xu F., Zhu Y., Sugimura J., Self-lubricating Al- $WS_2$  composites for efficient and greener tribological parts, *Scientific Reports* 2017, 7(1), 1-14.
- [10] Tosun G., Kurt M., The porosity, microstructure, and hardness of Al-Mg composites reinforced with microparticle SiC/ $Al_2O_3$  were produced using powder metallurgy, *Composites Part B, Engineering* 2019, 174, 106965.
- [11] Wu C., Ma K., Wu J., Fang P., Luo G., Chen F., Shen Q., Zhang L., Schoenung J.M., Lavernia E.J., Influence of particle size and spatial distribution of  $B_4C$  reinforcement on the microstructure and mechanical behaviour of precipitation-strengthened Al alloy matrix composites, *Materials Science and Engineering: A* 2016, 675, 421-430.
- [12] Singh J., Chauhan A., Characterization of hybrid aluminium matrix composites for advanced applications – A review, *Journal of Materials Research and Technology* 2016, 5(2), 159-169.
- [13] Kumar C.R., Malarvannan R.R.R., JaiGanesh V., Role of SiC on mechanical, tribological and thermal expansion characteristics of  $B_4C$ /Talc-reinforced Al-6061 hybrid composite, *Silicon* 2020, 12(6), 1491-1500.
- [14] Kaushik N.C., Rao R.N., Effect of applied load and grit size on wear coefficients of Al 6082-SiC-Gr hybrid composites under two-body abrasion, *Tribology International* 2016, 103, 298-308.
- [15] Sahoo S., Samal S., Bhoi B., Fabrication and characterization of novel Al-SiC-hBN self-lubricating hybrid composites, *Materials Today Communications* 2020, 25, 101402.
- [16] Rahimian M., Parvin N., Ehsani N., The effect of production parameters on microstructure and wear resistance of powder metallurgy Al- $Al_2O_3$  composite, *Materials and Design* 2011, 32, 1031-1038.
- [17] Umasankar V., Experimental evaluation of the influence of processing parameters on the mechanical properties of SiC particle reinforced AA6061 aluminium alloy matrix composite by powder processing, *Journal of Alloys and Compounds* 2014, 582, 380-386.
- [18] Singh N., Haq M.I.U., Raina A., Anand A., Kumar V., Sharma S.M., Synthesis and tribological investigation of Al-SiC based nanohybrid composite, *Alexandria Engineering Journal* 2018, 57, 1323-1330.
- [19] Cairn E., Ayyagari A., McCoy C., Berkebile S., Berman D., Aouadi S.M., Voevodin A.A., Tribological behaviour of molybdenum disulfide and tungsten disulfide sprayed coatings in low viscosity hydrocarbon environments, *Tribology International* 2023, 179, 108206.
- [20] Kang X., Sun Y., Zhang L., Self-lubricating and high wear resistance mechanism of silver matrix self-lubricating nanocomposite in high vacuum, *Vacuum* 2020, 182, 109768.
- [21] Sangaravadivel P., Sudarvannan V., Gukan R., Pradeep A.D., Investigation of mechanical properties on AA7075 matrix composites reinforced with tungsten disulfide particles, *Materials Today: Proceedings*, 2021, 45, 7959-7964.
- [22] Huang S.J., Peng W.Y., Visic B., Zak A., Al alloy metal matrix composites reinforced by  $WS_2$  inorganic nanomaterials, *Materials Science and Engineering A*, 2018, 709, 290-300.
- [23] Essa F.A., Zhang Q., Huang X., Investigation of the effects of mixtures of  $WS_2$  and ZnO solid lubricants on the sliding friction and wear of M50 steel against silicon nitride at elevated temperatures, *Wear* 2017, 374, 128-141.
- [24] Zhu J, Zeng Q, Yan C., He W.  $WS_2$  nanopowders as high-temperature lubricants: an experimental and theoretical study, *ACS Applied Nano Materials* 2019, 2(9), 5604-5613.
- [25] Al-Samarai R.A., Mahmood A.S., Ahmed O.M., Influence of  $WS_2$  nanoparticles lubricants on physical characteristics of wrought aluminium alloys, *Iraqi Journal of Science* 2019, 1240-1250.
- [26] Sun Y., Wu J., Zhang L., Fabrication of Ag- $WS_2$  composites with preferentially oriented  $WS_2$  and its anisotropic tribology behaviour, *Materials Letters* 2020, 260, 126975.
- [27] Joseph S., Mohan J., Lakshmy S., Thomas S., Chakraborty B., Thomas S., Kalarikkal N., A review of the synthesis, properties, and applications of 2D transition metal dichalcogenides and their heterostructures, *Materials Chemistry and Physics* 2023, 127332.
- [28] Biswal S.R., Sahoo S., Fabrication of  $WS_2$  dispersed Al-based hybrid composites processed by powder metallurgy: effect of compaction pressure and sintering temperature, *Journal of Inorganic and Organometallic Polymers and Materials* 2020, 30, 2971-2978.
- [29] Biswal S.R., Sahoo T., Sahoo S., Prediction of grain boundary of a composite microstructure using digital image processing: A comparative study, *Materials Today: Proceedings* 2021, 41(2), 357-362.
- [30] Ahmadi A., Toroghinejad M.R., Najafizadeh A., Evaluation of microstructure and mechanical properties of Al/ $Al_2O_3$ /SiC hybrid composite fabricated by accumulative roll bonding process, *Materials and Design* 2014, 5, 13-19.
- [31] Radhika N., Balaji T.V., Palaniappan S., Studies on mechanical properties and tribological behaviour of LM25/SiC/ $Al_2O_3$

- composites, *Journal of Engineering Science and technology* 2015, 10(2), 134-144.
- [32] Biswal S.R., Sahoo S., Structural and mechanical properties of a novel Al-Al<sub>2</sub>O<sub>3</sub>-WS<sub>2</sub> hybrid composites, *Materials Letters* 2022, 307, 131017.
- [33] Biswal S.R., Sahoo S., A comparative analysis on physical and mechanical properties of aluminum composites with Al<sub>2</sub>O<sub>3</sub> and WS<sub>2</sub> reinforcement, In: Pradhan P., Pattanayak B., Das H.C., Mahanta P. (eds), *Recent Advances in Mechanical Engineering, Lecture Notes in Mechanical Engineering*, Springer, Singapore 2023.