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A study on tribological behavior of Al-4%Mg incorporated with MoS₂

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Abstract

This investigation studies the dry sliding wear behavior of Al-4%Mg matrix composites reinforced with different wt% of molybdenum disulfide (2, 4 & 6 wt%) produced using powder metallurgy route. The processed powders are initially characterized by Scanning Electron Microscopy (SEM) equipped with Energy Dispersive x-ray Spectroscopy (EDS). To authenticate the decisive distribution of the reinforcement particles with the matrix material, SEM analysis has been employed. The wear test is conducted on a pin-on-disc machine based on L₂⁷ orthogonal array with three control factors (three level) sliding velocity (0.5, 2.0, 3.5 m s⁻¹), applied load (5, 15, 25 N) and sliding distance (500, 750, 1000 m) to inspect the responses namely wear loss and coefficient of friction. Moreover, the worn surface and wear debris analysis of the composite pin surface have been examined through SEM to identify the wear mechanism. It has been identified that the development of lubricant layer is diminished by increasing the wt% of MoS₂ particulates and the wear loss is diminished. Likewise, diminutive debris have attained in the worn surface of Al-Mg-6 wt% MoS₂ composite material, since effectual solid lubricant properties have been exposed. Higher plastic deformation is attained at amplified load and it affects the pin surface and thus, Coefficient of Friction (COF) is augmented.

1. Introduction

Composites have been discovered ubiquitously in the ecosystem certainly since the appearance of earth. However, as the technology raises, humans still need to expand some of the properties of the naturally extorted metal ores to accomplish the daily requirements. In order to accomplish the properties, it is intended to formulate the composites with distinguished properties and these are quantified as manmade or artificial composites. Generally, these synthetic metal composites are completed by taking a base (matrix) material and a particular property of it will be elevated by the accumulation of two or more particulates through several processes like Stir Casting, Powder Metallurgy (PM), Centrifugal Casting etc. Among these methods, powder metallurgy technique is the greatest technique to develop a composite material. Low density metals such as aluminum, magnesium, titanium, and alloys usually are used as matrix [1].

Aluminium and its alloys are the lightweight structural materials and extensively used in automotive and aerospace industries. Qian and Schaffer [2] proposed that the hard-ceramic reinforcement materials used in the fabrication of Aluminium composite materials is to make the materials with the combination of low density and high strength. Xie et al [3] investigated the effect of Mg on the sintering of Al-Mg alloy powders by pulse electric-current sintering method. They indicated that deoxidization mechanism of Mg, is an effective method for removing the oxide films on Al alloy powder surface. Asavavisithchai and Kennedy [4] found that in order to increase the wettability of Aluminium, highly reactive elements such as Mg are added. The addition of magnesium is likely to increase the wettability of the reinforcement, mechanical strength, and corrosion resistance.

Ravindran et al [5], described that dispersion of the reinforcement particles is challenging during the manufacturing of Metal Matrix Composites. Powder metallurgy overcomes the negative effects of liquid state
processing methods such as stir casting. In powder metallurgy, the reinforcement is homogeneously dispersed in the matrix. Akbarpour and Pouresmaeil [6] described that the density of the fabricated composites through powder metallurgy technique was nearer to the theoretical density with consistent dispersal in the matrix material. Hassan et al [7] disclosed that the COF was augmented by the occurrence of SiC particulates; nevertheless, the wear resistance was significantly obtained by initiating the SiC particles in Al-Mg-Cu materials. Woo and Zhang [8] fabricated the composite material by reinforcing the SiC nano material with Al–Si–Mg alloy through powder metallurgy method and achieved a better bonding between the reinforcement and the matrix material. Hou et al [9] examined the microstructural and mechanical properties of Mg-Al-Ni alloys prepared through powder metallurgy process at high temperatures.

Wear is the common problems in materials that leads to the replacement of components and assemblies in engineering. When two solid surfaces are placed in solid-state contact, it is not easy to envision the absence of some wear even in the most efficiently lubricated systems because of asperity contact. It is need to reduce the material problems encountered by replacing with the self-lubricating composite materials [10].

Narayanasamy and Selvakumar [11] proposed that Graphite and molybdenum disulphide are the major solid lubricants, having layered-lattice structure. Lot of researches have been made to improve the friction and wear properties of aluminum composites by adding solid lubricants. The reduction in wear loss happens with the addition of graphite which assist in the formation of a solid lubricating film. Also, this will reduce the friction coefficient of the aluminium matrix. Prabhu et al [12] described that Aluminium alloy—graphite particulate composites have importance as a self-lubricating material through the enhancement of the wear resistance, machinability and delayed onset of severe wear and seizure. Prabhu and vedantam [13] determined that the composite material attained through powder metallurgy process had attained inferior porosity with regulated stream of solid lubricant, however with extreme density ascribed to the deficiency of third-body wear which improved the braking function.

An investigation of machining Al–Gr composites by Krisnamurthy et al [14] showed a considerable reduction of cutting forces, which has been attributed to a possible reduction of friction due to the solid lubrication of Gr particulates. Narayanaswamy and Selvakumar [15] portrayed that the addition of MoS2 particulates was evenly distributed with better bonding in the matrix phase by compacting and sintering process. Corrochano et al [16] prepared Al-Mg-Si/MoS2 composites through powder metallurgy process and achieved a homogenous distribution of MoSi2 particulates in the matrix alloy. And discovered that by diminishing the size of the reinforcement particles, the resistance of wear was augmented. This was owed to the intensification of consistent dispersal of MoSi2 particulates with the matrix phase. Ram Prabhu et al [17] decided that the composite material that were sintered with 1000°C had superior densification and it resulted in augmented wear resistance and breaking function of the composite materials. Senthil Kumar et al [18] have discovered that the hardness of the composite material is enhanced by amplifying the wt% of MoS2 particles. Narayanasamy and Selvakumar [19] have defined that the hardness of the composite materials is augmented with increasing wt% of MoS2.

From the literature review it seems that no wear work has been conveyed out by reinforcing MoS2 with Al-Mg matrix material. In this present work, Mg is kept at constant wt% and MoS2 has been varied at diverse wt% to examine the wear performance. The preparation of the composite materials has undergone with powder metallurgy process. The main aim of this research work is to study the wear performance of Al-Mg based composite materials by varying the wt% of MoS2 particulates. To inspect the wear analysis of the composite materials, wear loss and coefficient of friction are investigated by analyzing the parameters like sliding speed or velocity (m s\(^{-1}\)), applied load (N) and sliding distance (m) through L\(_{27}\) orthogonal array. Moreover, to evaluate the worn surface and wear debris particles of the worn specimen, SEM inspection is utilized.

2. Materials and methods

2.1. Materials selection

Aluminium (Al) powder (99.5% purity, <44 \(\mu\)m particles size) procured from Uni-chemicals, India is used as the matrix material. Magnesium (Mg) powder (99.5% purity, 100 \(\mu\)m mean particles size) and molybdenum disulfide (MoS2) powder (99.5% purity, <30 \(\mu\)m mean particles size) supplied by M/s Sigma Aldrich, Germany are used as the reinforcements. The properties of the received powders are listed in table 1.

The SEM analysis of the purchased materials are examined as shown in figures 1(a)–(c). A uniform spherical shape is noticed for Aluminium powder figure 1(a). An irregular shape is seen for Magnesium powder in figure 1(b) and MoS2 have a layer-lattice structure (figure 1(c)).
2.2. Material processing

The fabrication of Al-Mg composite materials is conducted through powder metallurgy technique. Initially, the powders have been prepared as per the prepared wt% using weighing scale with an accuracy of ±0.1 mg. Totally, four combination of materials have been prepared to carry out the present research work by mixing Mg as constant wt% (4) and different wt% (2, 4 and 6) of MoS₂ particles as mentioned in table 2. The elemental powders are blended at 200 rpm for 1 h with a ball to powder weight ratio of 10:1 and toluene as a process control agent in argon atmosphere high energy ball mill as per the combination mentioned in table 2. After the mixing process, the mixed materials are heated in an oven up to 120 °C for 1.5 h to eradicate the unpredictable substances. Then, the mixed powders are compacted in a die with a diameter of 18 mm and height of 26 mm in a uniaxial hydraulic machine at a pressure of 650 MPa. Die wall lubrication using zinc stearate is performed.

Table 1. Properties of received powders.

| Sl No | Description                              | Aluminium powder | Magnesium powder | Molybdenum di sulphide powder |
|-------|------------------------------------------|------------------|------------------|------------------------------|
| 1.    | Density (kg m⁻³)                         | 2700             | 1738             | 5060                         |
| 2.    | Melting point (°C)                       | 660              | 650              | 1185                         |
| 3.    | Youngs Modulus (GPa)                     | 70               | 45               | 264                          |
| 4.    | Thermal conductivity (W mK⁻¹)            | 237              | 156              | 130                          |

Figure 1. SEM image of received (a) Aluminium powder; (b) magnesium powder; (c) molybdenum di sulphide powder. Moreover, Energy Dispersive x-ray Analysis (EDAX) analysis has been conducted as cited in figure 2(a)–(c) on the procured powder materials to confirm their presence.
manually before each run. Afterward, the sintering is done in a muffle furnace for all the compacted specimens. The green compacts are kept in a tray containing silica sand for uniform heating and sintered in a muffle furnace at 560 °C for 2 h and allowed to get cooled to room temperature in the furnace itself. The sintering process is completed under the argon atmosphere to avoid oxidation. After the completion of sintering process, the ends of the sintered specimens are polished with 600, 800 and 1000 grades of abrasive paper sequentially. Further, the disc polishing is done using 1 μm diamond paste suspended in distilled water. The detailed experimental sequence is illustrated in the flow diagram figure 3.

2.3. Micro hardness
The hardness test is handled to evaluate the strength of the composite materials. To estimate the micro hardness of the sintered samples, Vickers hardness test equipment is utilized. Utilizing disc polishing machine, the sintered samples are polished like a mirror finished surface for the hardness test. ASTM E3 84-99 is followed to conduct the micro hardness test.

2.4. Wear test
The sintered samples are machined in Wire Electrical Discharge Machining process to prepare composite pin as per ASTM: G99-05, with a diameter of 8 mm and length of 25 mm. Pin-on-disc apparatus is employed to conduct dry sliding wear test. EN-31 steel is used counter disc material. The wear test experiment is designed based on L27 orthogonal array. The wear performance is scrutinized by examining the control factors such as sliding speed (m s⁻¹), applied load (N) and sliding distance (m). Each control factor is analyzed at three levels.
and their values are shown in Table 3. Experimentation runs in accordance with the L27 orthogonal array are presented in Table 4.

The pin is placed in the wear test sample holder for testing. The composite pin is weighed before and after each test with an accuracy of ±0.1 mg to calculate the wear loss. Acetone is utilized to clean the pin and counter disc to remove the accumulated wear debris and eradicate the traces. During each wear experiment, the frictional force values are recorded. Frictional coefficient is calculated by dividing the frictional force with the applied load. The worn surface of the pin specimens and wear debris particles are examined using scanning electron microscopy (SEM) to determine the wear mechanisms.

3. Results and discussions

3.1. Microstructural analysis of composite materials

Figures 4(a)–(d) elucidates the microstructure analysis of the produced Al-4% Mg composite and Al-4% Mg-(2, 4 and 6) wt% MoS2 composite materials. Figure 4(a) displays the microstructure of the compacted and sintered Al-4% Mg composite sample. This illustrates that the magnesium powder is evenly distributed with the aluminum matrix. Figures 4(b)–(d) express the microstructure of the compacted and sintered Al-4% Mg-MoS2
composites. It confirms that MoS$_2$ are consistently dispersed with the matrix (Al-4% Mg) material. The MoS$_2$ are noticed on the surface of the samples with the incremental manner figure 4(d). From the microstructure of the sintered samples, it could be observed that the MoS$_2$ will dissipate uniformly and the original particle size is reduced because of its layered structure which creates the lubrication film during friction.

Further to verify the presence of incorporation of particles with the composite materials obtained through compacted and sintered process, EDAX analysis has been accompanied along with the SEM investigation. Figures 5(a)–(d) expresses the EDAX analysis of the fabricated Al-4% Mg composite and Al-4% Mg-(2, 4 and 6) wt% MoS$_2$ composite materials. Figure 5(a) has reckoned that larger concentration peak signifies aluminium and average concentration peak denotes magnesium material. Figures 5(b)–(d) estimates that higher peak implies aluminium, normal peak designates magnesium and low peak entitles molybdenum and sulphide materials.

### 3.2. Micro-hardness

The micro hardness results of the Al-4% Mg-MoS$_2$ composite materials are exhibited in figure 6. MoS$_2$ augments the hardness when compared to the Al-4% Mg composite as shown in figure 6. Furthermore, the interfacial bonding of MoS$_2$ particles with the Al matrix material augmented due to the densification of composite materials which amplifies the hardness. Figure 6 also states that the hardness value intensifies, with the increasing wt% of MoS$_2$.

### 3.3. Effect of process parameters on wear loss

#### 3.3.1. Effect of applied load

The utmost significant parameter for wear loss is applied load for any material and at any condition.

Figure 7(a) illustrates the effect of applied load on wear loss for Al-Mg composite and Al-Mg-MoS$_2$ composites. From the figure, it is exploded that the wear loss increases with the augmenting applied load for all the materials. A large amount of material is removed because of the formation of extended plastic deformation and more cracks on the subsurface. Additionally, due to plastic deformation, deep grooves are formed parallel to the sliding direction. Moreover, high pressure is exerted on the surface of the material at higher load and it eliminates more material. Likewise, when the load increases, the hard asperity of the counter face material

| Sl. No | Applied load (N) | Sliding speed (m s$^{-1}$) | Sliding distance (m) |
|--------|-----------------|---------------------------|----------------------|
| 1.     | 5               | 0.5                       | 500                  |
| 2.     | 5               | 0.5                       | 750                  |
| 3.     | 5               | 0.5                       | 1000                 |
| 4.     | 5               | 2                         | 500                  |
| 5.     | 5               | 2                         | 750                  |
| 6.     | 5               | 2                         | 1000                 |
| 7.     | 5               | 3.5                       | 500                  |
| 8.     | 5               | 3.5                       | 750                  |
| 9.     | 5               | 3.5                       | 1000                 |
| 10.    | 15              | 0.5                       | 500                  |
| 11.    | 15              | 0.5                       | 750                  |
| 12.    | 15              | 0.5                       | 1000                 |
| 13.    | 15              | 2                         | 500                  |
| 14.    | 15              | 2                         | 750                  |
| 15.    | 15              | 2                         | 1000                 |
| 16.    | 15              | 3.5                       | 500                  |
| 17.    | 15              | 3.5                       | 750                  |
| 18.    | 15              | 3.5                       | 1000                 |
| 19.    | 25              | 0.5                       | 500                  |
| 20.    | 25              | 0.5                       | 750                  |
| 21.    | 25              | 0.5                       | 1000                 |
| 22.    | 25              | 2                         | 500                  |
| 23.    | 25              | 2                         | 750                  |
| 24.    | 25              | 2                         | 1000                 |
| 25.    | 25              | 3.5                       | 500                  |
| 26.    | 25              | 3.5                       | 750                  |
| 27.    | 25              | 3.5                       | 1000                 |
ploughs the softer surface of the pin deeper and thus, wear loss is intensified. At increased load, the rigid acerbity of the counter face material deeply pulls out the softer surface of the pin. Moreover, at increased load, the lubricated tribo-layer is destroyed due to the amplified pressure over the layer. It leads to the decrement of area and consequently, the wear loss is increased.

3.3.2. Effect of sliding speed
The effect of sliding speed on wear loss is cited in figure 7(b). It illustrates that the wear loss augments, as the sliding speed amplifies.

This result has occurred for all the samples. As the sliding speed upsurges, the pin surface becomes softer, due to the formation of high temperature at the boundary of the pin. The smoother surface is attained due to the formation of protective layer by amplifying the amount of counteract body elements in the transmit layer. This produced tribo layer exhibits higher hardness for Al-Mg-MoS$_2$ composites. Henceforth, the work strengthened film between the surface of the pin and the disc, diminishes the wear rate at higher sliding speed. Moreover, at
Figure 5. EDAX image of (a) Al-4%Mg composite material; (b) Al-4%Mg-2 wt% MoS$_2$ composite material; (c) Al-4%Mg-4 wt% MoS$_2$ composite material and (d) Al-4%Mg-6 wt% MoS$_2$ composite material.

Figure 6. Micro-hardness of composite materials.
greater sliding speed, the wear debris accumulated between the pin and the disc reduces the depth of penetration.

### 3.3.3. Effect of sliding distance

Figure 7(c) exhibits the effect of sliding distance on wear loss. The figure illustrates that the wear loss is amplified with augmenting sliding distance.

This may be attributed that at intensified sliding distance, the surface of the pin attains the plastic condition and intense distortion occurs. It leads to larger surface and subsurface destruction and consequently, wear loss is amplified. Moreover, at higher sliding distance, the developed tribo-layer gets instability, therefore wear loss is augmented. Furthermore, at higher sliding distance, the formation of lubricant layer is insufficient because of unavailability of solid lubricant on the surface contact. Moreover, the debris formed during the wear analysis between the sliding surfaces augments the ploughing of material from the pin surface and thus, wear loss is larger at amplified sliding distance.

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**Figure 7.** (a) Effect of applied load on wear loss. (b) Effect of sliding speed on wear loss. (c) Effect of sliding distance on wear loss.
3.4. Effect of process parameters on coefficient of friction

3.4.1. Effect of applied load

The influence of load is destined in Figure 8(a) for all the materials. From the figure it is clear that the COF is amplified with the increasing load in the range of 5–25 N for Al-Mg composite and Al-Mg-MoS2 composites.

This shows that at increased load, more plastic deformation is occurred. It affects the workpiece surface and conversely surges the contact of Mg particles with the surface of matrix material. Moreover, the material from the pin transfers to the disc, due to the scrubbing dispute of the ruptured reinforcement. Furthermore, more heat is generated at higher applied load and thus, the COF gradually increases, as the applied load increases. Likewise, the COF is also diminished with the increasing wt% of MoS2 and hence the hardness property of the materials is amplified in the composite materials.

3.4.2. Effect of sliding speed

The discrepancy of coefficient of friction with sliding speed is shown in Figure 8(b). It is observed that the coefficient of friction is augmented with intensifying sliding speed for all the materials under the value of 0.5–3.5 m s⁻¹.

Figure 8. (a) Effect of applied load on coefficient of friction. (b) Effect of sliding speed on coefficient of friction. (c) Effect of sliding distance on coefficient of friction.
This attributes that the MoS2 present in the composite materials are squeezed out on the surfaces and they form a lubricant layer which is formed in the contact surface. This lubricant layer is present only for a shorter period due to the augmented sliding speed. Then, the lubricant layer is detached from the interaction surface and it is enforced away from the contact surface. Moreover, the COF decreases with the increasing wt% of MoS2 reinforcement. This happens due to the formation of tribo layer on the surface contact. This tribo layer strongly influences the wear behavior of the composite materials at sliding speed.

3.4.3. Effect of sliding distance

Figure 8(c) displays the consequence of sliding distance on coefficient of friction between the range of 500–1000 m for all the materials. From the figure, it is exemplified that the coefficient of friction is increased with the increase in sliding distance. The frictional force is more for longer sliding distances, may be due to the instability of the tribo-layer at contact surface. Also, it is not possible to form lubricant layer at higher sliding distance due to insufficient of MoS2 particles on the pin surface.
3.5. Worn surface analysis

Figures 9(a)–(d) displays the worn surface of the pin materials such as Al-Mg composite, Al-Mg-MoS2 composites under the conditions of sliding distance of 1000 m, sliding speed of 3.5 m s⁻¹ and load of 25 N. Figure 9(a) shows that the parallel lines and shear deformation are formed with micro cracks along the direction of the wear on the pin surface. Moreover, permanent deepest grooves are formed with grain pullouts. This indicates that more wear loss occurs on the Al-Mg composite. This happens due to the characterization of plastic deformation which has occurred on the pin surface with ploughing effects.

The worn surface analysis of the Al-4%Mg-2 wt% MoS2 composite material is displayed in figure 9(b). The figure illustrates that while sliding the composite pin, the MoS2 are wiped on the pin surface and it forms a tinny layer which defends the surface from wear loss. Figure 9(c) exhibits the worn surface analysis of the Al-Mg/4 wt% MoS2 composite material. It elucidates that a thicker delamination has occurred on the pin surface with fractured oxide layer. Moreover, by the addition of MoS2, the wear of the composite material is converted from delamination to the adhesive wear.

The worn surface analysis of Al-Mg/6 wt% MoS2 composite material is showed in figure 9(d). From the figure it, could be understood that micro pits and debris particles are accumulated on the worn surface. Moreover, an extensive oxide layer is formed on the pin surface and it results in the formation of lubricant layer which protects the surface from wear. Hence, there is an absence of plastic deformation in the worn surface compared to the worn surface of the Al-Mg composite. From this investigation, it could be concluded that the wear is reduced with the augmenting wt% of MoS2. It results in the formation of tribo-layer and thus, the wear loss diminishes.

3.6. Wear debris analysis

The debris particles are engendered between the sliding surfaces and they separate the pin surface and the disc. The wear debris is examined for the sliding distance of 1000 m, sliding speed of 3.5 m s⁻¹ and load of 25 N for Al-Mg composite and Al-Mg reinforced with 2,4,6 wt% of MoS2 composites as deliberated in figures 9(a)–(d). This wear debris examination is handled by SEM for all the materials. Figure 10(a) exhibits the obtained wear debris particles of Al-Mg material. From the figure, it could be noted that a thin sheet is formed on the pin surface. This shows that the noteworthy plastic deformation is attained on the Al-Mg material. Figure 10(b) reveals the attained wear debris particles of Al-Mg-2 wt% MoS2 composite material. It illustrates that the formation of wear debris particles is smaller compared to Al-Mg composite because the Mg are smeared on the matrix material and they minimize the wear particles.

Likewise, the wear debris of the Al-Mg-4 wt% MoS2 and Al-Mg-6 wt% MoS2 composite materials are deliberated in figures 10(c) and (d). From these figures, it is understood that the size of the debris particles is diminished, when the wt% of MoS2 is increased. This shows that the MoS2 has a solid lubricant property which augments the difficulty of micro cutting consequences. However, a combination of excellent and rough debris of Al-Mg-(2,4&6) wt% of MoS2 materials is presented on the pin surfaces. This is attributed to the presence of MoS2 particles. The Al-Mg-6 wt% MoS2 composite material has very tiny debris particles than the other composite materials such as Al-Mg-2 wt% MoS2 and Al-Mg-4 wt% MoS2, because more effective solid lubricant property has been exhibited in the composite material and it has the highest wt% of MoS2. Moreover, abrasive wear is produced in the Al-Mg-6 wt% MoS2 composite material with a result of smaller grooves on the worn surface and it produces tinny debris particles.

4. Conclusions

The preparation of Al-4% Mg composite and Al-4% Mg-MoS2 composite materials by varying the wt% of MoS2 has been successfully accomplished through powder metallurgy way. The prepared materials are subjected to the analysis of wear performance using pin-on-disc machine and the succeeding conclusions are derived.

1. The SEM analysis ensures that the MoS2 particulates are reliably distributed with the matrix (Al–4% Mg) material and it has been accomplished by compaction and sintering (PM) methods.
2. The materials are extremely ploughed from the softer pin surface to the hard asperity of the counter disc material and thus, wear loss is amplified.
3. Likewise, the developed tribo-layer and the lubrication layer get instability on the pin surface at higher sliding distance and hence, the wear loss is augmented.
4. The lubrication is formed on the composite pin surface due to the creation of widespread oxidation layer and the worn is prevented. Moreover, the plastic deformation on the worn surface is absent in the MoS$_2$ reinforced composite materials compared to the unreinforced Al-Mg composite.

5. The size of the wear debris is diminished with the increasing wt% of MoS$_2$ particulates because of the exhibition of solid lubricant property in the composite material.

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