EXAMINE THE OPTICAL AND DRY SLIDING WEAR CHARACTERISTICS OF ALUMINUM METAL MATRIX COMPOSITES REINFORCED WITH ILMENITE/GR/SN

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SUMMARY

The composite was created by adding a lubricating agent (tin, graphite, or both) and 10% of ilmenite (particle size range of 20–120 µm). To manufacture this composite material, stir casting—a low-cost method was used. Furthermore, three weight percent of solid lubricants (tin, graphite, or both) were added in order to study their effects on solid lubrication. Ilmenite particles were uniformly dispersed throughout the Al-matrix during optical microscope examination. The refinement of primary silicon when ilmenite reinforcement was added. Comparing the composite samples to the base alloy, LM30, wear analysis showed that the addition of ilmenite increased the samples resistance to wear. The results of the investigation showed that the use of solid lubricants in aluminum metal composites reduced material losses by 21% to 41%. According to wear testing, the use of two solid lubricants demonstrated even greater wear resistance, ranging from 46% to 56%.

KEYWORDS

Hypereutectic Al-alloy, Ilmenite, Optical microstructure, Wear rate

1. INTRODUCTION

High-strength aluminum alloys and aluminum matrix composites (AMCs) are crucial lightweight materials widely employed in industries such as aerospace, automotive, and marine (Bhowmik, Dey and Biswas, 2019; Narayana, Benal and Shivanand, 2021). Despite their significance, the broad acceptance of high-performance AMCs trails behind that of traditional materials like Al, and Ti alloy. Challenges arise from the competitive advancements in the matrix and, alongside constant efforts to reduce costs (Chaturvedi et al., 2022; Nandy, Fortunato, and Martins, 2022; Sharma, Sharma, and Kumar Saraswat, 2023). Recent research has focused on enhancing AMCs through micro-scale reinforcements (Rana, Purohit, and Das, 2012). Moreover, upcoming advancements in AMC technology are expected to prioritize cost-effectiveness, leveraging techniques such as direct and near-net molding, including additive manufacturing (AM) (Wen et al., 2020; Liu et al., 2021, 2023; Shao et al., 2021; Li et al., 2022; Wu et al., 2022; Fu et al., 2023; Xu et al., 2023; Zhang et al., 2023). While AMCs have found applications in various sectors over the years, they still face several issues. Challenges in the field include the uneven distribution of reinforcements within the Al alloy matrix. The propensity for reinforcements to snap at stress concentrations is another significant challenge (Kumar et al., 2019; Kumar et al., 2020; Kumar et al., 2021). These problems are further exacerbated by the low large-scale-making ability for reinforcements and the more costs associated with new material research and development. These issues can be partially mitigated through mechanical methods (stirring) during the casting process, though they do not offer comprehensive solutions. Therefore, novel materials and design concepts are necessary to address these challenges and enhance AMC's strength. Several studies have been conducted to evaluate the impact of sea beach materials on the wear parameters of Aluminum Matrix Composites, revealing promising enhancements in dry sliding wear characteristics (Singhal & Pandey, 2021b, 2021c, 2022; Singhal & Prakash Pandey, 2022a). Singh et al. (Singh et al., 2002) demonstrated improvements in wear and thermal properties brought about by Al_2SiO_5 reinforcement of the LM6 alloy. Arora et al. (Arora et al., 2016) varied the rutile amount in the LM13 matrix to optimize the wear rate on the EN31 disc. Investigators have also explored the addition of lubricating agents alongside reinforcements to mitigate wear loss. Commonly utilized solid lubricants (Gr, Sn, Pb, and MoS2) have found application in vehicle contexts to reduce friction and increase wear resistance (Omrani et al., 2016; Kumar, Sharma, and Dixit, 2020, 2023; Al-Muntaser et al., 2022; Saravanan et al., 2023). These alterations in AMC composition contribute to cost savings in maintenance and auxiliary components. Building upon our prior research (Singhal and Prakash Pandey, 2022a), the current study extends the inquiry by reinforcing LM30 Al alloy with a broad range of ilmenite particle sizes and incorporating 3 wt. % Sn. Gr and both as a lubricating agent to optimize wear performance. Stir casting was employed in the manufacturing process.

2. EXPERIMENTAL

2.1 MATERIALS AND PROCEDURE

LM30 aluminum serves as the matrix, while ilmenite ($FeTiO_3$) acts as the reinforcement for the production of composite. Previous research (Singhal & Prakash Pandey, 2022a) has reported the configuration of both LM30 Al and ilmenite. To reduce friction and wear between two sliding surfaces, solid lubricants such as tin (Sn) and graphite (Gr) were introduced into the melt. Due to low wettability, graphite agglomerates because it cannot establish an interface with reinforcement or aluminum. Therefore, an optimal quantity of graphite (3 wt. %) was selected. In addition, the melt included 3 wt. % of tin. At 627°C, the highest solubility of tin in aluminum is observed, constituting 0.1 weight %. Conversely, the solubility decreases to its lowest point of 0.05 weight % at the eutectic point of 228°C. Additionally, this solubility further diminishes at room temperature. Despite being immiscible with aluminum, a small amount of tin (3 wt. %) was added to provide solid lubrication. At room temperature, tin distributes along the grain boundary, enhancing lubricating properties (Gudić et al., 2010; Kaur & Pandey, 2013). More tin is released at the contact surface as the pin slides, and this additional tin is spread due to the increased load caused on by heat generated by friction while the pin is sliding continuously. Hence, an optimal tin quantity is crucial to maintain a continuous lubricated layer. Table 1 outlines the necessary steps for the fabrication of the composite. To make lubricated composite samples, a same procedure was followed, with Sn, Gr and both (3 wt. %) after adding the ilmenite reinforcement (10 wt. %) to the melt. The molten material was transferred into a mold, from which 8mm (cylindrical shape) samples were extracted from the central portion of the fabricated AMC brick, following ASTM standards. This procedure aimed to maintain uniformity in microstructural properties among all samples. Table 2 represents the designed fabricated samples.

Table 1. Steps for fabricating the composite

Step	Description
Melting	Material-LM30 Al alloy
	Weight-800 gm
	Melting Temperature-750°C
	Container- Graphite Crucible
	Stirring- 630 rpm for 6 min using a graphite Stirrer
	Furnace- Electric
Adition of Rein-forcement	Material- Preheated Ilmenite particles
	Lubricants- Gr and Sn
	Preheating Temperature-400 °C
	Stirring- 630 rpm for 8 min using a graphite Stirrer

Table 2. Characteristics of composite samples prepared with LM30 Al

Sample name	Matrix + Reinforcement (wt. %) + solid lubrication (wt.%)
3IG10	LM30+10 wt.% $FeTiO_3$+ 3 wt. % graphite
3IT10	LM30+10 wt.% $FeTiO_3$+ 3 wt. % tin
3ITG10	LM30+10 wt.% $FeTiO_3$+ 1.5 wt. % graphite+1.5 wt.% tin

2.2 CHARACTERIZATION

Following ASTM standard E3-11, specimens underwent microscopic analysis using the Eclipse MA-100 system from Tokyo, Japan, to assess the dispersion of reinforcement within the LM30 Al. Subsequently, wear tests were conducted using the pin-on-disc (POD) procedure employing the TR-20 model from Ducom Instruments in Bangalore, India. This system used 830 HV hardened EN31 die steel as the counter surface. The tests were carried out up to a sliding distance of 3000 m at a sliding velocity of 1.6 m/sec under various loading situations (range from 9.81 N to 68.67 N). The wear rate was calculated using prior study findings (Singhal & Pandey, 2021b; Singhal & Prakash Pandey, 2022b).

3. RESULTS AND DISCUSSION

3.1 OPTICAL MICROSCOPY

The microscopic examination displayed in Figure 1 showcases the microstructures of 3IG10, 3IT10, and 3ITG10 AMCs. Previous research has highlighted that primary Si tends to segregate in LM30 alloy, whereas the AMCs studied here demonstrate a uniform dispersion of primary Si within the matrix. Additionally, refinement of the α-Si was detected (Panwar & Pandey, 2013). Ilmenite, characterized by a lower coefficient of thermal expansion (CTE) compared to the Al-alloy, functioned as a heat sink, resulting in Si reorganization within the microstructure of the AMCs. Consequently, $FeTiO_3$ reinforcement acted as nucleation sites in the melted LM30 Al, initiating nucleation of the Si phase on the exterior of the $FeTiO_3$ reinforcement (Gupta et

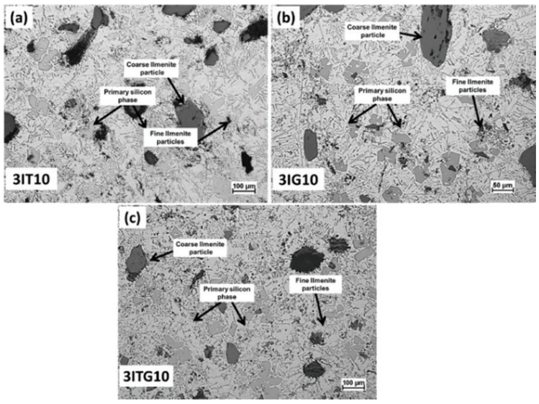

Figure 1. Optical microscopic analysis of (a) 3IG10, (b) 3IT10, and (c) 3ITG10 composites

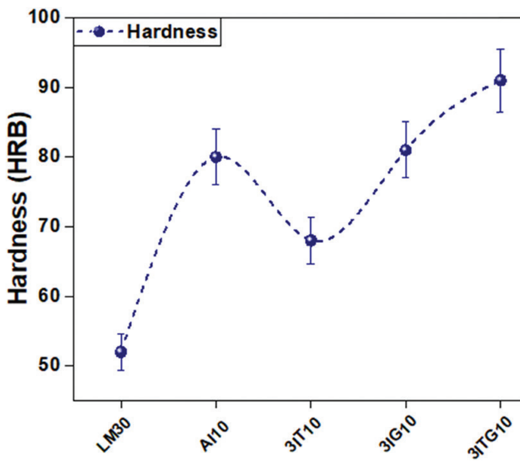

Figure 2. Bulk hardness values of synthesized composite

al., 2023; M. Sharma et al., 2023; Singhal et al., 2022). The presence of silicon accumulation around the exterior of the $FeTiO_3$ reinforcement is observable, as depicted in Figure 1(a-c). Furthermore, the addition of 3 wt. % Sn lubricating agent contributed to the refinement of the α-Si phase. Due to its low melting point, tin readily occupies surface pores as it moves across a counter (Abis et al., 1994). Tin atoms, with their substantial atomic size, exhibit a notable binding energy with vacancies. This characteristic facilitates the diffusion procedure, contributing significantly to the global refinement of the Al-Si eutectic mixture within the matrix. (Sofyan et al., 2005). Additionally, particles of graphite (Gr; 3 weight percent) act as an additional nucleation site, aiding in the Si phase refining.

3.2 HARDNESS

Figure 2 presents the bulk hardness values for both the LM30 Al and the composite material. Analysis of the graph suggests that the inclusion of $FeTiO_3$ reinforcement leads to a rise in the hardness of the AMCs sample. Additionally, the hardness of the 3IG10 specimen declines due to the incorporation of a soft Gr lubricating agent. Comparing the 3ITG10 composite to the other composites, there is a significant 51% improvement in hardness. This enhancement can be attributed to the refinement of the microstructure.

3.3 DRY SLIDING WEAR INVESTIGATION

3.3.1 OUTCOME OF SLIDING DISTANCE

The investigation into the wear performance of $FeTiO_3$ reinforced AMCs with 3wt.% solid lubricants was conducted under various applied loads ranging from 9.81 to 68.67N, with a sliding distance of 3000 m, as depicted in Figure 3. The wear rate versus sliding distance plot delineates two distinct regions: the run-in wear zone (RW) and the steady-state wear zone (SSW). Initially, a peak in wear loss occurred within the first 250 m of sliding, attributed to the samples inability to withstand abrupt shear stress, resulting in instability. Subsequently, wear loss diminished up to 1250 m for 3IT10, 3IG10, and 3ITG10 composites, as depicted in Figure 3a-c. The softer surface of the AMCs pin experienced a ploughing act and graze as the roughness on the EN31 disc (counter surface) breached during the initial phase. The AMCs pin surface developed abrasive grooves as a result of the pin asperities deforming due to the continuous motion. Because $FeTiO_3$ reinforcement and a Gr or Sn lubricants were added, the RW for both 3IT10 and 3IG10 was found to be higher than that of 3ITG10 under all applied load. This reinforcement served as load-carrying components in AMC samples, mitigating dry sliding wear loss. Moreover, the continuous sliding motion between the sample and the steel disc promoted oxidation of the sample interaction surface. This led to the creation of an oxide layer that reduced interaction between the pin and the disc, effectively halting additional wear. Furthermore, at 1250 m (for 3IT10 and 3IG10 and 3ITG10), the wear rate stabilized, marking the onset of SSW for the composite. The expansion and deformation of the oxide layer remained uniform despite changes in applied load and sliding motion.

3.3.2 INFLUENCE OF $FeTiO_3$, Gr AND Sn

In Figure 3 (a-c), the wear performance of the composites is observed in response to the presence of $FeTiO_3$, Gr, and Sn. The robust ilmenite particles bear a substantial interaction load, protecting the matrix against deformation during sliding against the counter surface. Moreover, ilmenite reinforcement, when present at an equivalent weight fraction, exhibits wider coverage within the metallic matrix with a uniform distribution, thereby amplifying load-carrying points and diminishing inter-particle spacing.

To mitigate wear losses, the incorporation of ilmenite reinforcement boosted the pin strength, consequently reducing plastic distortion of the sample interaction surface

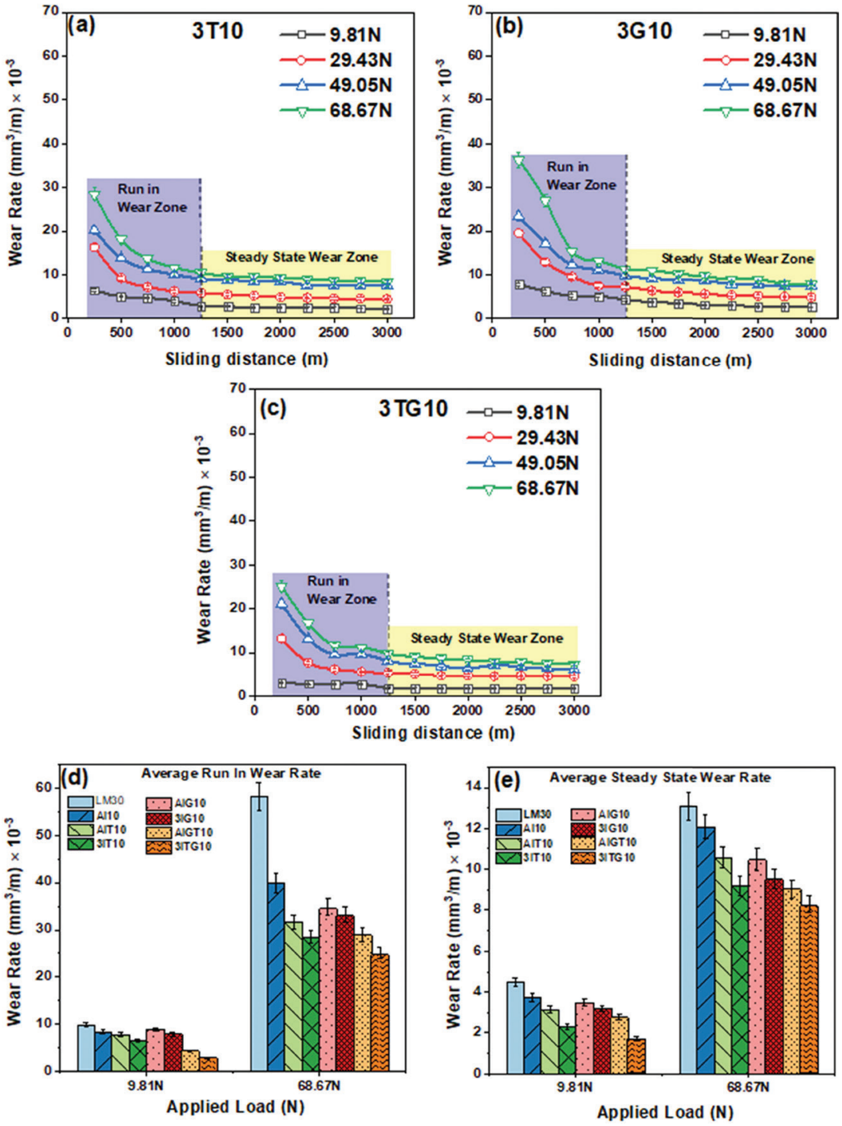

Figure 3. Wear examination of (a) 3IT10, (b) 3IG10, (c) 3ITG10, and (d, e) Comparative data analysis with previous study (Singhal & Prakash Pandey, 2022a) and current work at 9.81 and 68.67N load.

and lowering material loss rates in the AMC samples (Doddamani et al., 2017). The wear loss of AMCs was further diminished through the addition of 3 wt. % Gr and Sn lubricants both along with 10 wt.% ilmenite reinforcement. Tin, has a low melting point and is soft, mostly served as a lubricating material by forming at the Al-Alloy grain boundaries. Gr, characterized by its hexagonal shape, also functioned as a solid lubricant. Consequently, a tribo-layer induced by the solid lubricant was formed during the wear examination (Doddamani et al., 2017; Mahmoud, 2008). During motion, the LM30 Al facilitated the formation of a lubricating layer between the sample and the counter surface, as the sample surface became visible. This lubricating coating offered an anti-friction surface, subsequently reducing the material loss (Kaur & Pandey, 2013).

3.3.3 AMCS SAMPLE COMPARATIVE WEAR ANALYSIS

Comparative wear studies of the basic alloy (LM30), previously made composites (AI10, AIT10, AIG10, AIGT10), and current study samples (3IT10, 3IG10, and 3ITG10) composite at 9.81 N and 68.67 N, are shown in Figure 3(d, e). Notably, in comparison to the previously generated samples (AIT10, AIG10, and AIGT10), the wear resistance of samples 3IT10, 3IG10, and 3ITG10 is improved by up to 6% (at 9.81N) and 9% (at 68.67N) with the increase in solid lubricant content. With regard to wear resistance, sample 3ITG10 AMC stands out, showing improvements of 72% at 9.81N and 50% at 68.67N.

4. SEM-EDS ANALYSIS OF WORN SURFACE

To comprehend the wear mechanism, we conducted the SEM-EDS study of the worn surface of the 3ITG10 AMC under 9.81 N and 68.67 N applied load, as depicted in Figure 4 (a-b). At the 9.81 N load, the grooves along the sliding motion with minimal surface delamination (Figure 4a) show that abrasive wear phenomena predominantly caused wear loss under this condition. Additionally, during relative motion, a lubricating layer was observed on the interacted surface, effectively preventing sample and disc contact and protecting the surface from abrasion (Kumar, Pandey, et al., 2013; Kumar, Panwar, et al., 2013; Sharma et al., 2012). The smoother surface of sample 3IG10 suggests superior wear resistance compared to the unlubricated synthesized composite.

As the load increased to 68.67 N, the rise in contact temperature due to sliding motion resulted in increased shear stress and substantial plastic deformation. This led to the formation of extensive grooves and cracks on the worn track (Figure 4b). The cracks originated along the worn surface and propagated until they reached the subsurface, leading to the loss of material in large debries from the surface, a process known as delamination wear. As a result, significant wear losses have been detected at maximum load, suggesting an extreme wear operation. However, the occurrence of a lubricating layer mitigated the transition from mild to severe wear and prohibited the breakage of the layer from the worn surface (Figure 4b).

The EDX study of the synthesized composite worn surface at 9.81 N load and 68.67 N load conditions in Figure 4 (a-b) reveals the existence of several components (Al, Si, Cu, Fe, O, etc.). At 9.81 N load, Al, Si, and O elements predominate with small amounts of Fe, Ti, and Cu on the worn surface. At 68.67 N load, the concentration of Fe and O increases due to the elevated flash temperature (Singhal & Pandey, 2021a). Moreover, the interaction area of ilmenite particles with the steel counter surface increases, leading to more iron transfer and the formation of a mixed

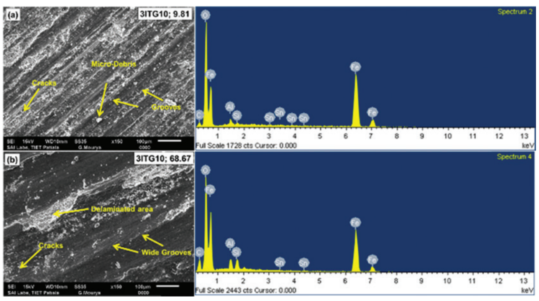

Figure 4. SEM-EDS analysis of fabricated composite 3ITG10 at (a) 9.81 N and (b) 68.67 N applied load

lubrication layer (MML) on the subsurface, enhancing the wear resistance of synthesized composites. The presence of oxides and iron is more pronounced in the 3ITG10 sample, resulting in increased wear resistance of the composite.

5. CONCLUSIONS

Stir casting technology was used to create self-lubricating, cost-effective, and environmentally acceptable Aluminum Matrix Composites (AMCs). Optical microscopy and dry slide wear analysis were utilized to assess the manufactured AMCs wear characteristics. The following is a summary of the study's primary experimental findings:

- The AMCs homogeneous dispersion of ilmenite minerals and refined primary silicon shape were seen by optical microscopy. lubricants (Sn and Gr), were also dispersed equally throughout the LM30 Al. Furthermore, both primary and eutectic silicon morphology were refined as a result of the addition of reinforcement.
- The ilmenite reinforcement improved the hardness of the fabricated composite.
- The wear loss of the AMCs decreased with the addition of solid lubricants. The composites that contained both Sn and Gr showed the best wear results.
- In comparison to AIT10, AIG10, and AIGT10, the wear resistance of 3IT10, 3IG10, and 3ITG10 AMCs was increased by up to 6% (at 9.81N) and 9% (at 68.67N) by increasing the amount of solid lubricant. With sillimanite reinforcement and both tin and graphite (at 3wt% each as a solid lubricant), the composites showed the highest wear performance, with wear reductions of 72% at 9.81N and 50% at 68.67N.
- An SEM study revealed that the main mechanisms causing wear losses were adhesion, delamination, and abrasive wear.

6. FUTURE SCOPE

The current study delved into the dry sliding wear characteristics of composites reinforced with natural minerals and solid lubricants at room temperature. Moving forward, the scope of future research could encompass an exploration of the wear and friction behavior of the prepared AMCs under elevated temperatures.

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