

Influences of Ag+LiAlO₂ on mechanical and high temperature wear behavior of magnesium hybrid composites

T. Suresh^{a,*}, P. Suresh^b, M. Prabu^c,

^aAssistant professor, Kumaraguru College of Technology, Tamilnadu, India

^bProfessor, Sona College of Technology, Tamilnadu, India

^cAssociate Professor, K.S.R College of Engineering, Tamilnadu, India

The objective of this research is to analyze the mechanical such as porosity; hardness and high temperature wear behavior of Magnesium (MgZE41A) hybrid composites reinforced with Silver (Ag) and Lithium Aluminate (LiAlO₂). The various wt. % such as Ag (1.0 & 1.5) and LiAlO₂ (1.0 & 1.5) was reinforced in MgZE41A using Powder Metallurgy (P/M) technique. The sintered composites were characterized using Scanning Electron Microscope (SEM) coupled with Energy Dispersive Spectroscopy (EDS) line mapping Technique. The porosity and Vicker's hardness of the samples were assessed. Further, the high temperature wear behavior of the composites was evaluated using tribometer with heater setup by varying the heat inputs such as 30°C, 50°C, 100°C, 150°C and 200°C. The results revealed that increasing the wt. % of Ag and LiAlO₂ led to decrease the porosity from 0.11 % to 0.09 % due to high compacting pressure and sintering temperature. High temperature Wear rate of the composites was reduced from $2.15 * 10^{-4}$ to $0.83 * 10^{-4}$ because of wt. % of reinforcement, high compacting pressure and better sintering temperature.

(Received March 22, 2022; Accepted July 4, 2022)

Keywords: MgZE41A, Ag, LiAlO₂, Porosity, Hardness and high temperature wear

1. Introduction

Pure magnesium and Magnesium alloys having a low density and better compatibility with various ceramics reinforcement. Magnesium density is around two thirds of aluminium and one fifth of steel. Though, magnesium and its alloys anticipated high specific strength, amongst conventional engineering alloys [1-3].

Many magnesium alloys are there such as AZ31, AZ61, AZ91, ZE41A etc. MgZE41A alloy is having higher amount of Zn, Ce and Zr alloying element in Mg. It provides better mechanical bonding and to increase the mechanical and tribological properties. However, Zn and Cr provides better corrosion resistance to Mg. Due to its sole properties, Mg is used in various light weight applications, such as sports items, fabrication of artificial legs etc. In general, degradation products of magnesium and its alloys are completely soluble and nontoxic to human body. In spite of this positive effects of magnesium and its alloys Mg are degradation and uses its on bone therapeutic systems. Moreover, these types of degradation of magnesium resulted to reduce the mechanical stability and following to flexible of bone tissue healings.

Many reinforcements are there such as SiC, W, WC, BN etc. [4-6]. Researchers are doing research in hybrid magnesium composites because of new material development and industrial requirements. The application of magnesium and its composites are minimum because of its poor creep resistance at high temperature conditions. Furthermore, it has low strength, low modulus of rigidity and poor wear resistance. Besides, magnesium having low corrosion resistance, and heavy galvanic and pitting corrosion are dominated and reduces the applications. In order to reduce corrosion reaction on magnesium alloy, there are many protective methods to be followed such as hybrid composite preparation, cladding, anodizing and usage of inhibitor's [7]. Size of the reinforcement particle is also act as a crucial role for fabrication of good composites. Micron sized ceramics particles creates micro-cracks during sintering in terms of voids, which generally reduces

* Corresponding author: sureshmcpd@gmail.com

<https://doi.org/10.15251/DJNB.2022.173.731>

the strength and ductility of the composites. These cracks reduce the mechanical and tribological properties such as wear and corrosion. Instead of micron sized particles, sub-micron or nano sized particles provides better properties.

Silver (Ag) which is having high density (10.49 g/cm^3) provides better strength to the magnesium. However, silver nano particles are having excellent antibacterial properties and also it had low toxic to the humans. Incorporation of Ag with magnesium provides notable increase in corrosion resistance of magnesium. LiAlO_2 having moderate density (2.62 g/cm^3) provides better ductility to the magnesium. Fabrication technique is one of the key factors for distribution of reinforcement in magnesium. P/M is one of the proved methods for even dispersion, less wastage of reinforcement, ease to fabricate the net shape. P/M having unique nature for tailor made material fabrication. So, the processing of composites cost efficient, plays a vital role while expanding the views of applications. Magnesium matrix composites by P/M show significant improvement in mechanical properties because of poor wettability between the molten matrix materials and solid reinforcement and strong metal-reinforcement interface. However, these types of fabrication technique provide the modification in microstructure of magnesium matrix in terms of grain refinement, particle destruction and distribution. After powder metallurgy technique, secondary processing methods adopted to enhance the mechanical properties in terms of reduction of internal voids. After this secondary processing, the microstructure would be refined significantly. However, during this technique, the grain sizes are reduced and the reinforcement particles are fragment and re-distributed in the magnesium matrix. Many researches are under gone research on magnesium hybrid composites preparation, following to that analyze the material characterization [8-10].

Therefore, the present work is intended to fabricated porosity free magnesium (MgZE41A) matrix composites reinforced with Silver (Ag) and Lithium Aluminate (LiAlO_2). The various wt. % such as Ag (1.0 & 1.5) and LiAlO_2 (1.0 & 1.5) was reinforced in MgZE41A using Powder Metallurgy (P/M) technique. The sintered composites were characterized using Scanning Electron Microscope (SEM) coupled with Energy Dispersive Spectroscopy (EDS) line mapping Technique. The porosity and Vicker's hardness of the samples were assessed. The high temperature wear behavior of the composites was evaluated using tribometer with heater setup by varying the heat inputs such as 30°C , 50°C , 100°C , 150°C and 200°C .

2. Experimental Procedure

High purity, magnesium with the particle size of less than $44 \mu\text{m}$, silver and (Ag) and Lithium Aluminate (LiAlO_2) are the particle size of less than $10 \mu\text{m}$ are purchased from M/s. Sigma Aldrich, Germany. These powders are mixed with ball mill with various wt. %, namely 100Mg, 99Mg (MgZE41A) +1Ag, 98Mg (MgZE41A) +1Ag+1 LiAlO_2 , 97.5Mg (MgZE41A) +1.5Ag+1 LiAlO_2 and 97Mg (MgZE41A) +1.5Ag+1.5 LiAlO_2 . The predetermined powders were taken in a stainless vial with stainless steel balls. Ball to powder ratio was taken as 5:1. Since, magnesium having less melting point, hence all the mixing process was in argon atmosphere with toluene as used a wetting agent. Consentingly, the mixing was performed for 15 minutes for homogeneous distribution of secondary particles in the matrix. After mixing, the composite powders were compressed using, compression testing machine with various compaction pressure such as 100 MPa, 200 MPa and 300 MPa respectively. After compaction, the composites were sintered using muffle furnace with varying the sintering temperature such as 300°C , 400°C and 500°C respectively.

Microstructural characterization of the fabricated composites was analyzed using SEM with EDS line mapping. For the effectiveness of reinforcement in the magnesium matrix on mechanical and tribological behavior was deliberate by conducting density, porosity, hardness and high temperature wear analysis. Vicker's microhardness with the test load of 0.5 kg with the dwell time of 10 s. Density following to that porosity of the composites was evaluated using Archimed's Principle. For ensuring, the reproducibility of data accuracy, five different surfaces were indented.

High temperature wear analysis of the composites was assessed using Pin-on-Disc tribometer with heater setup. Constantly, the heat was supplied to the composites, till the experiment was finished. Henceforth, the even heat input was supplied to the composites. In this study, five different temperatures such as 30°C , 50°C , 100°C , 150°C and 200°C was taken.

Following to that one of the conventional process parameter applied load (10 N, 20 N, 30 N, 40 N and 50 N) was varied.

3. Results and Discussion

3.1. Microstructural stability with Elemental analysis

Microstructural analysis offers a governing quality control as well as a significant analytical tool for the evaluation of grain structures, size and distribution of the particles was observed using SEM. Before performing, the microstructural analysis, the composite surface was polished using hand polishing technique. In-order to get a fine polished surface, the composites were furthered polished using 1 μm diamond paste suspended in distilled water. Fig. 1 (a-e) shows the SE (Secondary Electron) microstructure of the fabricated composites. Here, Ag and LiAlO_2 are found irregular in shape and it could bond comfortably with the magnesium matrix. In all the microstructure images, the secondary particles such as Ag and LiAlO_2 are evenly distributed in the matrix (Refer fig. 1 (b - d)).

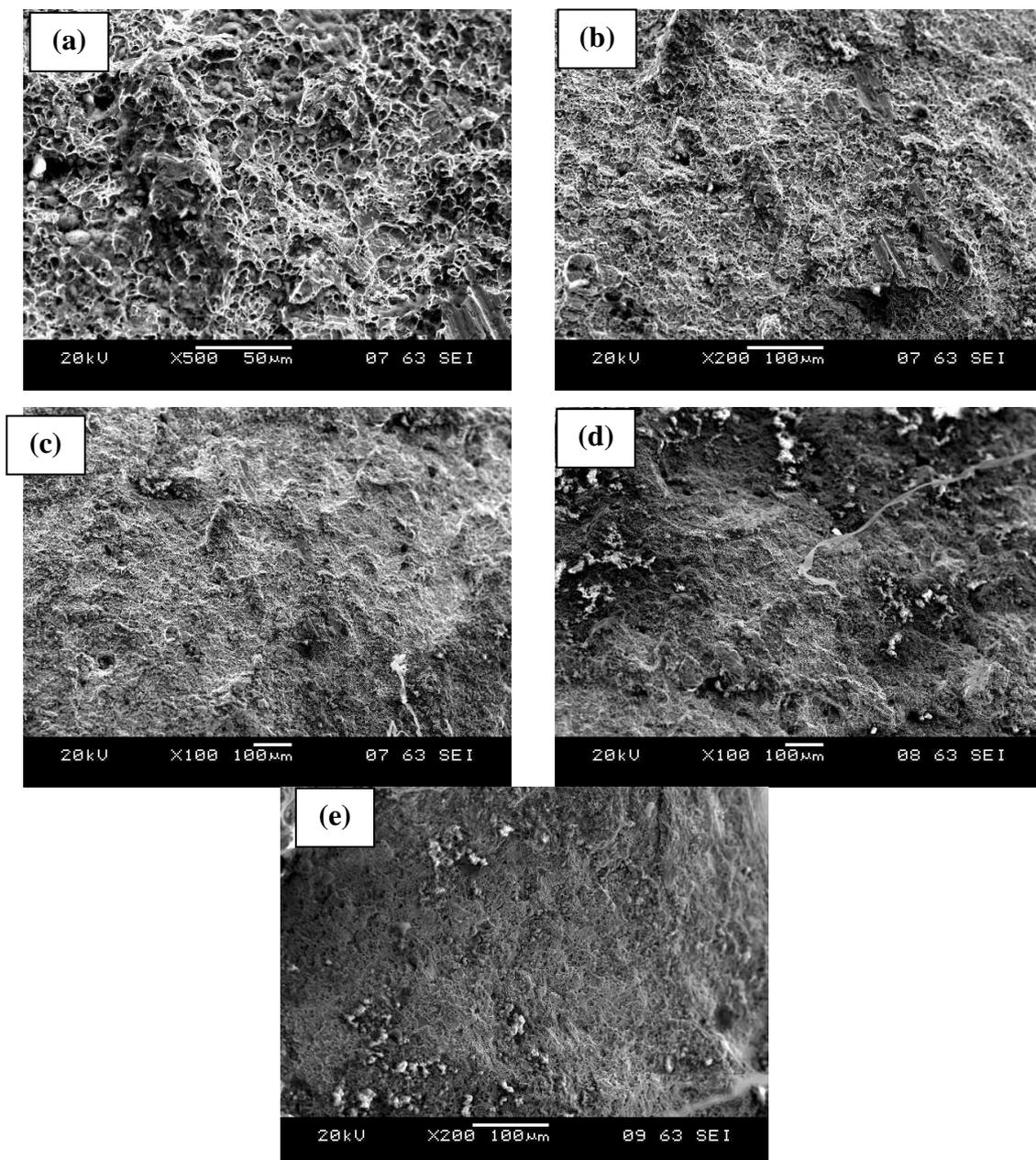


Fig. 1. Sintered Magnesium composites SE images such as a) 100Mg, b) 99Mg+1Ag, c) 98Mg+1Ag+1LiAlO₂, d) 97.5Mg+1.5Ag+1LiAlO₂ and e) 97Mg+1.5Ag+1.5 LiAlO₂.

However, some clusters, voids and defect zones are identified in fig. 1 (b-d). It was happening because of lack of bonding between the matrix and reinforcement during sintering. Increasing the sintering temperature from 400°C to 500°C, the thermal decomposition of matrix and reinforcement was enhanced. As per the sintering mechanism, increasing the sintering temperature will enhance the contact angle between the matrix and reinforcement [11-12]. Fig. 1 (e) shows the 1.5Ag+1.5 LiAlO₂ particles are distributed in the 97Mg (MgZE41A). Here more amount of proper distribution is viewed on the composite surface. However, some of the dendrite is also viewed in fig. 1 (e). The formation of dendrite is happening because of higher sintering temperature and it led to increase the mechanical and tribological properties.

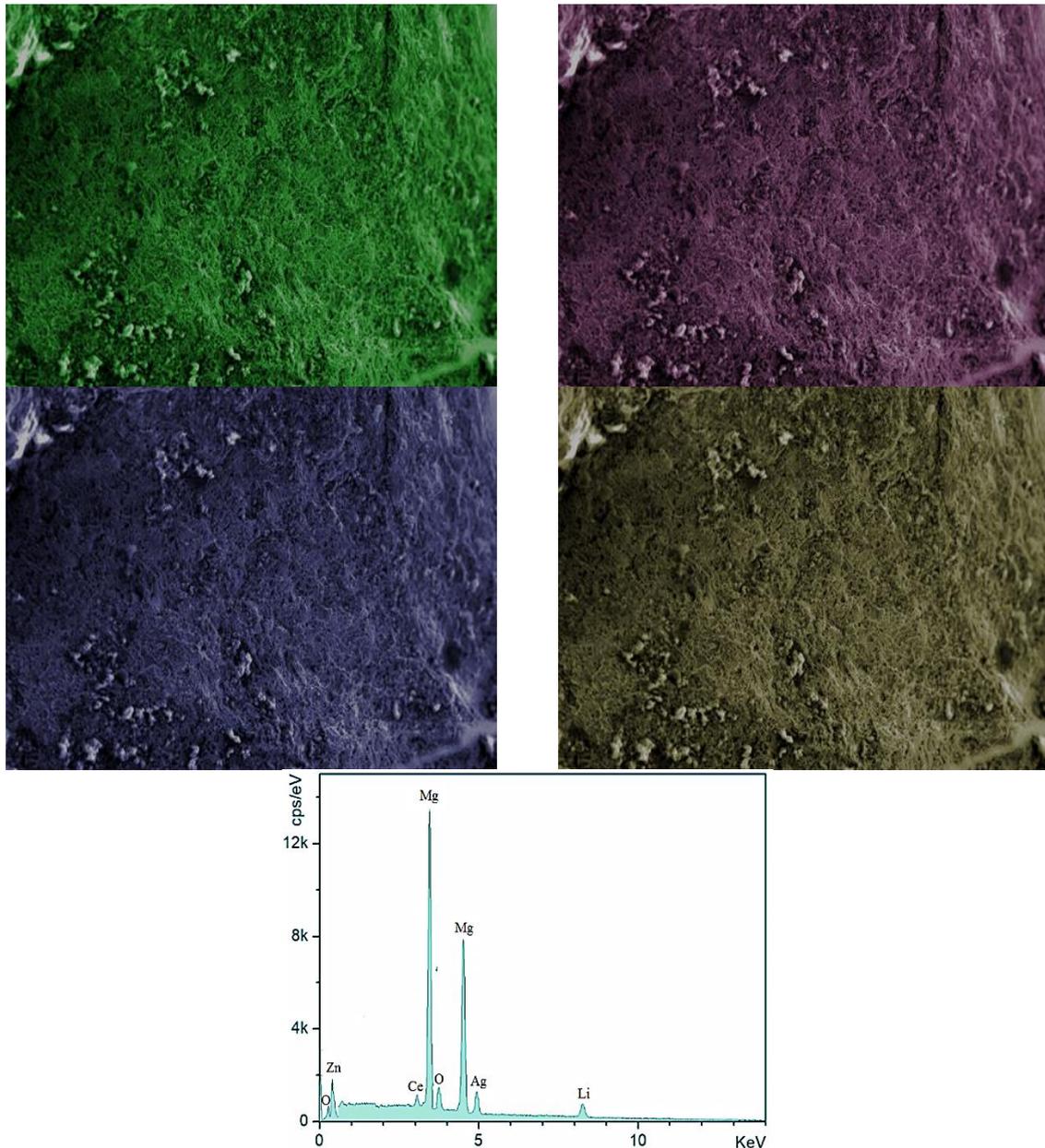


Fig. 2. (a) EDS Elemental Line Mapping of 99Mg (MgZE41A) +1Ag.

The elemental confirmation is not yet defined with SEM. Henceforth, EDS line mapping was essential for verifying the elemental confirmation of the fabricated composites and it displayed in fig. 2 (a and b). Fig. 2 shows the elemental line mapping of 99Mg (MgZE41A) +1Ag and 97Mg (MgZE41A) +1.5Ag+1.5 LiAlO₂. In fig. 2 (a) four shaded colors such as green, pink,

violet and yellow represents the Mg, Zn, Ce and Ag. It also confirms by the EDS analysis. The intensity of magnesium, zinc and cerium is high when compare to Ag. It illustrates that the presence of magnesium, zinc and cerium is high compared to Ag. Six shaded colors are viewed in fig. 2 (b) namely green, pink, violet yellow, orange, and light green. It seems that Mg, Zn, Ce, Ag, Li and Al. The same is confirmed by the EDAX analysis.

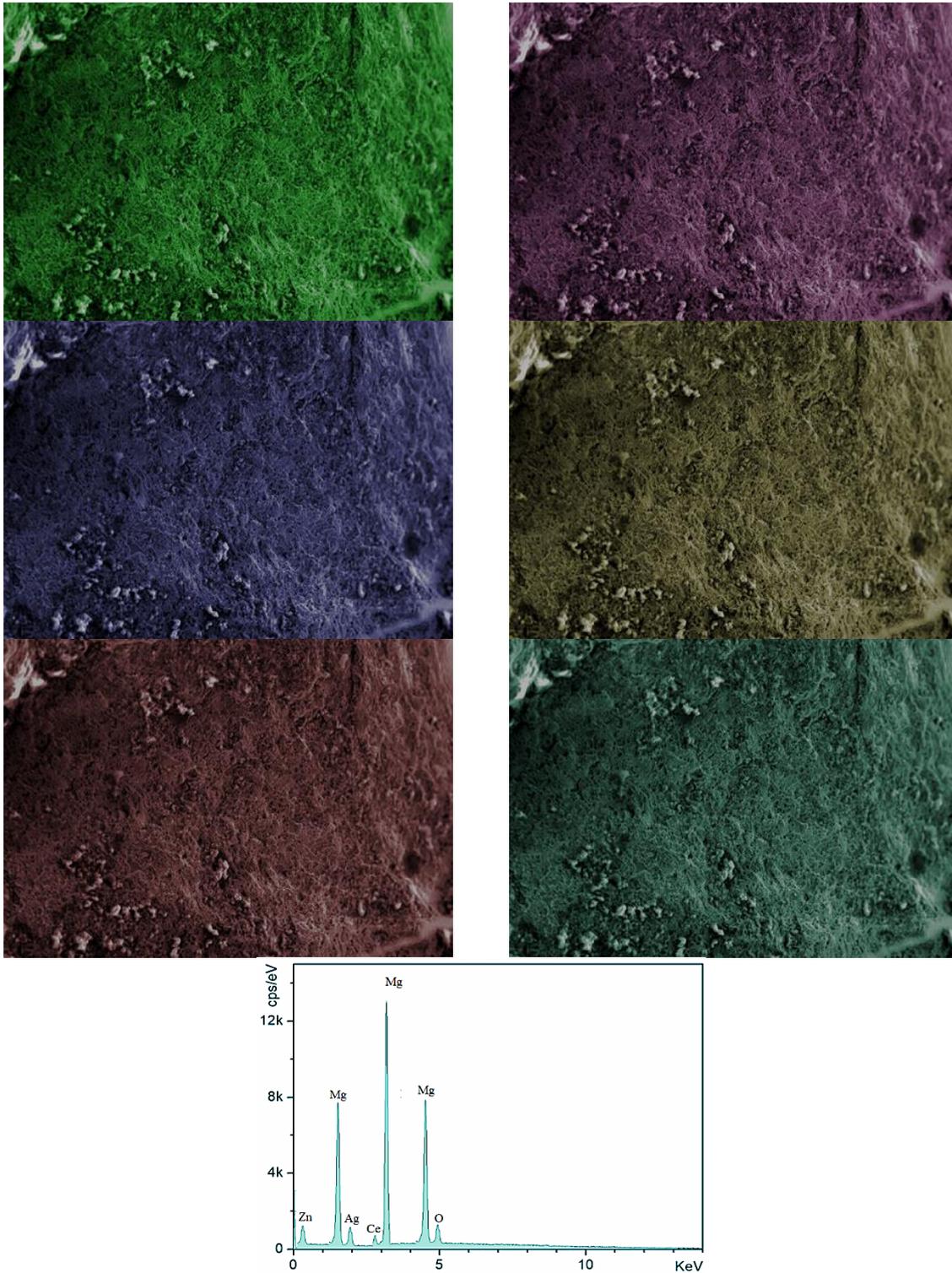


Fig. 2 (b) EDS Elemental Line Mapping of $97\text{Mg} (\text{MgZE41A}) + 1.5\text{Ag} + 1.5 \text{LiAlO}_2$

3.2. Density and Porosity

Fig. 3 (a, a1, b and b1) shows the relative density and porosity of the fabricated composites with various compaction pressure and sintering temperature. Increasing the compaction pressure and sintering temperature led to increase the relative density and reduce the porosity of irrespective of composites. Increasing the compaction pressure, makes the composites tougher, resulted to increase in relative density following to that enhance the mechanical and tribological properties [5]. While increasing the compacting pressure from 100 MPa to 300 MPa, the interface or bonding between the matrix and reinforcement was increased. It also acts as barrier for grain movement dislocation and make a pinning action of hard reinforcement. However, increasing the sintering temperature from 300°C to 500°C, the grain nucleation and grain growth are taken place in a proper manner according to grain boundary strengthening mechanism. In other hand, increasing the sintering temperature creates effective cold welding between the adjacent particles such as Mg, Ag and LiAlO_2 . Henceforth, effective diffusion mechanism was taken place and forms a good bonding/ interface between the matrix and reinforcements [6-8]. It could increases the hardness of the composites from 60 HV to 72 HV.

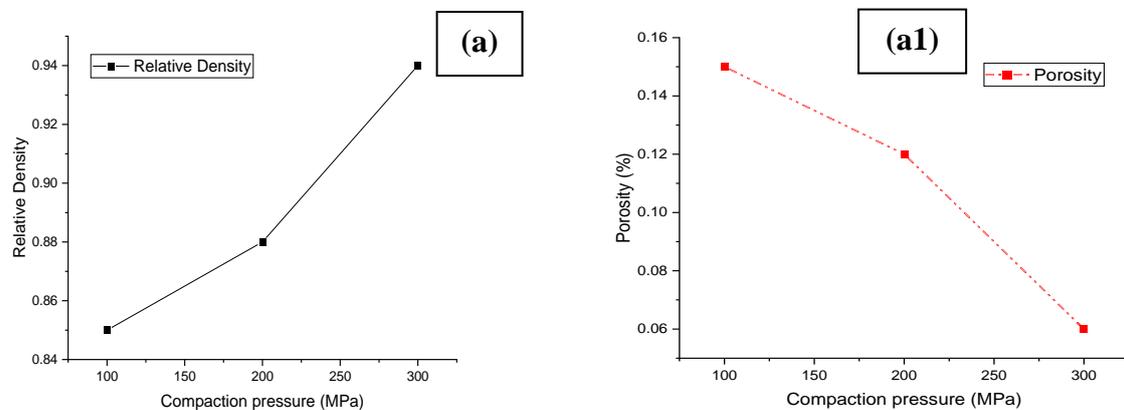


Fig. 3. (a and a1) Variation of relative density and Porosity with respect to compaction pressure.

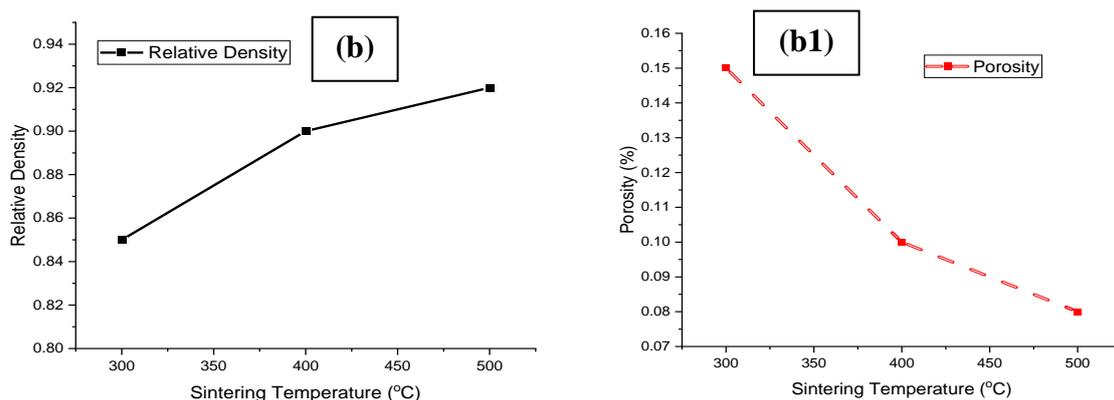


Fig. 3. (b and b1) Variation of relative density and Porosity with respect to sintering temperature.

3.3. High temperature wear evaluation

Fig. 4 shows the high temperature wear performance of fabricated composites with various wear temperatures. For the high temperature wear performance higher compacting pressure (300 MPa) and higher sintering temperature (500°C) samples was taken and analyzed. The distinguish variations are noticed at initial stage and it could linearly stabilize till the end of the experiment at all temperatures. In this process, thermal softening was happened in the composites and it creates

significant impact on the performance of wear. After material softening, recrystallization resistance was happening and it as plays vital role in the experimentation. In this study five different temperature such as 30°C, 50°C, 100°C, 150°C and 200°C and five different loads 10N, 20N, 30N, 40N and 50N are taken. For the better understanding of wear performance higher load 50 N was taken and detail observation was made on the specimen at all temperatures. According to fig. 4 is concern, increasing the temperature will lead to increase the wear rate / wear performance because of during higher temperature the inter-atomic molecules are get soften and its ease to get exhausted. This behavior can be attributed to the breaking of the composite molecules and settle down on the sample surface [10-15]. The highest wear rate was found around in between of $2.15 \times 10^{-4} \text{ mm}^3/\text{m}$ for 200°C sample temperature and it suggestively reduces for hybrid composites. According to the concern, of wt. % of the reinforcement 97Mg+1.5Ag+1.5 LiAlO₂ shows less wear rate and seem that $0.8 \times 10^{-4} \text{ mm}^3/\text{m}$. This clearly evident that composite 1.5Ag+1.5 LiAlO₂ provides excellent anti-wear properties. When the wear temperature increases to 200°C the wear rate gradually increases for the longer time period and it could be reaches stable state with an average value of $1.32 \times 10^{-4} \text{ mm}^3/\text{m}$. This may closely relate to the composite high temperature softening effect. During the temperature and environment is concern, oxide layer during sliding is occurred at high temperature regime [16-20].

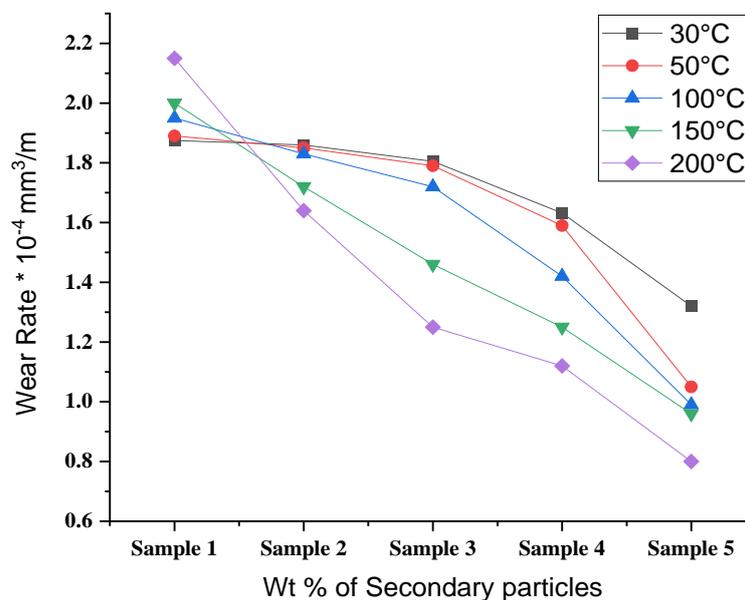


Fig. 4. High temperature wear performance.

3.4. Wear mechanism

The wear morphology of worn surfaces of the composites under 50 N at various temperatures is displayed in fig. 5 (a-e). The dominant wear mechanism of all the temperatures is an oxidative wear at 30°C, 50°C, 100°C, 150°C and 200°C. Fig. 5 (a) presented flat and some course visible material peeling was observed. While increasing the temperature from 30°C to 50°C smooth material peeling was observed with some oxide tribo layers (Refer fig. 5(b)). Noticeably some of the scattered debris and pitting grooves are observed along the sliding direction. It increases the wear rate (Refer fig. 5(c)). Fig. 5 (d) shows the wear morphology of 150°C and it reveals that severe plastic deformation was observed on adjacent to the sliding direction [21-25]. This could be articulated to the presence of oxygen while sliding at elevated temperatures. This feature represents the prime wear mechanism are abrasive wear at elevated temperature. This may create the micro-cutting of the composites and resulted that ploughing of material which shows like grooves and debris. While increasing further temperature from 150°C to 200°C shallow grooves are observed on the local area of the wear tracks and it creates rough surfaces and it displayed in fig. 5 (e). In very specific, some of the wear debris are adheres to the tribofilm on the

surface at high oxygen concentrations. In other hand, it could articulate that reduce the micro-cutting effect. However, during the longer period of sliding, larger size debris accumulate and compact on the surface and it forms the tribofilm [26-30]. These types of characteristics illustrates that the dominant wear mechanism at 200°C are abrasive and adhesive, although some sliding oxidation wear also exists.

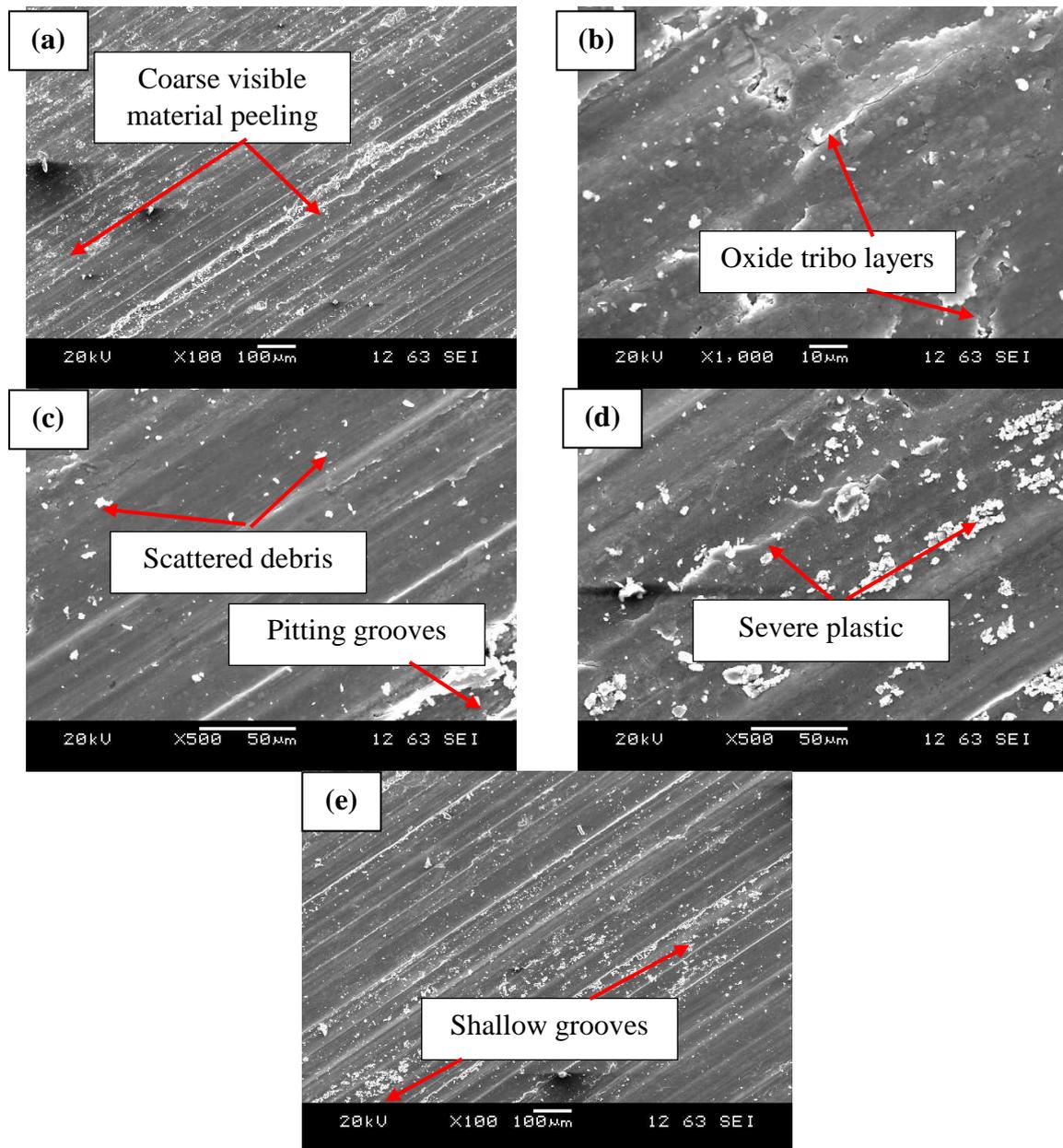


Fig. 5. Worn surfaces of the composites under 50 N at various temperatures a) 30°C, b) 50°C, c) 100°C, d) 150°C and e) 200°C.

4. Conclusion

High temperature wear performance was evaluated for Mg hybrid composites with various wt. % of reinforcement such as Ag and LiAlO_2 with various compaction pressure and sintering temperature. Further the samples were analyzed for high temperature wear behavior for various temperature such as 30°C, 50°C, 100°C, 150°C and 200°C. From the experimentation, the following conclusion was derived.

Increasing the compaction pressure and sintering temperature will led to increase the relative density of the composites from 0.85 to 0.94 and 0.85 to 0.92. Increasing the compaction pressure and sintering temperature will led to decrease the porosity of the composites from 0.15 to 0.06 and 0.15 to 0.08.

The high temperature wear resistance of 97Mg+1.5Ag+1.5 LiAlO₂ was significantly better than other composites because of its admirable microstructure stability. This was happening for all 97Mg+1.5Ag+1.5 LiAlO₂ composites irrespective of compaction pressure and sintering temperature. At 200°C the dominant wear mechanisms are abrasive and adhesive, although some sliding oxidation wear also exists.

References

- [1] An, L. Y., Chang, C. G., Meng, L. C., Peng, J. H., Yuan, J., Wang, Z., & Kang, D. S. (2022). Digest Journal of Nanomaterials & Biostructures (DJNB), 17(1); <https://doi.org/10.15251/DJNB.2022.171.89>
- [2] Xia ZHOU, Zi-fan LIU, Feng SU, Ya-fu FAN (2021), Transactions of Nonferrous Metals Society of China, 31, 636-647; [https://doi.org/10.1016/S1003-6326\(21\)65525-5](https://doi.org/10.1016/S1003-6326(21)65525-5)
- [3] Mohd Rafiq Parray, Noor Zaman Khan, Annayath Maqbool (2021), Materials Today: Proceedings, 46, 6507-6512; <https://doi.org/10.1016/j.matpr.2021.03.694>
- [4] S. Jayabharathy, P. Mathiazhagan (2020), Materials Today: Proceedings, 27, 2394-2397; <https://doi.org/10.1016/j.matpr.2019.09.142>
- [5] Wahaaj Ali, Ali Mehboob, Min-Gu Han, Seung-Hwan Chang (2020), Composite Structures, 245, 112322; <https://doi.org/10.1016/j.compstruct.2020.112322>
- [6] Ehsan Ghasali, Yasin Orooji, Masoud Alizadeh, Touradj Ebadzadeh (2020), Materials Science and Engineering: A, 789, 139662; <https://doi.org/10.1016/j.msea.2020.139662>
- [7] Pubo Li, Wanting Tan, Mangmang Gao, Keren Shi (2021), Journal of Alloys and Compounds, 858, 157673; <https://doi.org/10.1016/j.jallcom.2020.157673>
- [8] Song-Jeng Huang, Murugan Subramani, Chao-Ching Chiang (2021), Composites Communications, 25, 100772; <https://doi.org/10.1016/j.coco.2021.100772>
- [9] M. Kasaeian-Naeini, M. Sedighi, R. Hashemi (2021); <https://doi.org/10.1016/j.jma.2021.11.006>
- [10] M. Tacikowski, P. Kobus, J. Kamiński, B. Kucharska, K. Kulikowski, P. Marchlewski, Marcin Pisarek (2019), Vacuum, 160, 325-332; <https://doi.org/10.1016/j.vacuum.2018.11.044>
- [11] Sanjay Sharma, Amit Handa, Sahib Sartaj Singh, Deepak Verma (2019), Journal of Magnesium and Alloys, 7, 487-500; <https://doi.org/10.1016/j.jma.2019.07.001>
- [12] Junxiang Zhou, Luyang Ren, Xinyu Geng, Li Fang, Henry Hu (2019), Materials Science and Engineering: A, 740, 305-314; <https://doi.org/10.1016/j.msea.2017.10.070>
- [13] R. Sivabalana, K. R. Thangaduraib, K. Lenin (2021), Journal of Ceramic Processing Research, 22(6): 605-614.
- [14] Mahdi Heidari Bagherabadi, Rahim Naghizadeh, Hamid reza Rezaie, Mohsen Fallah Vostakola (2018), Journal of Ceramic Processing Research, 19(3): 218-223.
- [15] C.K.L. Jeffrey, S. Ramesh, C.Y. Tan, W.D. Teng (2016), " Journal of Ceramic Processing Research, 17(6): 620-625.
- [16] W.C. Vittayakorn, D. Bunjong, R. Muanghlua, N. Vittayakorn (2011), Journal of Ceramic Processing Research, 12(5): 493-495.
- [17] J. Umar Mohamed, PL. K. Palaniappan, P. Maran, R. Pandiyarajan (2021), Journal of Ceramic Processing Research, 22(3): 306-316.
- [18] T. Boyraz, A. Akkus (2021), Journal of Ceramic Processing Research, 22(2): 226-231.
- [19] Hoseok Nam, Ki-Woo Nam (2021), Journal of Ceramic Processing Research, 22(1): 66-73.
- [20] Muhammet Ceylana, İsmail Topcu (2020), Journal of Ceramic Processing Research, 21(5): 539-546.

- [21] Young-Min Byoun, Sun-Kyo Seo, Jin-doo Yoon, Sang-Jo Na (2019), Journal of Ceramic Processing Research, 20(5): 556-562; <https://doi.org/10.36410/jcpr.2019.20.5.556>
- [22] Agus Edy Pramono, Muhammad Zaki Nura, Johny Wahyuadi M. Soedarsono, Nanik Indayaningsih (2019), Journal of Ceramic Processing Research, 20(4): 333-346; <https://doi.org/10.36410/jcpr.2019.20.1.1>
- [23] Ahmet Akkus, Tahsin Boyraz (2018), "Investigation of wear properties of CaO, MgO added stabilized zirconia ceramics produced by different pressing methods", Journal of Ceramic Processing Research, 19(3): 249-252.
- [24] Hamza Simsir, Yasin Akgul (2022), Materials Chemistry and Physics, 281, 125886; <https://doi.org/10.1016/j.matchemphys.2022.125886>
- [25] O. Esmailzadeh, A.R. Eivani, M. Mehdizade, S.M.A. Boutorabi, S.M. Masoudpanah (2022), Journal of Alloys and Compounds, 164437; <https://doi.org/10.1016/j.jallcom.2022.164437>
- [26] Lingyun Wang, Tijun Chen, Pengpeng Pu (2022), Materials Science and Engineering: A, 830, 142307; <https://doi.org/10.1016/j.msea.2021.142307>
- [27] Rasoul Parichehr, Changiz Dehghanian, Aida Nikbakht (2021), Journal of Alloys and Compounds, 876, 159995; <https://doi.org/10.1016/j.jallcom.2021.159995>
- [28] Yakup Say, Omer Guler, Burak Dikici (2020), Materials Science and Engineering: A, 798, 139636; <https://doi.org/10.1016/j.msea.2020.139636>
- [29] Dengchuan Zhao, Guoqing Wu (2020), Materials Science and Engineering: A, 788, 139607; <https://doi.org/10.1016/j.msea.2020.139607>
- [30] Moara M. Castro, Shima Sabbaghianrad, Pedro Henrique R. Pereira, Eric M. Mazzer, Augusta Isaac, Terence G. Langdon, Roberto B. Figueiredo (2019), Journal of Alloys and Compounds, 804, 421-426; <https://doi.org/10.1016/j.jallcom.2019.07.007>