Mechanical properties of surface-modified magnesium alloy AZ61 with nanoparticles of aluminum oxide and titanium dioxide by friction stir processing

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Abstract

The present work investigates the mechanical properties of surface-modified magnesium alloy AZ61 reinforced with Al_2O_3 and TiO_2 nanoparticles by using the friction stir processing (FSP) technique. Surface-modified AZ61 alloys were fabricated by the addition of dufferent amount of Al_2O_3 and TiO_2 nanoparticles (5, 10, and 15 vol.%). The developed surface composites were studied regarding microstructure, revealing a uniform dispersion of the added nanoparticles, which resulted in improved mechanical properties of the obtained composites by FSP. The ultimate tensile strength, impact strength, and microhardness improved by 20, 45, and 67 % by reinforcing the alloy with nanoTiO_2 particles when compared to the as-cast alloy. The results of this study indicate that the reinforced AZ61 Mg alloy can be a potential material for applications in automobile sectors due to its high strength and lightweight components.

Keywords: Characterization; metallography; high-speed tool; nano-fillers.

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1. INTRODUCTION

Magnesium and its alloys exhibit a promising potential for usage in automobile, aerospace, and marine applications, attributed to their high strength-to-weight ratio and lightweight nature. Researchers have demonstrated that reinforcing by the addition of secondary particles to magnesium in the fabrication of Mg matrix composites significantly improves both grain refinement and mechanical properties. Currently, various techniques such as stir casting, spray deposition, in-situ fabrication, and powder metallurgy are utilized for composite fabrication. Mechanical stir casting was used to fabricate hybrid composites of AZ61 magnesium alloys reinforced with alumina (Al_2O_3: 2 wt.%) and silicon carbide (SiC) nanoparticles at various proportions showing that compressive, microhardness and tensile properties are directly related to SiC concentration so that maximal values were obtained for composites with 1 wt.% SiC [1]. Physical vapor deposition and chemical vapor deposition are used to generate thin films and coatings from 50 nm to a few microns to protect the materials and improve their properties. Still, these coatings cannot withstand significant mechanical loadings. [2]. Thermal spraying is an efficient technique to produce thick ceramic layers so it is the most preferred method. However, these coatings could be microporous with microcracks and show poor substrate adherence. Therefore, these methods often result in defects leading to increased costs and longer processing times. Therefore, to overcome these drawbacks, it is essential to develop an effecient technique for preparing defect-free high-quality surface composites. friction stir processing (FSP) is a solid-state processing method that employs principles of friction stir welding (FSW) to produce improved magnesium matrix composites, potentially resolving the limitations mentioned above [3]. By FSP, simultaneous homogenization, densification, and grain refinement of the microstructure can be achieved [4], resulting in improved mechanical properties of the materials. It has been widely reported that the

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FSP technique can be used to significantly refine the grain size and enhance the mechanical properties of magnesium alloys [5-7]. FSP was utilized to add various reinforcement particles like ZrC [8], B_4C [9], Si_3N_4 [10], TiC [11], 304 stainless steel powders, fly ash, Ti-6Al-4V, palm kernel shell ash [12], TiAlC [13] and $NiTi_p$ [14] to Mg alloys to improve alloy mechanical properties. The present research aimed to attempt the addition of nano-particles of Al_2O_3 and TiO_2 in the AZ61 Mg alloy by using this technique.

2. EXPERIMENTAL SETUP

The magnesium alloy AZ61 (Venuka Engineering Private Limited, India) with a chemical composition of 5.98 wt.% Al, 0.95 wt.% Zn, 0.001 wt.% Fe, and Mg in balance was chosen for this study with the dimensions of 150×50×10 mm and nanoparticles of Al_2O_3 (Nano Labs, India) and TiO_2 (Nano Labs, India) were employed as reinforcements. The powders were analyzed by scanning electron microscopy, (SEM) so that the average particle size was determined to be between 10 and 20 nm. In this application, the area of interest is a semicylindrical groove about 3 mm in diameter, which was drilled into the workpiece and filled with nanoparticles. The semicylindrical groove of was drilled in a zigzag manner along the length of the plate. FSP was performed by using a specially designed, non-consumable tool made of H13 (K.R. Engineering, Chennai, India) and an FSP machine (Fig. 1, Annamalai University, India) with binary elements namely a pin and a shoulder. The tool is made to revolve at the required speed, and by applying downward force to the tool, the pin penetrates the base material, and the shoulder just touches the surface of the material.

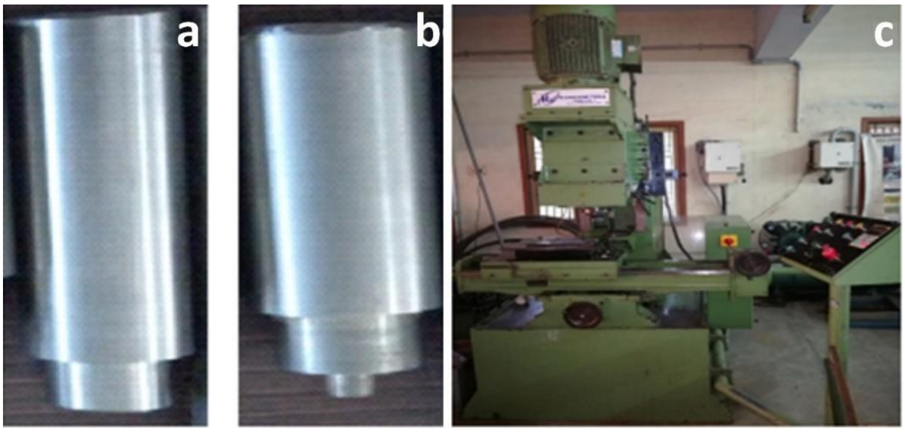

Figure 1. Photographs of the experimental friction stir processing (FSP) set-up: FSP tool (a) without the pin for specimen preparation, (b) with a cylindrical pin profile for processing, and (c) FSP machine

FSP was performed with the following parameters: tool rotation speed of 1000 rpm, travel speed of the tool of 10 mm/min, axial force of 4.5 kN, and the tilt angle of the tool of 2°. Surface-modified specimens at various powder contents (*i.e.* 5, 10 and 15 vol.%) are displayed in Figure 2.

a b

Figure 2. FSPed specimens with a) nano-Al_2O_3, b) nano-TiO_2

SEM micrographs were taken by a scanning electron microscope (Evo 18, Carel Zeiss, India) above the surface of the coated and intersection region to study the microstructure of the friction stir processed samples. FSP samples were cut into needed dimensions 1×1 cm via wire electrical discharge machining (Chmer CNC wire cut, Taiwan) to carry out the microscopy analysis. Samples were refined *via*SiC emery sheets, followed by polishing of the sample surfaces using diamond paste. Chromic acid was used as an etchant after polishing.

Along with morphological features, the modified alloy surfaces were tested and characterized for their improved mechanical properties. Tensile strength was determined by a computerized micro-tensile machine (Model FIE5000PF, Fuel Instruments and Engineers, India) as per the ASTM standard E-8. Sample dimensions 55×10×10 mm with a 45° notch used to carry out impact test (XJJU-5.5, Kystal Equipments, India) as per ASTM standard E-24 and the hardness of the surface modified specimen was measured by the Vicker's hardness tester (HDNS-Kelly Instruments, China) as per E-94 standard.

3. RESULTS AND DISCUSSION

3.1. Metallographic characterization

The surface morphology of AZ61 magnesium alloy samples reinforced with nano Al_2O_3powders at different contents produced by FSP was examined by using SEM (Figure 3). FSP is a suitable technique to distribute the reinforcement particulates uniformly in the stir region, in contrast to the stir casting process which results in non-uniform distribution of reinforcements because of density variation of matrix and reinforcing phases and solidification connected factors. Nevertheless, the FSP process parameters have to be adjusted to attain appropriate dispersal of particles. In this study, the traverse speed and number of passes, in addition to the percentage of nanoTiO_2 particulates, were varied in pursuit of uniform dispersal, *i.e.* the aim was to achieve the microstructure of the whole stir region with distributed particles.

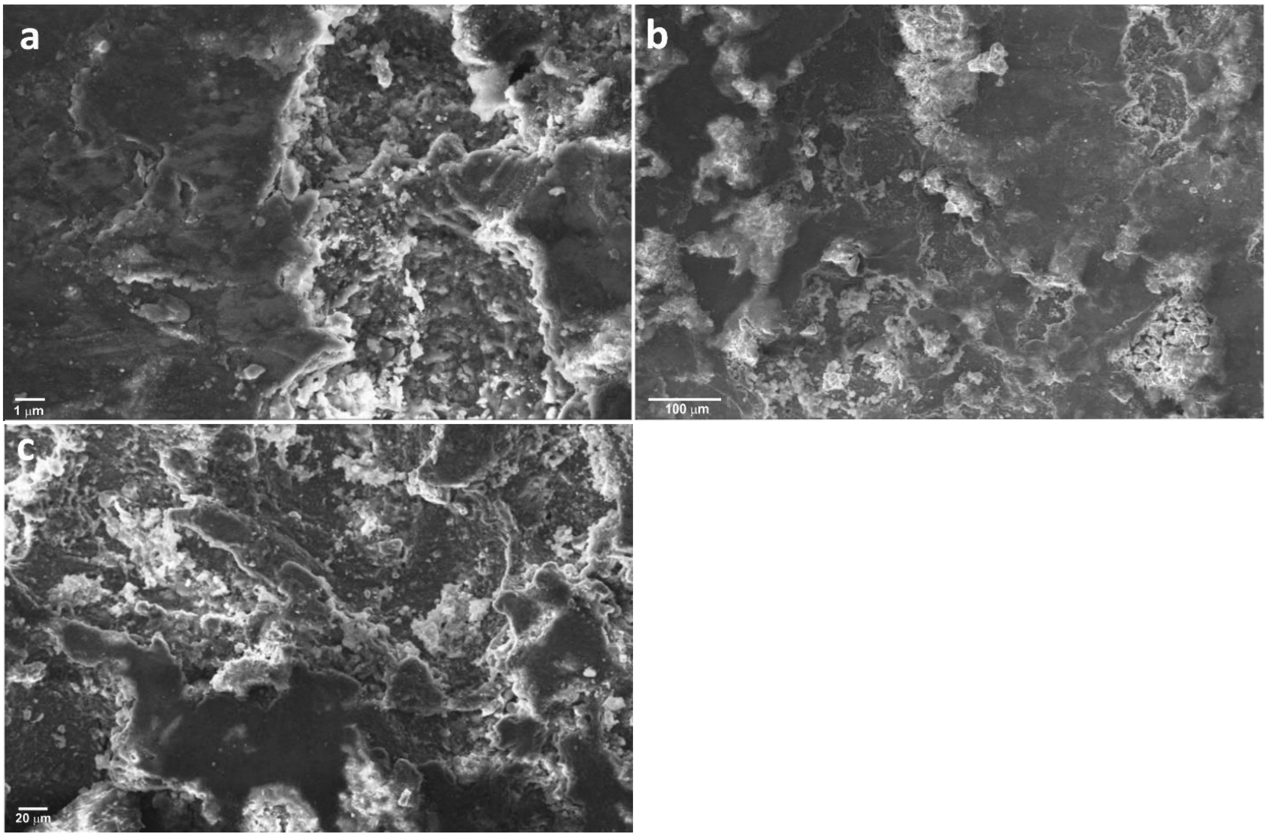

Figure 3. Microstructure of AZ61 Mg alloys reinforced with (a) 5 vol.% nanoAl_2O_3, (c) 10 vol.% nanoAl_2O_3 and (c) 15 vol.% nanoAl_2O_3

The microstructure is notably affected by two FSP factors: frictional heat and mechanical stirring. Frictional heat alters the grain size, thus enhancing the ease of plasticization. Nonetheless, it does not have a straight effect on the drive of particulates. Mechanical stirring distorts the heated materials and directly disturbs the drive of particulates. Traverse speed determines instantaneouslyboth thefrictional heat and mechanical stirring. The increase in traverse speed induces a decrease in both factors. Consequently, the dispersal became worse as the traverse speed increased despite the reduction in stirring and lower frictional heat, which lessened the grade of plasticization. It was observed that circulation improved at a traverse speed of 30 mm min^{-1}. The particulates couldn't touch altogether the areas of the stir region [15]. Figure 4 displays the friction stir processed nanoTiO_2 reinforced AZ 61 magnesium alloy.

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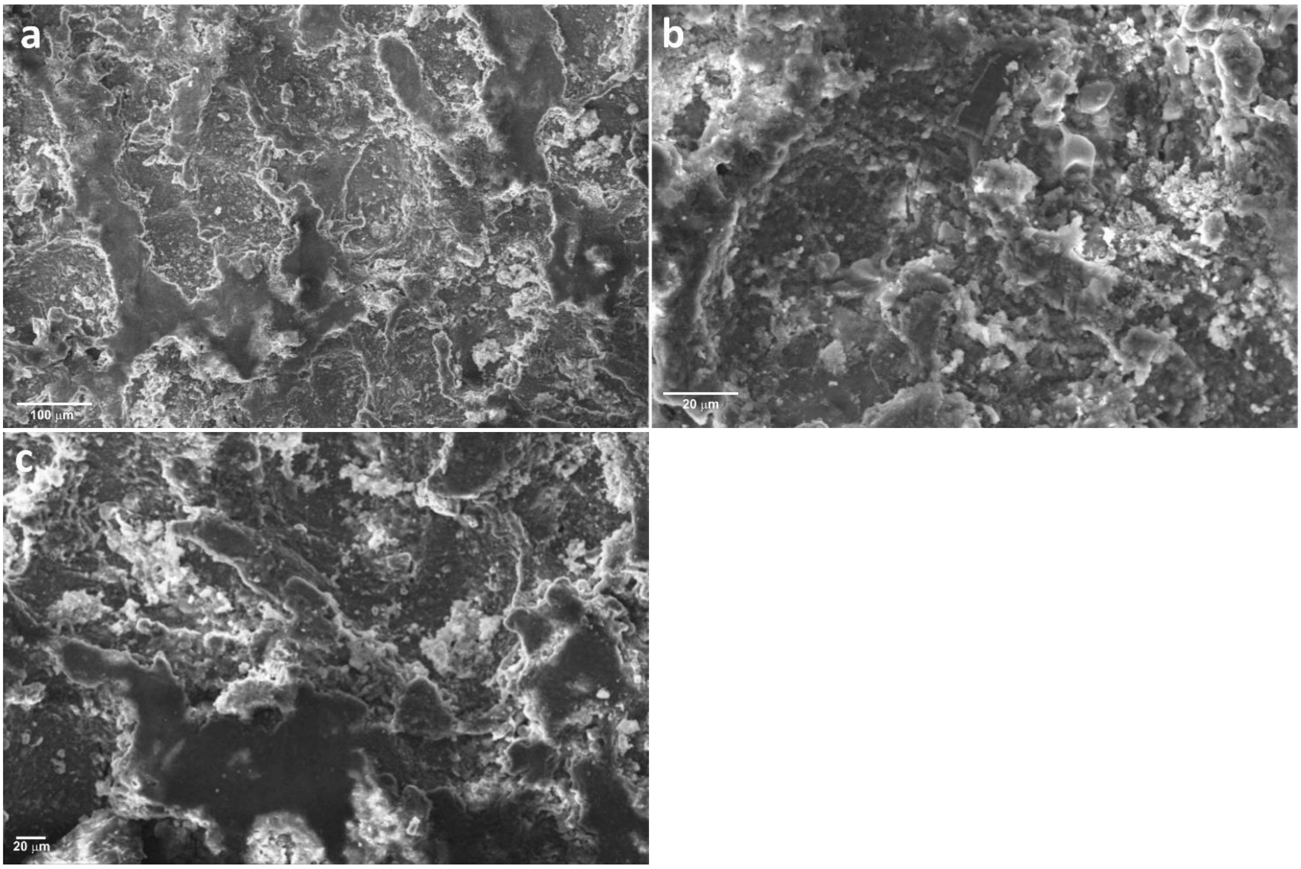

Figure 4. Microstructure of AZ61 Mg alloy reinforced with (a) 5 vol.% nanoTiO_2 (b) 10 vol.% nano TiO_2 and (c) 15 vol.% nanoTiO_2

3.2. Tensile strength of the surface-modified specimens

Ensuring the quality of the metal specifications' tensile properties is the most influential factor in selecting the material for engineering applications. Tensile tests were carried out on the friction stir processed AZ61 magnesium alloy strength-henedwith different proportions of nanoAl_2O_3 and nanoTiO_2 powders as well as the unmodified AZ61 magnesium alloy for comparison. It was found that nano-TiO_2 reinforced alloy possesses higher tensile strength (203 MPa) than unreinforced and nano-Al_2O_3 reinforced alloy, as shown in Figure 5.

The improvement in tensile strength was achieved with recrystallization and modification of grains, homogeneous distribution, and definite boundary. The tensile load is effectively moved to the particle to the matrix material, without any interfaces. Nano-TiO_2 reinforcement enhanced the plastic flow of the AZ61 Mg alloy. The crack surface of the alloy shows a system of modest pores, and nano-TiO_2 particles are seen on the crack surface. Fine voids appear on the fractured surface of the AZ61 with nano-TiO_2 particles being pulled out from the surface by debonding/fracture during the test. The results indicate that nanoAl_2O_3-reinforced AZ61 Mg alloys exhibit slightly lower tensile strength than that of nano-TiO_2-reinforced alloys. The major reason for this result could be that nano-TiO_2 particles are distributed more evenly in the alloy in addition to the higher strength of these particles.

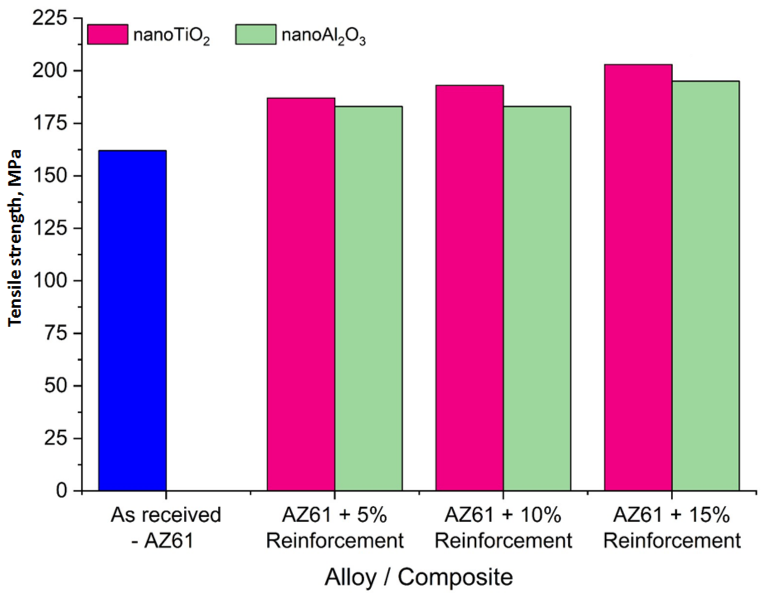

Figure 5. Tensile strengths of the non-modified AZ61 Mg alloy and reinforced with Al_2O_3 or TiO_2 nanoparticles at different concentrations

3.3. Impact test

Impact tests were performed to determine the toughness of the unreinforced AZ61 Mg alloy and those reinforced by nano-Al_2O_3/nano-TiO_2. The impact strength comparison is shown in Figure 6.

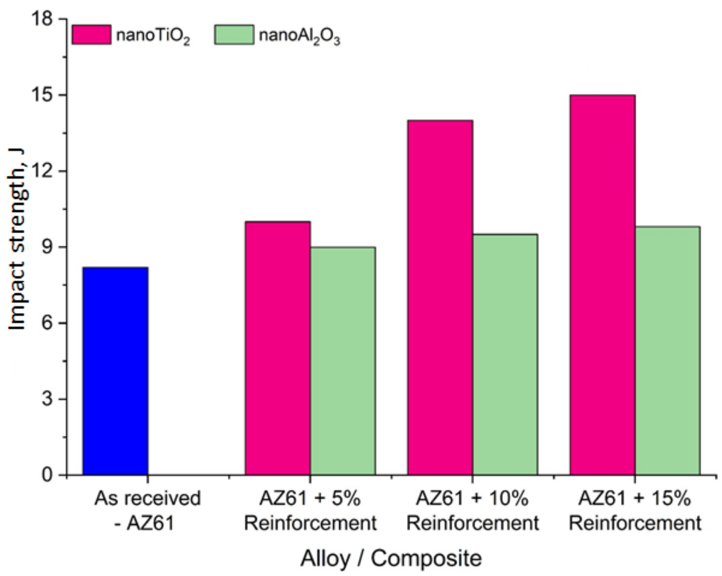

Figure 6. Impact strength of the non-modified AZ61 Mg alloy reinforced with nano-Al_2O_3 or nano-TiO_2 nanoparticles at different concentrations

The maximal impact strength was determined for the alloy reinforced with 15 vol.% nano-TiO_2 amounting to 15 J. In contrast, the alloys reinforced with nano Al_2O_3 showed negligible difference as compared to the received alloy amounting to 9 J. This is due to the influence of reinforced nanoparticles that resist fracture and can withstand higher energy generated during the impact loads. With the increase in the percentage of nano-TiO_2 from 5 to 15 vol.% the impact strength is increased. However, the increase in the content of nano-Al_2O_3 did not improve the impact strength of the alloy. This result might be because of the grain alteration in the friction stir region despite the maximum heat production [16].

3.4. Microhardness

Comparison of measured microhardness values for the as-received AZ61 Mg alloy and those reinforced by nano-Al_2O_3/nano-TiO_2 is shown in Figure 7. All reinforced specimens show higher microhardness in the nugget zone than that of the unmodified specimen. Nano-TiO_2 reinforced AZ61 Mg alloys generally exhibited higher microhardness than those of nano-Al_2O_3 reinforced alloys except for the alloy with 10 vol.% nano-Al_2O_3.

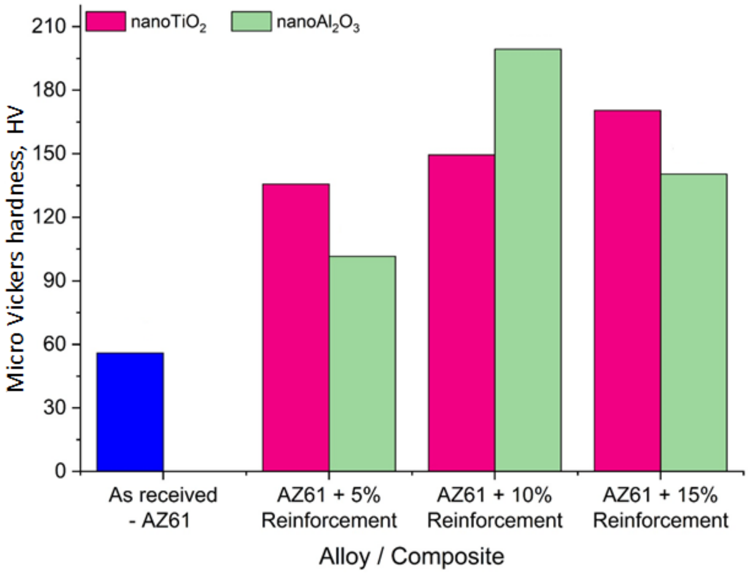

Figure 7. Microhardness of the non-modified AZ61 reinforced with Al_2O_3 or TiO_2 nanoparticles at different concentrations

Orowan strengthening, grain refinement, and substructure strengthening are the major mechanisms identified that improve the strength of the nano-ceramic strengthened surface-modified composite that is obtained with the uniform dispersal of nano-TiO_2 particles and better bonding with the AZ61 matrix. The material drifts in a multifaceted fashion from the receding to the progressing side amid FSP, offering an increase in the inclines in temperature, strain, and strain rate through the stir region. Furthermore, since a groove was created in the middle of the specimen to receive nano-particles, the specimen must drift into the drilled groove to seal it to provide a defect-free continuous stir region. A lower microhardness of nano-Al_2O_3 reinforced AZ61 Mg alloys could be due to grain modification in the FSP region because of higher heat generation [17].

4. CONCLUSION

1. The new magnesium surface composite created in this work revealed improved mechanical properties as compared to the neat Mg alloy, proving that reinforcementby the surface modification method used in the present study is a viable way for qualities such an improvement also result in desirable surface properties.
2. The best mechanical properties were achieved with 15 vol.% of nano-TiO_2 although microhardness was lower as compared to 10 vol.% due to lower homogeneity of nanoparticles and weaker bonding under loads.

3. Mg alloy strengthened with nano-TiO_2 powder has the potential for utilization in the automotive industry due to its high mechanical strength. This will lead to even wider applications of already broadly used magnesium alloys, significantly increasing their use.

REFERENCES

[1] Huang, S.J, Subramani, M, Chiang, CC. Effect of hybrid reinforcement on microstructure and mechanical properties of AZ61 magnesium alloy processed by stir casting method.*Compos Commun*. 2021; 25: 100772. https://doi.org/10.1016/j.coco.2021.100772

[2] Billard, A., Maury, F., Aubry, P., Balbaud-Célérier, F., Bernard, B., Lomello, F., Maskrot, H., Meillot, E., Michau, A. and Schuster, F. Emerging processes for metallurgical coatings and thin films. *CR Phys*. 2018: 19; 755-768.https://doi.org/10.1016/j.crhy.2018.10.005

[3] Mahoney MW, Binget WH, Mishra RS. Microstructural Modification and Resultant Properties of Friction Stir Processed Cast NiAl Bronze. *Mater Sci Forum.* 2003; 426: 2843-2853. https://doi.org/10.4028/www.scientific.net/MSF.426-432.2843

[4] Ammouri A H, Kridli G, Ayoub G, Hamade RF. Relating grain size to the Zener–Hollomon parameter for twin-roll-cast AZ31B alloy refined by friction stir processing. *J Mater Process Technol*. 2015; 222: 301-306.https://doi.org/10.1016/j.jmatprotec.2015.02.037

[5] Xue P, Xiao BL, Ma ZY. Enhanced strength and ductility of friction stir processed Cu–Al alloys with abundant twin boundaries.*Scr Mater*. 2013; 68: 751-754. https://doi.org/10.1016/j.scriptamat.2013.01.003

[6] Chai F, Zhang D, Li Y, Microstructures and tensile properties of submerged friction stir processed AZ91 magnesium alloy. *J Magnes Alloy*. 2015; 3: 203–209. https://doi.org/10.1016/j.jma.2015.08.001

[7] Patel V, Li W, Vairis A, Badheka V. Recent development in friction stir processing as a solid-state grain refinement technique: microstructural evolution and property enhancement.*Crit Rev Solid State Mater Sci*. 2019; 44: 378-426. https://doi.org/10.1080/10408436.2018.1490251

[8] Satish Kumar T, Shalini S, Thankachan T. Friction stir processing based surface modification of AZ31 magnesium alloy. *Mater Manuf Process*. 2023; 38(11): 1426-1435. https://doi.org/10.1080/10426914.2023.2165670

[9] Aatthisugan I, Rose AR, Jebadurai DS. Mechanical and wear behaviour of AZ91D magnesium matrix hybrid composite reinforced with boron carbide and graphite.*J Magnes Alloy*. 2017; 5: 20-25. https://doi.org/10.1016/j.jma.2016.12.004

[10] Balaji E, Sathiya Moorthy R. Investigation on Mechanical and Wear Properties of ZE43 Magnesium Composites Reinforced with Silicon Nitride by Friction Stir Processing. *Silicon*. 2022; 6: 11881-11890. https://doi.org/10.1007/s12633-022-01914-1

[11] Sagar P, Handa A, Kumar G. Metallurgical, mechanical and tribological behavior of Reinforced magnesium-based composite developed Via Friction stir processing. *Proc Inst Mech Eng E:* 2022; 236: 1440-1451. https://doi.org/10.1177/09544089211063099

[12] Adetunla A, Akinlabi E. Influence of reinforcements in friction stir processed magnesium alloys: insight in medical applications. *Mater Res Express*. 2018; 6: 025406. https://doi.org/10.1088/2053-1591/aaeea8

[13] Gobara M, Shamekh M, Akid R. Improving the corrosion resistance of AZ91D magnesium alloy through reinforcement with titanium carbides and borides. *J Magnes Alloy*. 2015; 39: 112-120. https://doi.org/10.1016/j.jma.2015.03.002

[14] Gangil N, Nagar H, Mohammed SMAK, Singh D, Siddiquee AN, Maheshwari S, Chen DL. Fabrication of magnesium–NiTip composites via friction stir processing: Effect of tool profile. *Metals*. 2020; 10: 1425. https://doi.org/10.3390/met10111425

[15] Dinaharan I, Zhang S, Chen G, Shi Q. Titanium particulate reinforced AZ31 magnesium matrix composites with improved ductility prepared using friction stir processing. *Mater Sci Eng*. 2020; 772: 138793. https://doi.org/10.1016/j.msea.2019.138793

[16] Das U, Toppo V. Effect of Tool Rotational Speed on Temperature and Impact. *Mater Today Proc*. 2018; 5: 6170-6175. https://doi.org/10.1016/j.matpr.2017.12.223

[17] Thirumalvalavan S, Senthilkumar N, Experimental Investigation and Optimization of HVOF Spray parameters on wear resistance behaviour of Ti-6Al-4V Alloy, ,*C R Acad Bulg Sci*.2019; 72: 665-674. https://doi.org/10.7546/CRABS.2019.05.15

Mehanička svojstva površinski modifikovane legure magnezijuma AZ61 sa nanočesticama aluminijum oksida i titanijum dioksida obradom putem tehnike trenja sa mešanjem

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(Stručni rad)

Izvod

U ovom radu su prikazani rezultati istraživanja mehaničkih osobina površinski modifikovane legure magnezijuma AZ61, ojačane nanočesticama Al_2O_3 i TiO_2 primenom tehnike trenja sa mešanjem (engl. *friction stir processing* - FSP). Površinski modifikovane legure AZ61 su proizvedene dodavanjem različitih količina nanočestica Al_2O_3 i TiO_2 (5, 10 i 15 vol.%). Mikrostruktura dobijenih površinskih kompozita pokazuje ujednačenu disperziju dodatih nanočestica, što je rezultiralo poboljšanjem njihovih mehaničkih svojstava primenom FSP. Ojačavanjem legure nanočesticama TiO_2 krajnja zatezna, udarna i mikro-tvrdoća su poboljšane za 20, 45 i 67 %, redom, u poređenju sa livenom legurom. Rezultati ove studije ukazuju da ojačana legura AZ61 Mg može biti potencijalni materijal za primenu u automobilskoj industriji, jer poseduje veliku čvrstoću i malu specifičnu težinu.

Ključne reči: Karakterizacija; metalografija; oruđa velike brzine; nano-filteri