INVESTIGATION OF MICROSTRUCTURE, HARDNESS AND WEAR PROPERTIES OF MWCNT REINFORCED AZ91 MAGNESIUM MATRIX COMPOSITES

[1]YAVUZ SUN, [2]M. EMRE TURAN, [3]HAYRETTIN AHLATCI, [4]YUNUS TUREN, [5]MEHMET UNAL

[1]Karabuk University, Department of Metallurgy and Materials Engineering, TURKEY
[2]Karabuk University, Department of Manufacturing Engineering, TURKEY
Email: [1]ysun@karabuk.edu.tr, [2]memreturan@karabuk.edu.tr, [3]hahlatci@karabuk.edu.tr, [4]yturen@karabuk.edu.tr, [5]munal@karabuk.edu.tr

Abstract: In this study, 0.25 wt.% Multi-Wall Carbon Nanotube (MWCNT) reinforced magnesium matrix composites were synthesized by semi-powder metallurgy technique. Uniform distribution of MWCNT and improvement of hardness and wear performance were aimed at this work. Unreinforced AZ91 magnesium alloy and MWCNT reinforced composite were fabricated in hot press test device. Argon atmosphere was used to prevent oxidation due to having poor oxidation resistance of magnesium and its alloys. Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) were used to characterize of the present phases and microstructure. Tribological test was conducted by reciprocating apparatus integrated with tribometer test machine. Different loads (5N, 10N, 20N and 40N) were applied in dry sliding conditions. Based on the results, hardness behavior is improved about 10% with the addition of MWCNT. Samples have free of microstructural defects according to the SEM results. MWCNT reinforced sample has also lower wear rate than unreinforced magnesium alloy.

Keywords: Magnesium, MWCNT, Semi Powder Metallurgy, Tribology.

I. INTRODUCTION

Magnesium and its alloys are promising lightweight structural materials for automotive and aerospace applications [1]. However, the usage of these alloys was limited because of their low modulus [2], poor corrosion performance [3] and losing mechanical performances at elevated temperatures [4]. Thus, researchers have made effort to solve problems of magnesium alloy. One of these attempts can be explained using nano-size particles as reinforcement for magnesium based composites [5]–[7].

In recent years, nano carbon reinforcements such as one dimensional (1D) carbon nanotube [8] and two dimensional (2D) graphene [9] have become interest due to their high strength and stiffness properties. Carbon nanotubes (CNT) are one of the allotropes of carbon and formed as sheets of graphene rolled into cylindrical shape [10]. There is numerous reports show that addition of CNT to magnesium matrix composites causes to increase of strength of composites [11]–[15]. However, carbon nanotube may cause an agglomeration because of Van der Waals attractions between carbon atoms [16]. This is a result of non-homogenous dispersion of CNT in matrix, so it can be still serious problem for practical applications.

In this study, powders were mixed using semi powder metallurgy which includes ultrasonication and distillation in order to overcome agglomeration problems. MWCNT reinforced AZ91 magnesium alloy composite was fabricated then microstructure, hardness and wear performance were evaluated.

II. DETAILS EXPERIMENTAL

AZ91 magnesium powder has 100 µm size ranges and Multiwall Carbon Nanotube (MWCNT) has 9.5 nm thickness. They were supplied from Nanografi Co. Ltd. Turkey. 0.25 wt.% MWCNT reinforced composite was fabricated by powder metallurgy using hot pressing. MWCNT powder was exposed to ultrasonication in 50 ml. ethanol solution for an hour to weaken bonding between carbon atoms. AZ91 magnesium alloy powder was added into the solution. This was the important stage to prevent an agglomeration of carbon atoms. Vacuum distillation system was used to remove ethanol from the system during mixing of powders. AZ91 and MWCNT were mixed in this system via magnetic stirring. At the end, powders were dried at 90 °C in atmosphere-controlled furnace under argon gas overnight. **Table 1** illustrates the pressing and sintering parameter of prepared powders.

Table 1:Sintering parameters of powders.

Mold	Graphite
Sintering Temperature	490 ^{0}C
Sintering Time	1.5 hours
Pressure at 490 ^{0}C	40 MPa
Pressure Time	15 minutes

Metallographic processes were performed for unreinforced AZ91 magnesium alloy and MWCNT reinforced composite. 120, 240, 400, 600, 800, 1000, 1200 and 2000 SiC grit papers were used for mechanically grinding. Polishing process with 9 µm, 3 µm and 1 µm diamond paste was applied respectively. The phases were analysed with Rigaku

X-ray test device. Samples were etched by picric acid solution during 10 sec. Microstructure of samples were investigated by Carl Zeiss Ultraplus Field Emission Scanning Electron Microscope (FESEM). Vickers hardness test was performed applying 500 g load.

Tribological tests were conducting by a reciprocating type test machine. 5N, 10N, 20N and 40N were applied. Wear rates, wear depth and friction coefficients of the specimens were recorded. Sliding speed was determined as 0.25 m/s during 1000 m. sliding distance.

III. RESULTS AND DISCUSSION

3.1. X-Ray Diffraction Test Results

Fig.1 shows the X-ray diffraction analysis of samples. Peaks of Mg17Al12 intermetallic phase are clearly seen in certain ranges. The expected peak of purchased MWCNT is 2θ equal to 23.90 could not be detected with full continuous scanning. So, fix time scanning method (slower detection) was conducted in the range of 2θ equal to 23-250 and new peak (belonging to the MWCNT) was obtained and inset of **Fig. 1** indicates this peak formation. As a result, the new peak formation supports the existence of MWCNT in fabricated composite.

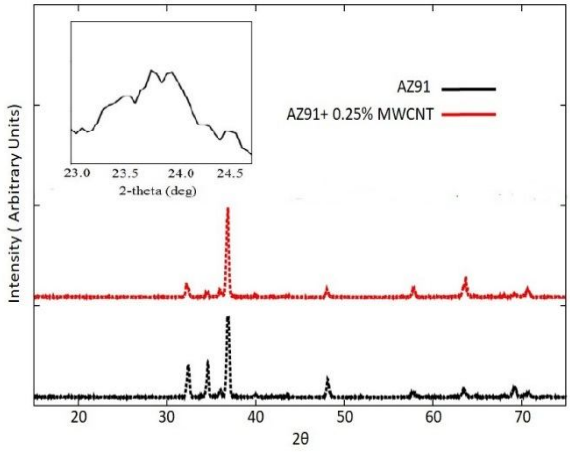


Fig. 1. XRD patterns of unreinforced and MWCNT samples.

3.2. Microstructural Analysis

SEM images of samples are given in **Fig.2.** The Mg17Al12 (eutectic β) phases can be seen in both of unreinforced and MWCNT reinforced sample. Also discontinuous β precipitates are visible in grains and eutectic β phases are located around the grains. **Fig.2.b** shows the MWCNT reinforced composite. The intermetallic phases are finer than unreinforced sample. There are no cracks or macro porosities in this sample. It can be attributed that there is a good metallurgical bonding between matrix and reinforcement.

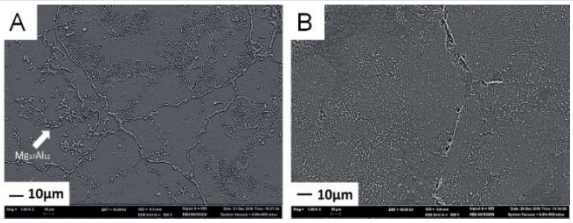


Fig. 2. SEM Microstructure of specimens (A) AZ91 (B) AZ91-0.25 wt.% MWCNT.

3.2. Hardness Results

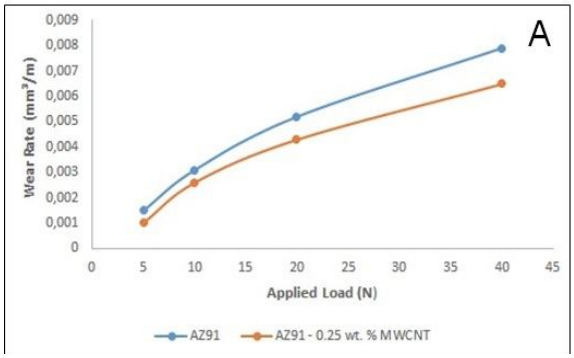
Vickers hardness values of produced samples are presented in **Table 2.** Hardness performance of magnesium alloy was improved considerably with the addition of carbonaceous reinforcement. This can be explained by the addition of harder particle in matrix and the bonding between matrix and reinforcement can play an effective role to determine the motion of dislocation. When the density of dislocation enhances, dislocation movement is restricted.

Table 2. Hardness Results of specimens.

Materials	Hardness (HV)
AZ91 Magnesium alloy	72.4 ± 0.6
AZ91-0.25 wt.% MWCNT	80.3 ± 0.8

3.2. Wear Properties

Fig. 3 shows the wear rate and coefficient of friction values versus applied loads of specimens. Wear rate was calculated using volume loss of specimens at the end of sliding distance. MWCNT reinforced composite exhibits better wear resistance than unreinforced magnesium alloy. Especially, under load of 40 N, the difference of wear rate values is more obvious. The enhancement of wear performance might be attributed to lubricant effect of carbonaceous reinforcement. This sample also has higher hardness and it supports the Archard's law which explains the relationship between hardness and wear [17]. Softer materials generally represent the lower wear resistance than harder material. When the coefficient of friction (COF) values was evaluated, reinforced material has lower coefficient of frictions. This can be a result of composite having enough solid lubricant from the surface.



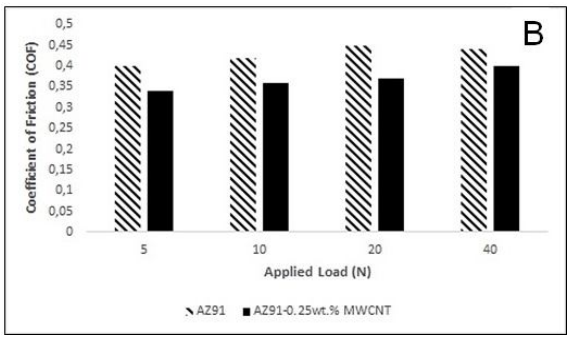


Fig. 3. Wear rates (A) and COF (B) results of specimens.

CONCLUSIONS

Effect of MWCNT on hardness, microstructure and wear performances of magnesium alloy was investigated with this work. According to the obtained results, the findings are summarized as follows:

1. Uniform distributed MWCNT reinforced composite was fabricated successfully.
2. MWCNT reinforced sample has finer intermetallic phases than unreinforced AZ91 alloy. Both samples have free of macro porosities and cracks.
3. Hardness was improved with the addition of MWCNT.
4. AZ91-0.25 wt.% MWCNT composite has better wear resistance than AZ91 alloy.

ACKNOWLEDGMENTS

This research is supported by the Scientific Research Projects of Karabuk University (BAP) with project no. KBU-BAP-18-YD-064..

REFERENCES

[1] M. F. Montemor, "Corrosion issues in joining lightweight materials: A review of the latest achievements," Phys. Sci. Rev., vol. 1, no. 2, 2016.

[2] R. Willumeit-Römer and A. Weinberg, "Special issue 'Biodegradable magnesium as implant material,'" BioNanoMaterials, vol. 16, no. 1, pp. 1–2, 2015.

[3] D. Thirumalaikumarasamy, K. Shanmugam, and V. Balasubramanian, "Developing Empirical Relationships to Predict Porosity and Microhardness of Atmospheric Plasma-Sprayed Alumina Coatings on AZ31B Magnesium Alloy," J. Manuf. Sci. Prod., vol. 15, no. 2, pp. 169–181, 2015.

[4] P. S. Roodposhti, A. Sarkar, K. L. Murty, H. Brody, and R. Scattergood, "Grain boundary sliding mechanism during high temperature deformation of AZ31 Magnesium alloy," Mater. Sci. Eng. A, vol. 669, pp. 171–177, 2016.

[5] S. C. Tjong, "Novel nanoparticle-reinforced metal matrix composites with enhanced mechanical properties," Adv. Eng. Mater., vol. 9, no. 8, pp. 639–652, 2007.

[6] K. B. Nie, X. J. Wang, L. Xu, K. Wu, X. S. Hu, and M. Y. Zheng, "Effect of hot extrusion on microstructures and mechanical properties of SiC nanoparticles reinforced magnesium matrix composite," J. Alloys Compd., vol. 512, no. 1, pp. 355–360, 2012.

[7] X. L. Zhong, W. L. E. Wong, and M. Gupta, "Enhancing strength and ductility of magnesium by integrating it with aluminum nanoparticles," Acta Mater., vol. 55, no. 18, pp. 6338–6344, 2007.

[8] W. Zhou, T. Yamaguchi, K. Kikuchi, N. Nomura, and A. Kawasaki, "Effectively enhanced load transfer by interfacial reactions in multi-walled carbon nanotube reinforced Al matrix composites," Acta Mater., vol. 125, pp. 369–376, 2017.

[9] M. Rashad, F. Pan, M. Asif, and X. Chen, "Corrosion behavior of magnesium-graphene composites in sodium chloride solutions," J. Magnes. Alloys, Jul. 2017.

[10] R. K. Pandyan, S. Seenithurai, S. V. Kumar, and M. Mahendran, "Magnesium Hydride Doped on Single-Walled Carbon Nanotubes for Hydrogen Adsorption," Fuller. Nanotub. Carbon Nanostructures, vol. 23, no. 2, pp. 175–180, 2015.

[11] Y. Shimizu et al., "Multi-walled carbon nanotube-reinforced magnesium alloy composites," Scr. Mater., vol. 58, no. 4, pp. 267–270, 2008.

[12] C. S. Goh, J. Wei, L. C. Lee, and M. Gupta, "Development of novel carbon nanotube reinforced magnesium nanocomposites using the powder metallurgy technique," Nanotechnology, vol. 17, no. 1, p. 7, 2005.

[13] K. Kondoh, H. Fukuda, J. Umeda, H. Imai, B. Fugetsu, and M. Endo, "Microstructural and mechanical analysis of carbon nanotube reinforced magnesium alloy powder composites," Mater. Sci. Eng. A, vol. 527, no. 16, pp. 4103–4108, 2010.

[14] M. Rashad, F. Pan, A. Tang, M. Asif, and M. Aamir, "Synergetic effect of graphene nanoplatelets (GNPs) and multi-walled carbon nanotube (MW-CNTs) on mechanical properties of pure magnesium," J. Alloys Compd., vol. 603, pp. 111–118, 2014.

[15] C. S. Goh, J. Wei, L. C. Lee, and M. Gupta, "Simultaneous enhancement in strength and ductility by reinforcing magnesium with carbon nanotubes," Mater. Sci. Eng. A, vol. 423, no. 1, pp. 153–156, 2006.

[16] L. Y. Jiang et al., "A cohesive law for carbon nanotube/polymer interfaces based on the van der Waals force," J. Mech. Phys. Solids, vol. 54, no. 11, pp. 2436–2452, 2006.

[17] J. Archard, "Contact and rubbing of flat surfaces," J. Appl. Phys., vol. 24, no. 8, pp. 981–988, 1953.

★★★