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Article

Effect of WS_2 Nanotubes on the Mechanical and Wear Behaviors of AZ31 Stir Casted Magnesium Metal Matrix Composites

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Abstract: In this study, the AZ31 magnesium alloy was reinforced with tungsten disulfide (WS_2) nanotubes to fabricate the nanocomposite using the stir casting method. The microstructural analysis, mechanical and wear behaviors were investigated with the effect of WS_2 on the AZ31 alloy. Scanning electron microscopy (SEM) was used to conduct the microstructural analysis. The microstructures are revealed to incorporate the aluminum content with the WS_2 nanotube, disclose the presence of the secondary phase, which was increased compared with the AZ31 alloy and was detected by energy dispersive spectroscopy (EDS). The mechanical properties of hardness and yield strength (YS) were significantly improved with the addition of WS_2 nanotubes. This is mainly due to the strengthening mechanisms of Orowan, the coefficient of thermal expansion (CTE) mismatch and the load transfer mechanism. The theoretical YS was calculated and compared with the experimental results. However, the ultimate tensile strength (UTS) and the fracture strain were decreased with the addition of reinforcement which might be owing to the clustering of nanotubes. Finally, the wear behavior of the wear weight loss and depth of cut was investigated. This test revealed that the addition of WS_2 nanotubes reduced the weight loss and depth of the material cutting that was mainly due to the presence of hard WS_2 nanotubes.

Keywords: AZ31 magnesium alloy; tungsten disulfide; strengthening mechanism; wear behavior

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1. Introduction

Nowadays, magnesium and its alloys are widely attractive in applications in automobile and aerospace industries because of their low density compared with other structural metals [1,2]. In particular, the AZ series magnesium alloys have great attention due to their excellent castability and high strength. However, magnesium and its alloys have a limited number of slip systems and poor formability and workability at room temperature [3–5]. These limitations can be controlled by the addition of ceramic particles and whiskers such as Al_2O_3, SiC, B_4C, CNT and WS_2, etc., [5–13]. The combined effects of the matrix and ceramic particles are greatly advantageous in magnesium alloy metal matrix composites (MMCs). The selection of proper conventional reinforcement can produce excellent mechanical characteristics such as high strength and rigidity and low CTE mismatch, which are all suitable for modern applications [13]. In terms of high strength and stiffness, high hardness, high wear resistance, high melting point and stability, chemical inertness, facile dispersion and low cost, tungsten disulfide (WS2) microparticles outperform alternative reinforcements [14]. According to reports, WS2 has the highest stiffness and strength of any other reinforced material when dispersion is good [15]. Due to their superior mechanical properties, WS2 microparticles were chosen. Continuous and discontinuous $Mg_{17}Al_{12}$ precipitates in molten AZ91 alloy are hypothesized to be accelerated by WS2 [14]. $Mg_{17}Al_{12}$ degrades the mechanical characteristics of magnesium-based composites and WS2 microparticles modify the $Mg_{17}Al_{12}$ phase precipitation kinetics in AZ91 magnesium alloy [13]. In addition, the WS_2 could play a vital role in the evolution of MMCs due to the coefficient thermal expansion (CTE) mismatch and its cost-effectiveness [14,15]. Furthermore, the incorporation of aluminum in the magnesium alloy usually enhances the mechanical and wear behaviors of MMCs. The addition of aluminum can increase strength and hardness when the aluminum content reaches 6 wt.%. If the aluminum content increases more than 6 wt.%, there will be an effect of grain refinement, but the ductility will decrease. The formations of $Mg_{17}Al_{12}$ particles (β-phase) solid solution at 437 °C under non-equilibrium solidification conditions are the main parameters for the improvement of the mechanical properties [16]. The size of the particles is an important role in the investigation of mechanical and wear behaviors. The nanoparticles are revealed to have better mechanical properties and a superior wear resistance than microparticle-reinforced composites due to their higher surface-to-volume ratio [17,18]. The MMCs were fabricated using various fabrication techniques such as stir casting, squeezing die casting and powder metallurgy. Among these, stir casting is commonly accepted as an economical route practiced commercially due to advantages such as a high production rate, ability to produce a complex-shaped product, simplicity and the production capacity of large ingots. So, particle-reinforced MMCs manufactured by stir casting are a low-cost and promising technique for mass production [5,17,19]. WS2 is stable material at high temperatures. This research work is aimed to develop and characterize a new type of composite with a small amount of reinforcement to improve the mechanical properties. We have used a very tiny amount of them which causes a minor change in the price of the final product. Up to now, it was traditionally used as a lubricant or as a reinforcement of a polymer matrix. However, its introduction into a metallic matrix may have a positive input to obtain reinforcement. Therefore, the topic of this study has an interest in the fields of composite materials.

The present study aims to investigate the mechanical and wear behavior of as-cast magnesium alloy (AZ31) reinforced with WS_2 nanotubes by the stir casting technique. The effect of WS_2 nanotubes have been investigated on the microstructural analysis and mechanical and wear behaviors.

2. Materials and Methods

In this study, AZ31 magnesium alloy was used as a matrix material and 0.1 wt.% of tungsten disulfide nanotube (WS_2) [13] was used as reinforcement material. The average size of the WS2 nanotubes was around 40 nm and the microstructure is shown in Figure 1a. Furthermore, 3 wt.% of aluminum content was incorporated into the nanocomposite. The chemical composition of the AZ31 magnesium alloy is presented in Table 1. The nanocomposites were fabricated by the stir casting method. The fabrication procedures were described in our previous research [5].

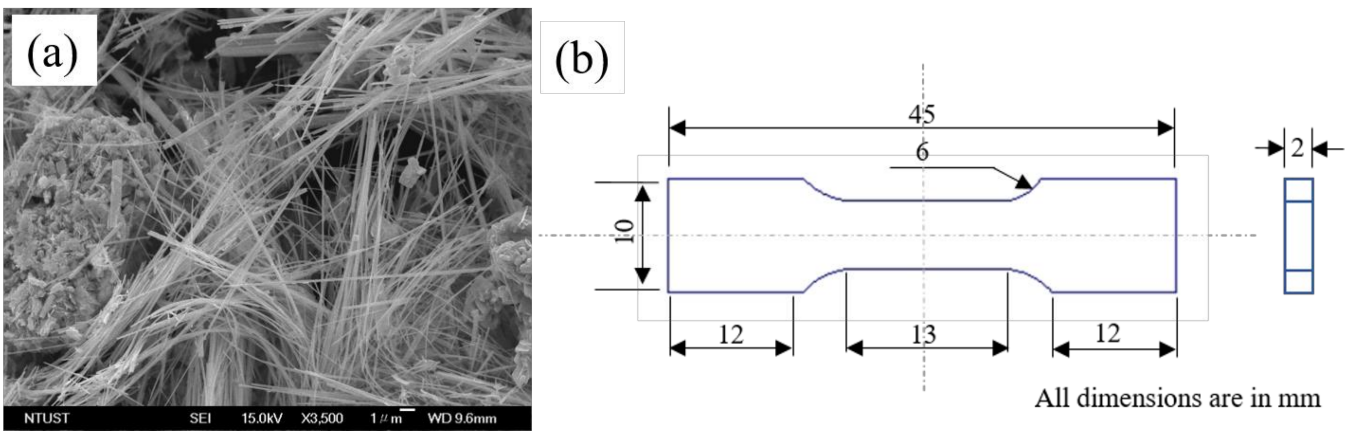


Figure 1. (**a**) Microstructure of WS_2 and (**b**) tensile specimen.

Table 1. The chemical composition of the AZ31 magnesium alloy.

Al	Zn	Mn	Fe	Mg
2.95	0.64	0.26	0.005	balance

First, the fabricated composite ingots were cut into 10 × 10 × 5 mm^3 to analyze the microstructures using a SEM-equipped EDS analysis. Secondly, the tensile samples were prepared according to the ASTM standards, as shown in Figure 1a, and wear test samples were cut into 10 × 10 mm^2. The prepared samples were polished with different grit waterproof silicon carbide abrasive paper. The hardness measurements and tensile and abrasion tests were conducted to determine the mechanical and wear behaviors of the fabricated composites. Before every test, the samples were used to polish with fine abrasive paper to avoid the micro cracks which might be induced during the machining process. A tensile test was performed using Insight—100 MTS universal tensile testing machine. The crosshead speed was used at 0.5 mm/min. The average of four tested samples was taken into account for final result. The Vickers hardness measurement was performed using an FV—810 tester. The load of 10 kgf for 10 s was used to measure the Vickers hardness. Ten measurements of each sample and the average were taken into account for the final results. The wear test was conducted using Linear abraser 5750 with a Si_3N_4 ball (diameter of 1/4 in) in dry conditions (distance—25.4 m, speed—75 cycles/min and load—750 g). After the wear test, the Ion BM-5 microbalance machine, with an accuracy of 0.001 mg, was used to measure the weight loss and an average of five measurements is presented. The worn-out surface images were performed using an RH—2000 digital microscope.

3. Results and Discussions

3.1. Microstructural Characteristics

The SEM microstructural analysis followed by EDS mapping analysis of the fabricated as-cast AZ31/WS_2 nanocomposites is presented in Figure 2. The presence of the primary α—Mg phase (red arrow), secondary β—$Mg_{17}Al_{12}$ phases (green arrow) and the AlMn (white arrow) phase can clearly be detected in the images. The unreinforced nanocomposites show a small number of secondary phases which is due to the low quantity of aluminum content in the AZ31 alloy. However, the addition of the reinforcement in AZ31 revealed the increase in the continuous secondary phase and the uniform distribution that might be due to the additional incorporation of the aluminum dissolution and WS_2 nanotubes, as previously described in [16]. Another research also reported that when the content of aluminum increased by more than 1%, it promoted the formation of the secondary phases [20]. Moreover, the presence of the phases was confirmed by the elemental mapping analysis. However, phases related to reinforcement were not detected, which is mainly due to the very low weight fraction of the WS_2 nanotubes (0.1%). However, using scanning electron microscopy, Huang et al. [21] investigated the agglomeration or dispersion uniformity of the WS2 nanotubes in the AZ31 matrix morphology and microstructure. They discovered the agglomerated and dispersion particles of WS2 near the AlMn phase. Near the b phase, AlMn was also found. As a result, they believe that the WS2 compound is important in the microfracturing of Mg—Al alloys.

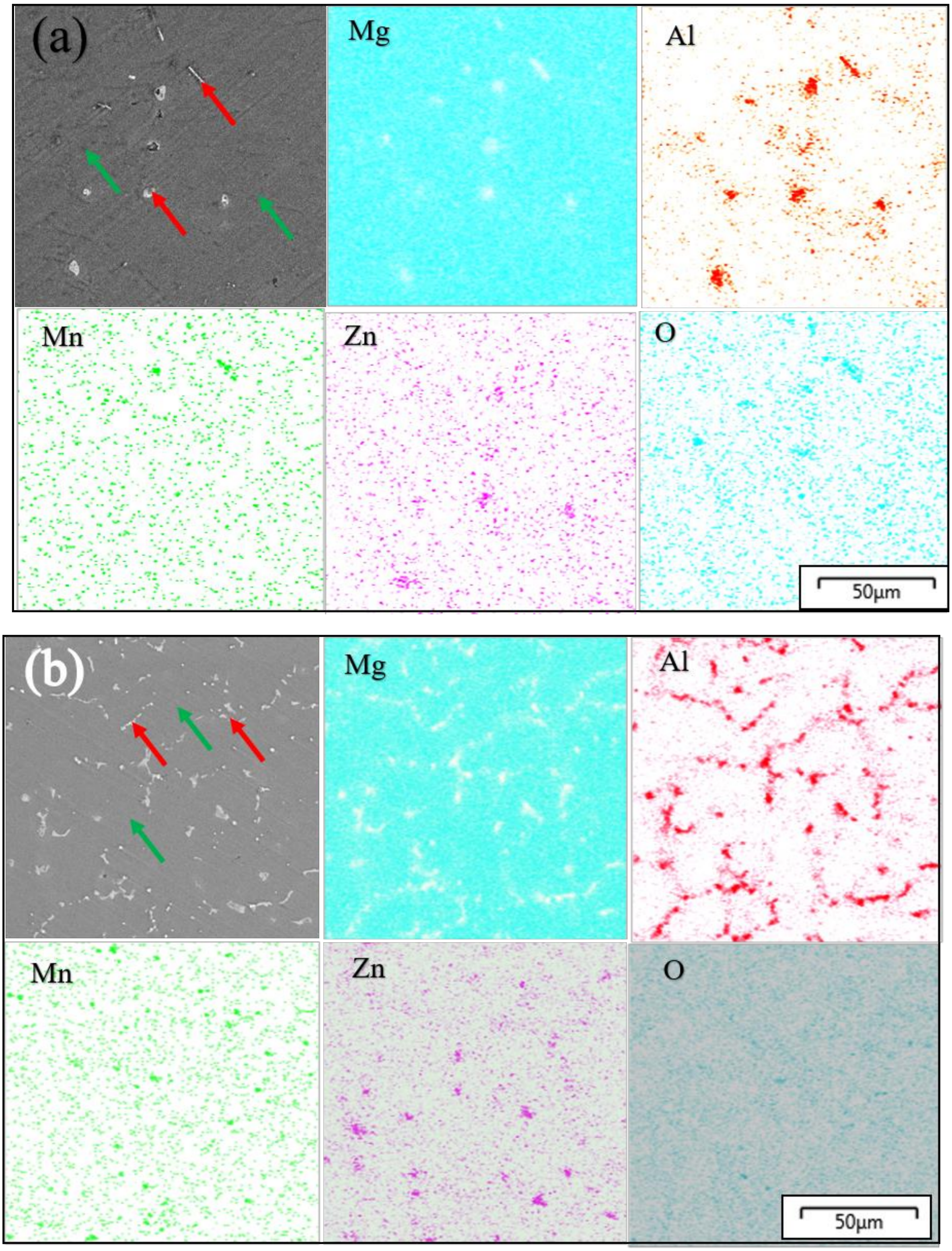
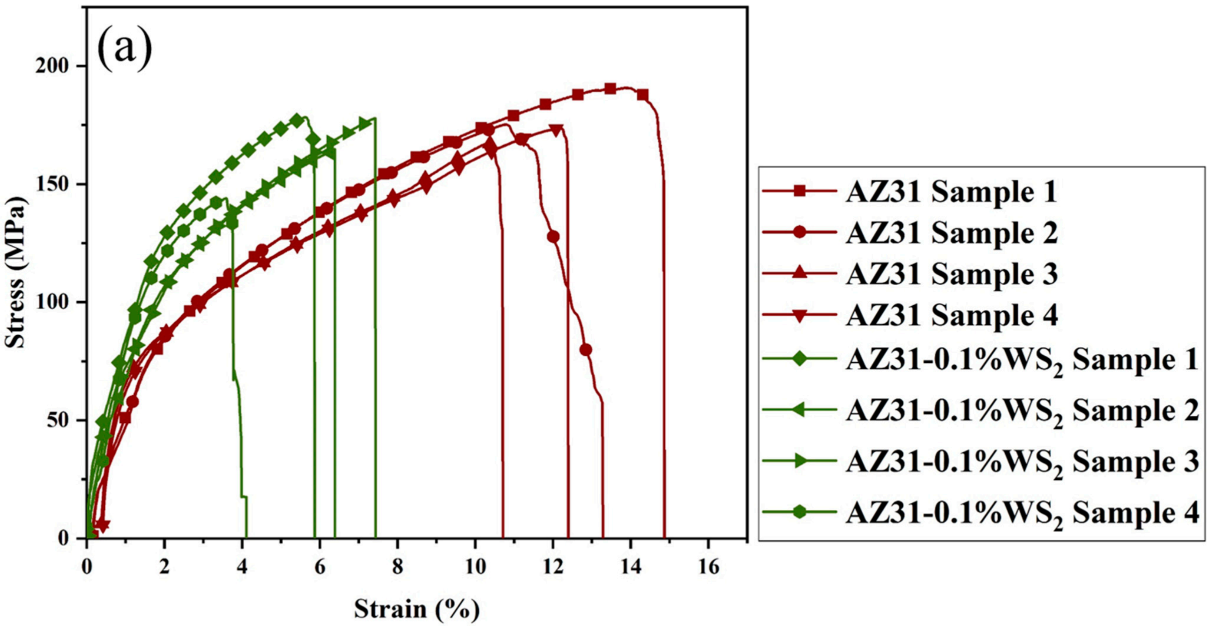


Figure 2. SEM images followed by EDS mapping analysis of (**a**) AZ31 and (**b**) AZ31/ WS_2 composites.

3.2. Mechanical Characterization

The Vickers hardness measurements of the fabricated composites are presented in Table 2. The hardness value for AZ31 was 40.44 HV and for WS_2-reinforced AZ31 nanocomposites was 55.61 HV, which point to a significant increase of 37.5%. The enhancement of the hardness is due to the presence of hard reinforcement WS_2 nanotubes which restricted the highly localized plastic deformations during hardness indentation. The experimental stress–strain curves of the fabricated AZ31/WS_2 nanocomposites are shown in Figure 3a. The UTS, YS and fracture strain values were presented in Table 2 (average of four tested samples of each composites). It can be seen that the addition of WS_2 nanotubes significantly improved the YS of the AZ31/ WS_2 nanocomposites from 86.7 MPa to 112.9 MPa. The YS of the nanocomposite was increased by 30.2%. The enhancement of hardness and YS is mainly due to several strengthening mechanisms: (i) Orowan strengthening, (ii) CTE mismatch and (iii) the load transfer mechanism. The strengthening contribution of the WS_2 nanotubes is quite significant in the YS. The WS_2 strengthening contribution was conducted using the theoretical study with the following equation [4].

$$\sigma_p = \sqrt{\left[(\Delta\alpha_{CTE})^2 + (\Delta\sigma_{OL})^2 + (\Delta\sigma_{LT})^2\right]} \quad (1)$$



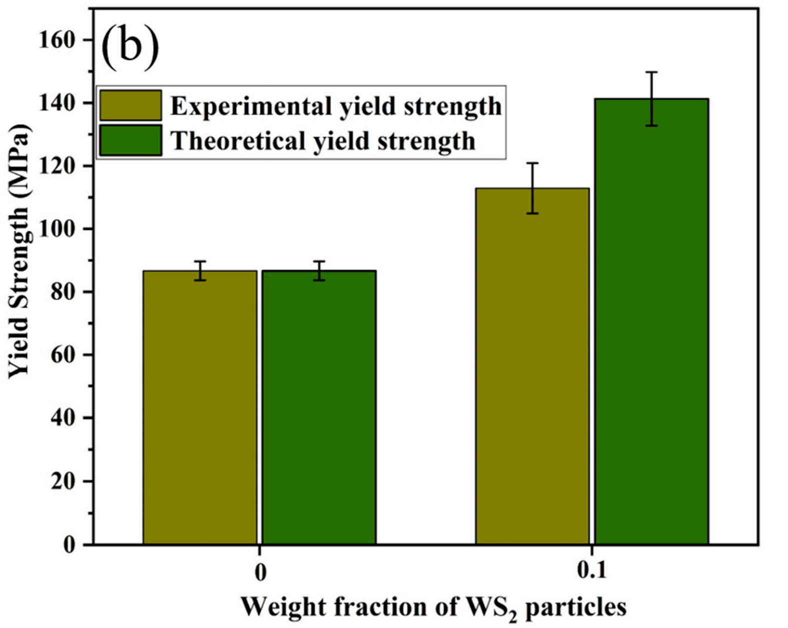


Figure 3. (**a**) Stress–strain curve and (**b**) experimental and theoretical values of the fabricated nanocomposites.

In the composite materials, homogeneously dispersed nanotubes should enhance the YS owing to Orowan strengthening. The dispersed reinforcement can obstruct the dislocation motion. However, the Orowan strengthening effects mainly depend on the mean particle size because it is inversely proportional to the particle size. Therefore, when the reinforcement particle size decreases, the contribution of the Orowan strengthening effect increases. The contribution of the Orowan strength has been calculated by the following equation [22].

$$\Delta\sigma_{Orowan} = \frac{0.13 \times G_m \times b}{d_r \times \left[\left(\frac{1}{2V_r} \right)^{\frac{1}{3}} - 1 \right]} \ln\left(\frac{d_r}{2b} \right) \quad (2)$$

Table 2. Mechanical properties of the AZ31/ WS_2 nanocomposite.

Material	YS (MPa)	UTS (MPa)	Fracture Strain (%)	Hardness (HV)	Theoretical YS (MPa)
AZ31	86.7 ± 9	180.5 ± 10	12.81 ± 3	40.4 ± 2	-
AZ31/WS_2	112.9 ± 8	169.23 ± 13	5.9 ± 2	51.7 ± 4	139.64
AZ61 + 0.4%Sb [23]	99	175	5	-	-
AZ61 + 0.56%Sn [23]	92	161	4.3	-	-
AZ61 + 2%SiC [5]	100.64	166.64	3.44	-	-
AZ91 + 0.6%WS_2 [13]	73.66	151.06	10.21	-	-
AZ91 + 0.5%CNT [9]	76.5	153.5	5.7	-	-

The next contribution mechanism for the YS is the *CTE* mismatch. The *CTE* mismatch between the matrix and reinforcement generates a high dislocation density around the reinforcement compared to the matrix and, hence, the composite shows an improved YS with the addition of WS_2 nanotubes. The contribution of the *CTE* mismatch on the YS has been determined using the following equation [22].

$$\alpha_{CTE} = 1.25 \times G_m \times b \sqrt{\frac{12 \times \Delta\alpha \times \Delta T \times V_r}{b \times d_r \times (1 - V_r)}} \quad (3)$$

Finally, the load transfer mechanism plays a role to improve the YS due to the uniform distribution of the harder WS_2 nanotubes that can bear the load transferred from the soft matrix material. The contribution of the load transfer effect is calculated by the following equation [4].

$$\Delta\sigma_{LT} = 0.5 \times V_r \times \sigma_m \quad (4)$$

The used notations and their values are presented in Table 3.

Table 3. The notations and values for yield strength [16,22].

Notations	Value	Meaning
G_m (GPa)	17.3	Shear modulus
b (nm)	0.32	Burger's vector of the matrix
d_r (nm)	0.07	Average particle size
V_r	0.02	The volume fraction of particle
$\Delta\alpha$ (K^{-1})	18.4×10^{-6}	Difference in the coefficients of the thermal expansion
ΔT (K)	280	Difference between the processing and test temperatures
σ_m (MPa)	81.96	Yield strength of the monolithic matrix

The theoretical YS was calculated using Equation (1) and presented in Figure 3b. The theoretical values of the yield strength were higher than the experimental. This deviation is mainly due to the theoretical calculation being determined based on approximations and assumptions.

Furthermore, the WS2 has a smaller surface area to bond to the substrate. In other words, the WS2 and the substrate material have a smaller overall interaction area. As a result of the possibility that the WS2 clustering could be lower, the threshold value was not met. With less of a strengthening phase, clustering is more likely to occur, resulting in weaker bonds at the strengthening phase/substrate material contact. As a result, the yield strength of the WS2 nanotubes can be improved [21].

However, the UTS and fracture strain of the AZ31/WS_2 nanocomposites decreased compared to the AZ31 alloy. This might be due to the clustering of the nanotubes; a similar result was reported by Lu et al. for different types of reinforcements [24]. Furthermore, the formation of the sharp-corner continuous network of the secondary phase possibly has higher stress concentrations. The addition of WS2 nanotubes might have increased the porosity and agglomeration effects which are responsible for the decrease in ductility and UTS for the AZ31/WS2 composite. Furthermore, void nucleation with an addition of reinforcement leads to a loss in ductility. Another factor contributing to this difference is that WS2 particles behave as stress concentrators [13]. This possibly acts as a site for crack initiation; once the crack is initiated, the microcracks are propagated through the continuous network leading to a decrease in the UTS and fracture strain [25].

The tensile properties of the as-composites fabricated in this present study were compared with those of the AZ61 and AZ91 alloys reinforced with SiC, WS_2, Sb and Sn particulates, as shown in Table 2. Note that the tensile properties of the AZ31/WS_2 composites are higher than those of the previously reported composites synthesized by conventional stir casting methods.

3.3. Wear Behavior

Figures 4 and 5 depict the wear weight loss and the three-dimensional worn-out surface of the AZ31/WS_2 nanocomposites. Figure 4 reveals that the addition of WS_2 nanotubes can reduce the weight loss during the wear test. The weight loss for AZ31 (1.5608 mg) is higher than the AZ31/WS_2 (1.035 mg) which points to an increase in the wear resistance of the AZ31/WS_2 nanocomposites compared to the AZ31 matrix alloy. Further, Figure 5 reveals the depth of the penetration of the worn-out surface. It can be seen that the penetration depth of the WS_2 reinforced nanocomposite was reduced (from 170.8 μm to 143.1 μm) which is well related to the weight loss results. The weight loss and penetration depth of the nanocomposite were restricted and reduced mainly due to the presence of the hard reinforcement WS_2 nanotubes in the nanocomposite. The size of the reinforcements also has a vital role in the wear behavior. Previous researches shows that the nano-sized reinforcements are better for wear behavior compared with micron-sized reinforcements. The determined wear behavior of this work was supported by Archard's well-known work which determined that the wear weight loss of a soft matrix, such as Mg, is inversely proportional to the hardness [26].

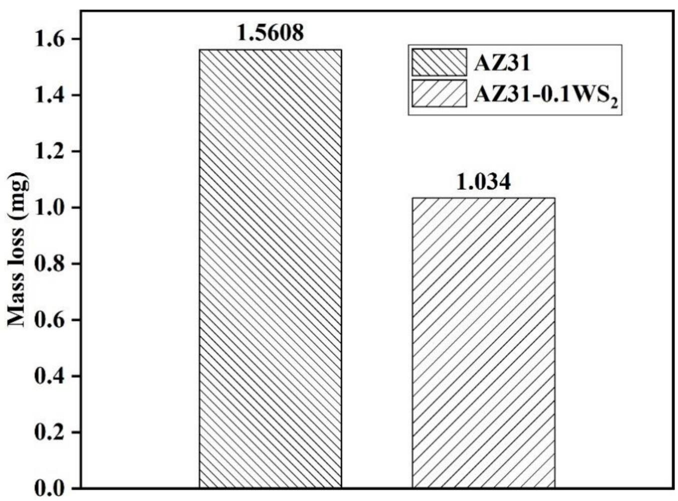


Figure 4. Wear behavior of wear weight loss of the fabricated AZ31/ WS_2 nanocomposites.

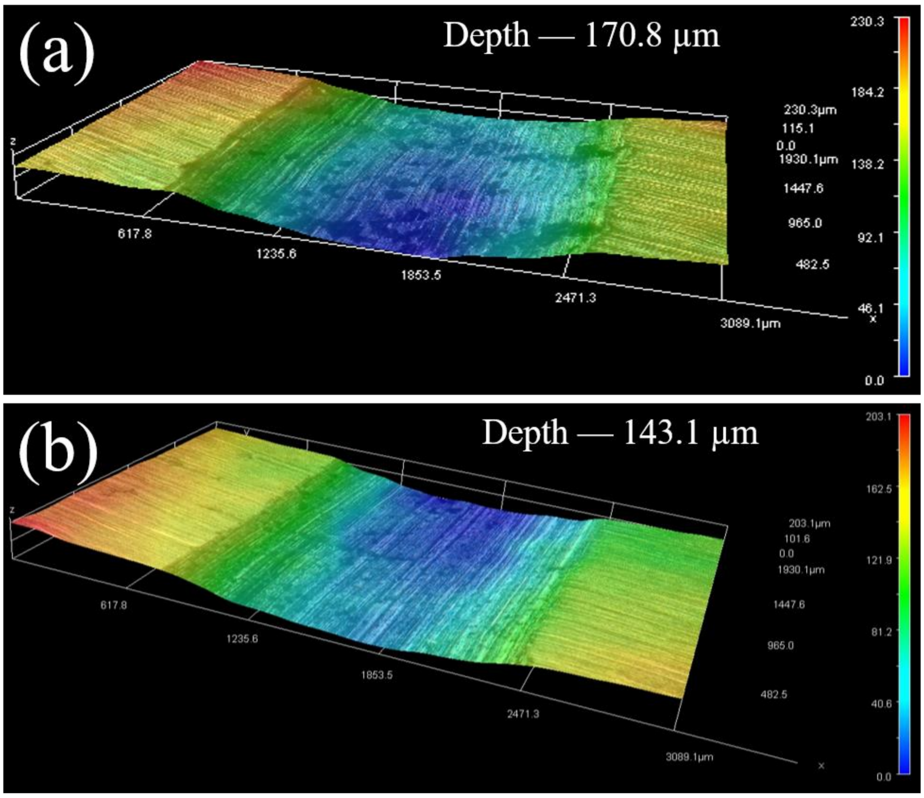


Figure 5. Three dimensional microscope images of the worn surface of (**a**) AZ31 and (**b**) AZ31/WS_2 nanocomposites.

4. Conclusions

The stir casting method was used to fabricate the AZ31/WS_2 nanocomposite. The effect of WS_2 nanotubes on the mechanical and wear behaviors were investigated. The important findings are summarized as follows.

1. The microstructure analysis revealed that the addition of aluminum and WS_2 nanotubes significantly increased the presence of the secondary phase.
2. The hardness and yield strength of the AZ31/ WS_2 nanocomposite was enhanced significantly due to the strengthening mechanisms of the CTE, Orowan and load transfer.
3. The WS_2 nanotubes-incorporated nanocomposite shows better wear behaviors compared with the AZ31 alloy.

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