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INVESTIGATIONS INTO MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Mg-5WT.%Cu-TiB_2 COMPOSITES PRODUCED VIA POWDER METALLURGY ROUTE

B. Stalin [a,*], M. Ravichandran [b], V. Mohanavel [c], L.P. Raj [d]

[a]Anna University, Regional Campus Madurai, Department of Mechanical Engineering, Madurai, India
[b] K.Ramakrishnan College of Engineering, Department of Mechanical Engineering, Tiruchirappalli, India
[c] Chennai Institute of Technology, Department of Mechanical Engineering, Chennai, India
[d] Chendhuran College of Engineering and Technology, Department of Mechanical Engineering, Pudukkottai, India

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Abstract

Magnesium alloy matrix composite reinforced with constant weight fraction of 5% Cu and various weight fractions of (0%, 5%, 10%, 15%) titanium diboride (TiB_2) fabricated by Powder Metallurgy route. In this work, the mechanical properties like hardness, impact strength, compression strength, and wear rate of the Mg-Cu alloy and fabricated composites were investigated. The results showed that the addition of weight percentage of 15% TiB_2 increased the hardness value about 58.56 HV, due to better bonding between the Mg-Cu and TiB_2. Further, impact and compressive strengths improved, as the weight percentage of TiB_2 increased. Uniform distribution of reinforced particles enhanced the impact strength and the work hardening effect improved the compression strength. Moreover, the wear rate decreased about 0.0112 mg by the addition of weight percentage of 15% TiB_2. X-ray diffraction (XRD) analysis was carried out for each composition. Optical microscopy, scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) tests were conducted to study the characterization of the base alloy and the prepared new composite.

Keywords: *Magnesium; Titanium diboride; Powder metallurgy; Mechanical properties; Wear*

1. Introduction

In the recent years, the modern aircraft, automobile, and marine industries have been looking towards lighter weight and superior strength materials to enhance their overall efficiency [1-3]. Magnesium (Mg) alloys are continuously substituting the aluminium and steel in the aeronautical/aerospace and automotive industries, and plastic in the electronic and computer industries, owing to their lighter weight and excellent thermal conductivity as well as electrical conductivity. Other foremost advantages of Mg and its alloys are fine machinability, excellent castability and high die casting capability [4-6]. Fevzi Bedir [7] studied tribological properties of Al-Cu-SiC_p and Al-Cu-B_4C_p composites manufactured by powder metallurgy method and they have concluded that the addition of copper particles in a metallic matrix has resulted in enhanced wear resistance when compared to the base matrix alloy. Gökçe et al. [8] studied the effect of copper particles on microstructure and physical properties of Al_5Cu and $Al_5Cu_{0.5}Mg$ compositions. They concluded that the presence of 5% Cu particle in the metallic matrix offers quite significantly increased the hardness and strength of the alloy.

The study of Hassan and Gupta [9] has shown the fabrication of Mg-Cu composites through disintegrated melt deposition method and they reported that the mechanical behavior of the composites is improved for the addition of Cu particles. Sun et al. [10] identified that the copper particles reinforced metallic matrix based composites exhibit improved mechanical properties.

In the current scenario, the magnesium metal matrix composites are under serious consideration to replace the traditional engineering materials which have drawn the attention of transportation, defense, structural and non-structural industries, owing to their remarkable combination of superior specific strength, high stiffness, light weight, and enhanced wear resistance compared to the unreinforced alloy matrix

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*Corresponding author: stalin1312@gmail.com

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[11-13]. The synthesizing techniques play a crucial part in developing magnesium matrix composites with better properties. The foremost challenges faced during the preparation of magnesium composites are wettability, oxidation and dissemination of the reinforcement particles in the matrix. Several processing routes such as rheo casting, stir casting, powder metallurgy, spray deposition, liquid infiltration, and squeeze casting, etc., have been attempted and discussed to synthesize ceramic particles reinforced magnesium matrix composites [14, 15].

Among those techniques, powder Metallurgy (PM) method is the most preferred processing technique for preparing magnesium matrix composites (MMCs). Powder mixing or blending, consolidation, and sintering are the several steps involved in the process of preparing powder metallurgy and secondary operations like forging, extrusion, rolling, etc. are required to attain near-net-shaped powder metallurgy parts. The main advantage of the powder metallurgy method is that the homogeneous dispersion of reinforcement particles in the matrix can be achieved [16, 17]. The properties of composites are desired by the size, shape, mass fraction of the reinforcement, matrix material, and reaction at the interface. The interface between the matrix and the reinforcement serves a pivotal role in determining the properties of MMCs. MMCs can be reinforced with silicon carbide (SiC), boron carbide (B_4C), aluminium nitride (AlN), titanium carbide (TiC) and aluminium oxide (Al_2O_3). Very few studies have been conducted on TiB_2 reinforced MMCs, because of the high cost of TiB_2 powders. TiB_2 has been employed as a to produce MMCs due to its superior hardness, low density, high elastic modulus, and remarkable wear resistance [18, 19].

Only a few research findings on processing and the characterization of magnesium alloys reinforced with ceramic particulate composites have been stated so far. Poddar et al. [20] manufactured AZ91D/SiC composites using rheo casting methods and studied the mechanical behavior of the composite. They also stated that the presence of SiC particulate leads to significant enhancement in hardness and yield strength whereas tensile strength and ductility decrease. Shen et al. [21] prepared the AZ31B/SiC composites using semisolid stirring assisted ultrasonic vibration method and examined the mechanical properties and the microstructure of the MMCs. Nguyen and Gupta [22] synthesized AZ31B/Al_2O_3 composites by the disintegrated melt deposition method and they observed an increase in hardness and compression strength of the composites with an increase in the volume percentage of Al_2O_3 particulates in the aluminium matrix. Nie et al. [23] exhibited the effects of SiC particles on the mechanical behavior of the AZ91/SiC composites and they concluded that the mechanical properties increase along with the increasing volume fraction of the reinforcements compared to base cast AZ91 matrix alloy. Nguyen et al. [24] produced the AZ31B/Al_2O_3 magnesium matrix composites using the disintegrated melt deposition method and they analyzed the oxidation resistance and microstructural characterization of the composite.

Turan et al. [25] prepared Mg alloy reinforced with fullerene by the powder metallurgy method. Purohit et al. [26] manufactured Mg/SiC composites by powder metallurgy process and they evaluated the physical and mechanical properties of the composites. Zhao et al. [27] prepared AZ91D magnesium alloy reinforced with Al_2O_3 composite by the squeeze casting technique and they noticed that the microhardness, the yield strength and the ultimate tensile strength increase with the increase Al_2O_3 particles. Kaviti et al. [28] investigated the tribological behavior and the wear characterization of Mg/BN composite produced by the powder metallurgy technique and its tribological properties were tested employing pin-on-disk equipment. Kumar et al. [29] synthesized AZ91/SiC composites by the stir casting technique and they investigated the microstructure and tensile strength of the composites.

To the best of the researchers' knowledge, very limited levels of literature related to the mechanical behavior of the composites reinforced with titanium diboride particle are available. No systematic investigation has been done on the Mg/TiB_2 composite fabricated by powder metallurgy route. In the present study, a novel attempt has been made to fabricate Mg/TiB_2 MMCs with various weight fractions ranging from 0 to 15%.

2. Experimental details

Magnesium of 99% purity bought from Perfect Tubes, Mumbai, India, was used as the matrix. Copper and titanium diboride were purchased from Parshwamani Metals, Mumbai and Momentive, Ludhiyana, respectively. All the primary and secondary materials were 60µm in size. The composites were fabricated by powder metallurgy route [30]. A constant weight fraction of 5% copper was used in all the compositions. In general, copper reduces the resistance to general corrosion of magnesium alloys, but increases the resistance to stress corrosion. The 5% Cu was chosen from the previous literatures [7, 8] to improve the mechanical properties without compromising the ductlity of the composites. The weight percent of TiB_2 was increased in the range 0%, 5%, 10% and 15%.

After carefully weighing the four compositions, each composition was ball milled at 400 rpm for 6 hours in a planetary ball milling machine. Zirconia balls were employed. The ball milling was carried out in the ethyl alcohol medium. Once the milling was over, each composition was compacted in a 10 ton capacity hydraulic press. The compaction was made at 6 ton pressure and the dwell time of the compaction was about 12 seconds. The size of the die was about 20 mm diameter and 15 mm length. Followed by the compaction, the specimens were sintered in a furnace in the argon gas medium in order to avoid the burning of magnesium. The specimens were heated to about 500°C. The temperature gradually increased at the 2°C/min and the heating time consumed more than 2 hours to attain 500°C. All the four specimens were sintered in the same way.

The microstructures of the specimens were observed using optical microscope. The SEM micrograph test was conducted with VEGA3 TESCAN. SEM and EDX measurements were taken to investigate the microstructure. SEM analysis was carried out to identify the orientation of the materials. Phase identification was performed on the specimens with the highest mechanical strength using X-ray diffraction analysis. Mechanical properties were examined using Vickers hardness measurement at room temperature. The impact strength and compression strength tests were also performed [34]. The compression test was carried out by using a universal testing machine at Delta metallurgical lab Chennai. The test was carried out using ASTM E9 standard.

Dry sliding wear tests were performed in accordance with the ASTM G99-05 test standards employing pin-on-disk apparatus. The counter disc material was made of EN31 steel. The wear test was done with the following parameters such as abrasive grid size 220 mm, sliding velocity 1 m/s, applied load 10 N, 20 N, 30 N, and sliding distance 1500 m.

3. Results and discussion

3.1 Microstructure analysis of Mg-Cu alloy-TiB$_2$ composites

Microstructure analysis was carried out using the optical Microscopy technique and the results are shown in Figs. 1a-d. Figure 1a shows the secondary phase precipitate along the grain boundary in the Mg matrix. Figure 1b presents the appearance of fine

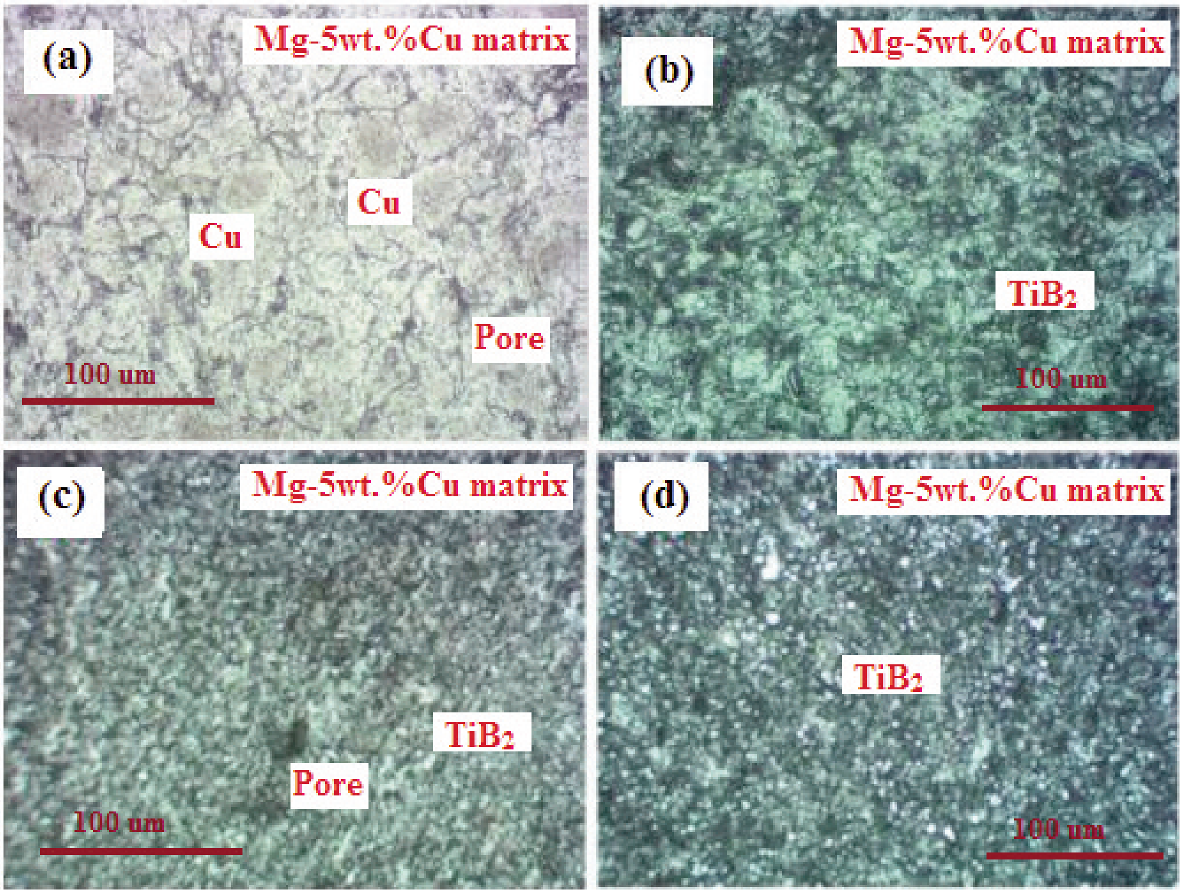

Figure 1. *Image of optical microscopy of (a) parent material (b) Mg/Cu+5%TiB$_2$ (c) Mg/Cu+10%TiB$_2$ (d) Mg/Cu+15%TiB$_2$ respectively*

irregular precipitates of Cu and globular particles of TiB_2. Similarly, in Figs. 1c and 1d, the appearance of fine precipitates of the reinforcement can be seen. A little amount of porosity is observed in the samples. The pores are distributed arbitrarily and they exhibit an average size. The specimen comprising 15% TiB_2 shows the perfect orientation of the reinforcement materials and it is a desirable property required by a composite (Fig. 1d). The increase in the addition of weight percentage of TiB_2 occupies the pores and it acts as a pore closer for the Mg matrix composite [35].

The Figs. 2a-d reveal the location and characterization of the SEM micrographs of the composites with different compositions of TiB_2 (a) Parent alloy (b) Mg/Cu+5%TiB_2 (c) Mg/Cu+10%TiB_2 and (d) Mg/Cu+15%TiB_2. The SEM image clearly displays the uniform distribution of the copper that enhances the mechanical and tribological properties. The addition of 10% TiB_2 to the Mg-5% Cu alloy makes the bonding stronger, as the density of TiB_2 is

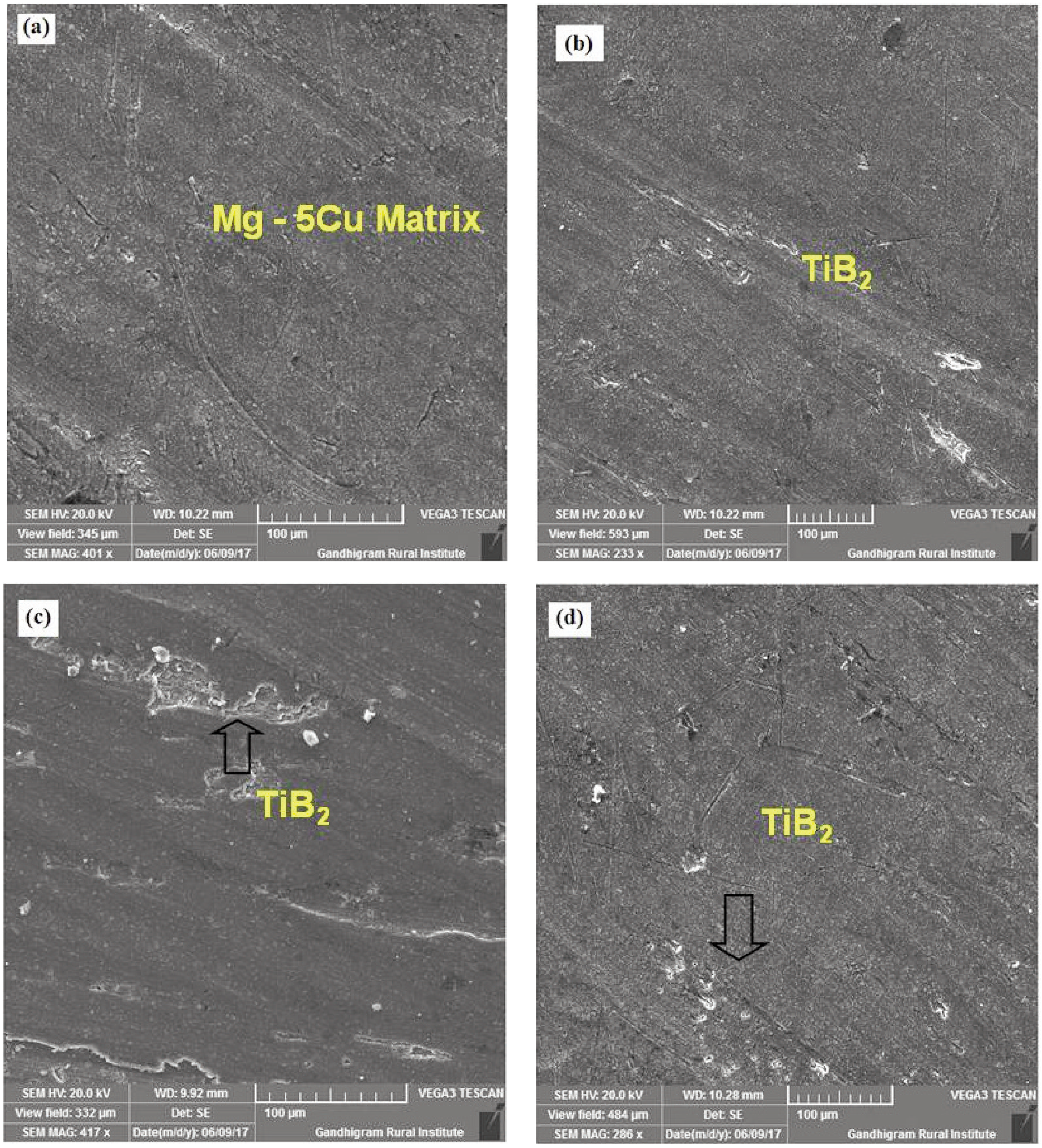

Figure 2. *SEM micrographs of the composites with different composition of TiB_2 (a) parent alloy (b) Mg/Cu+5%TiB_2 (c) Mg/Cu+10%TiB_2 (d) Mg/Cu+15%TiB_2*

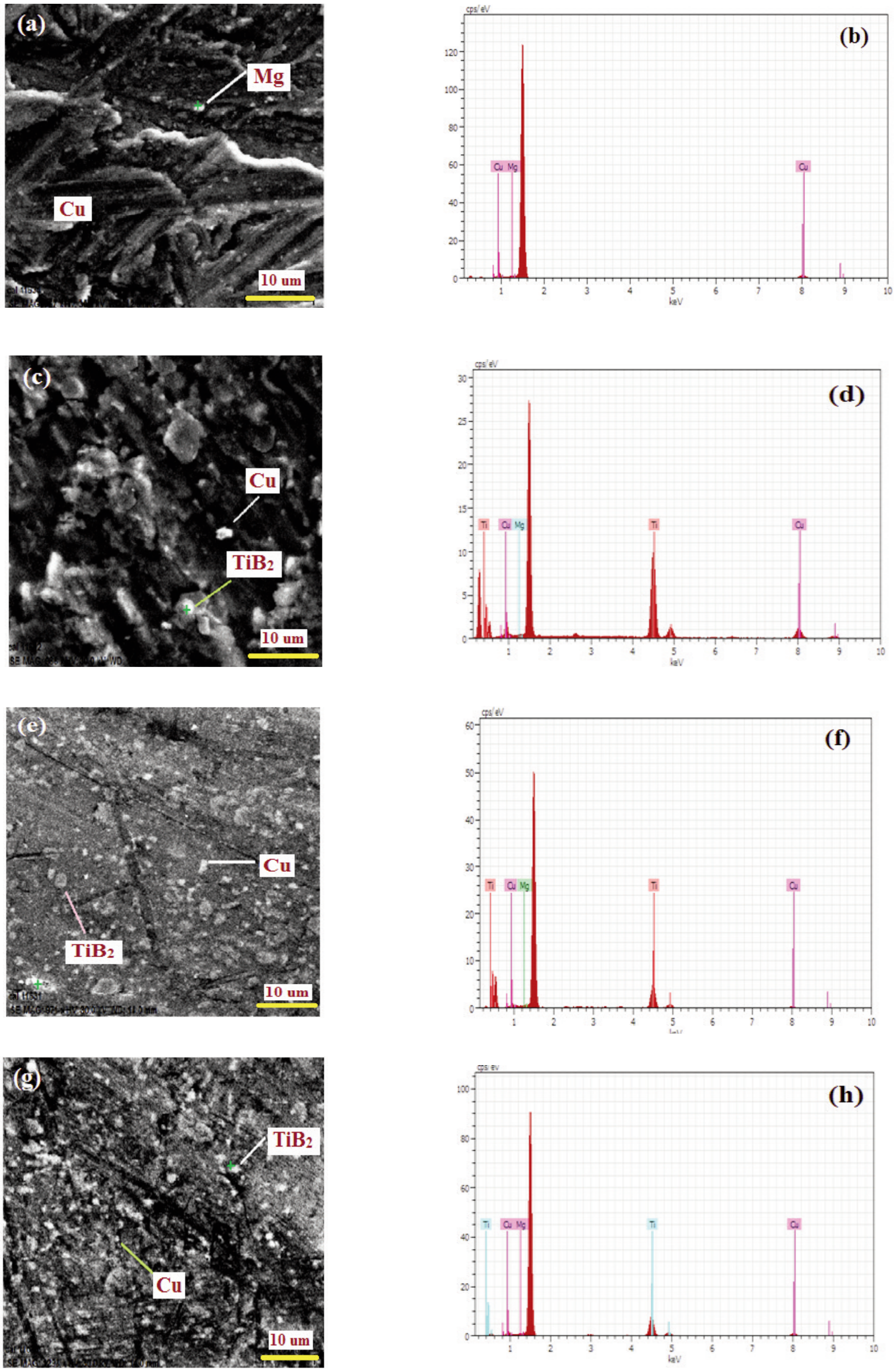

Figure 3. *SEM micrographs with EDX analysis of matrix alloy and Mg/Cu/TiB$_2$ composites (a & b) parent alloy (c & d) Mg/Cu+5%TiB$_2$ (e & f) Mg/Cu+10%TiB$_2$ (g & h) Mg/Cu+15%TiB$_2$*

high [11]. The SEM image of the composition with 90% Mg, 5% Cu and 5% TiB_2 is illustrated in the Fig. 2b. The images clearly depict the proper orientation of the Mg, Cu, and TiB_2 throughout the surface of the matrix. The SEM micrographs of the compositions that are 85% Mg, 5% Cu, 10% TiB_2, 80% Mg, 5% Cu, and 15% TiB_2 are illustrated in Figs. 2c and 2d, respectively. The results show that the reinforcement materials are uniformly distributed along the matrix. There is no identification of any interstitial compounds like porosity which means that there is a good interfacial bonding between the materials [18]. This is because of the unique characteristics of the powder metallurgy feature that produces materials with the uniform distribution of other elements

The final composition of the matrix with 5% Cu and 15% TiB_2 has exhibited a uniform distribution leading to a very fine equiaxed grain orientation. Though the composite exhibits higher weight fraction than the other, there is no agglomerating regions found in the fabricated composite. Homogenization of the reinforcement materials and the matrix material, the during ball milling led to the fine structure of the materials. It enhances the fine particle distribution and provides a good interfacial bonding. These results well agreewith the findings of Nie et al. reported for the SiC nanoparticles reinforced magnesium matrix composites [4].

Energy dispersive x-ray diffraction test conducted on the specimens shows the orientation of the reinforcement material along the surface of the matrix. The EDS image for the specimen 1 and the details of the testing parameters are portrayed in Fig. 3a (512x etched image). The area marked with the green cross in Fig. 3a defines the orientation of Cu with the Mg matrix. Figures 3c-d depict the influence of TiB_2, as it gets increased in weight fraction of the composite. All the Figs. 3e-h ensure the presence of desired elements to be presented in the produced composites. The increased intensity of the peaks confirms the incorporation of a large quantity of reinforcement with the matrix.

The XRD results displayed in the Figs. 4a-d demonstrate the XRD pattern of the Mg-Cu alloy and its composites. Figure 4a shows extra peaks that can be assigned to Cu which is small in size. The presence of TiB_2 is ascribed to the slight addition of TiB_2 in the composite (Fig. 4b), whereas there are no extra peaks found in the XRD spectrum. It indicates that there is no chemical reaction between the reinforcement materials, and it is beneficial for maintaining the bioactivity of the matrix (Fig. 4c). The highest peak is attributed to Mg in all the images. The second high peak in Fig. 4a shows a diffraction pattern of Cu. Figures 4b-d show the second highest peak that is attributed to TiB_2 followed by Cu. The intensity of Mg peak reduces drastically. It shows that the intensity of Mg is presumably associated with reducing the weight fraction of Mg in the composites. The intensity of TiB_2 increases with an increasing addition of weight fraction of TiB_2 in the alloy matrix.

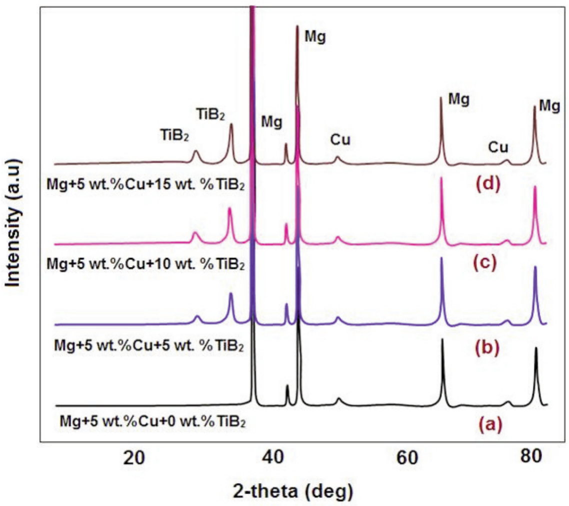

Figure 4. *XRD pattern of matrix alloy and Mg/Cu/TiB_2 composites (a) Mg/Cu+0%TiB_2 (b) Mg/Cu+5%TiB_2 (c) Mg/Cu+10%TiB_2 (d) Mg/Cu+15%TiB_2*

3.2 Mechanical properties of Mg-Cu alloy-TiB_2 composites

Figure 5 depicts the effect of the increasing weight fraction of TiB_2 on the hardness of the composite. Pure Mg with 5% copper has resulted in a hardness value of 23.75 HV. It gradually increases with the addition of 5% TiB_2 and reaches a value of 32.99 HV, which is an appreciable enhancement. By further increasing the weight fraction of TiB_2 to 15%, the hardness value reaches a maximum of 58.56 HV. The

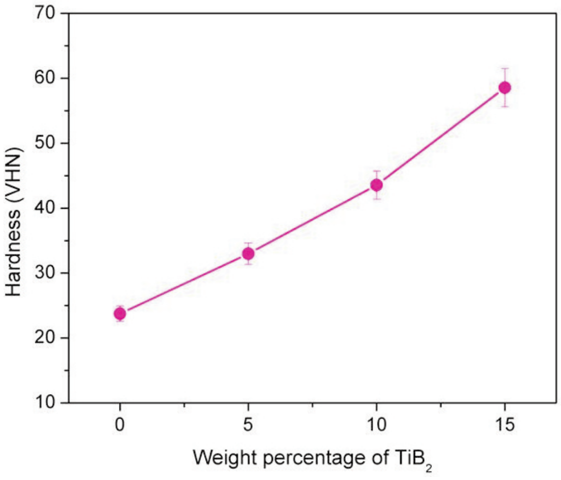

Figure 5. *Variation of hardness with varying content of TiB_2 particle*

interface between the matrix and the reinforcement phase plays a major role in enhancing their mechanical and tribological properties [31, 32]. In the present study, the interface between the Mg-Cu matrix and the reinforcement is good, as shown in Figs. 1b-d. From this, it is concluded that the addition of 15 wt.% of the TiB_2 has proved the enhanced hardness of the composite. Moreover, the increase in micro-hardness of the composites containing TiB_2 particles is more enhanced resistance against plastic deformation.

The effect of TiB_2 on compressive strength of the composite is shown in Fig. 6. The compressive strength is high, when 95% Mg is reinforced with 5% copper, and it results in 632.91 MPa. But, the compressive strength of the composite gradually increases with the increase in weight percentage of TiB_2. It is well known that the strengthening effect of the composite depends on (a) load-bearing effects, due to the presence of reinforcements, and (b) Hall–Petch effect, due to the grain size refinement and the generation of geometrically necessary dislocations to accommodate thermal and elastic modulus mismatch between the matrix and reinforcements [31]. The reason for increasing compressive strength is that the work hardening occurs, when the composite is strained. The strain disparity between the matrix and the reinforcement usually creates a higher density of dislocations in the matrix around the reinforcement [33]. These results are well agreed with Aatthisugan et al. reported for the boron carbide reinforced Mg matrix composites.

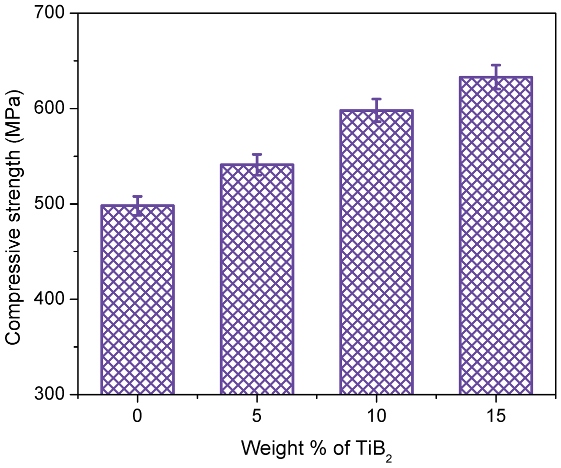

Figure 6. *Variation of compressive strength with varying content of TiB_2 particle*

The impact strength of the composites was determined by using Izod and Charpy impact testing equipments. The effect of weight percentage of TiB_2 is presented in Fig. 7. The results show that the impact value is about 3 Joules for the first specimen with 95% Mg and 5% Cu, whereas the weight fractions of 5%, 10%, and 15% TiB_2 result in improved impact strengths. The reason for the improvement in impact strength is the uniform distribution of hard reinforcement particles in the Mg matrix as shown in the microstructure Fig. 7. In general, the mechanical bonding between the magnesium and filler particles leads to an increase in the strength of the developed composites. In this case, the increase in TiB_2 content decreases the inter particle spacing and porosity and increases the strength of the Mg matirx composites [16]. The strengthening mechanisms involve the increase of mechanical properties which are Orowan strengthening, solid solution strengthening, load-bearing effects, and grain boundary strengthening, as reported by the previous researchers [36, 37].

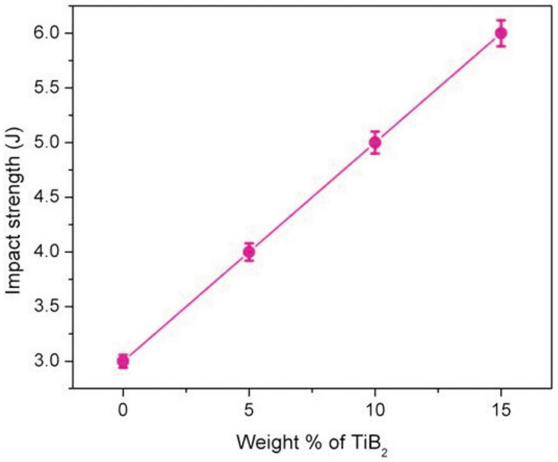

Figure 7. *Variation of impact strength with varying content of TiB_2 particle*

3.3 Wear behavior of Mg-Cu alloy-TiB_2 composites

The wear characteristics of the composites have been analyzed using a pin on the disc machine. The wear test is performed with the following parameters such as applied load 10 N, 20 N, 30 N, and sliding distance 1500 m. The results confirm that as the weight percentage of TiB_2 increases, the wear rate of the composite also decreases. As a result, the wear rate is reduced with the presence of TiB_2 at a load of 30 N and the wear rate is about 0.00885 x 10^{-3} (mm^3/m) but with 15% of TiB_2, at a load of 10 N and the wear rate of the composite is appreciably reduced to 0.006822 x 10^{-3} (mm^3/m). The effect of weight percentage of TiB_2 on the wear rate is shown in Fig. 8. The reason for the decrease in the wear rate is that there is a strong interfacial attachment of the TiB_2 reinforcement materials with Mg-5% Cu alloy.

The compression and impact strengths of the composite get increased with an increase in the weight percentage of TiB_2. The hard nature of TiB_2 particles acts as a wear resistant agent for the matrix materials.

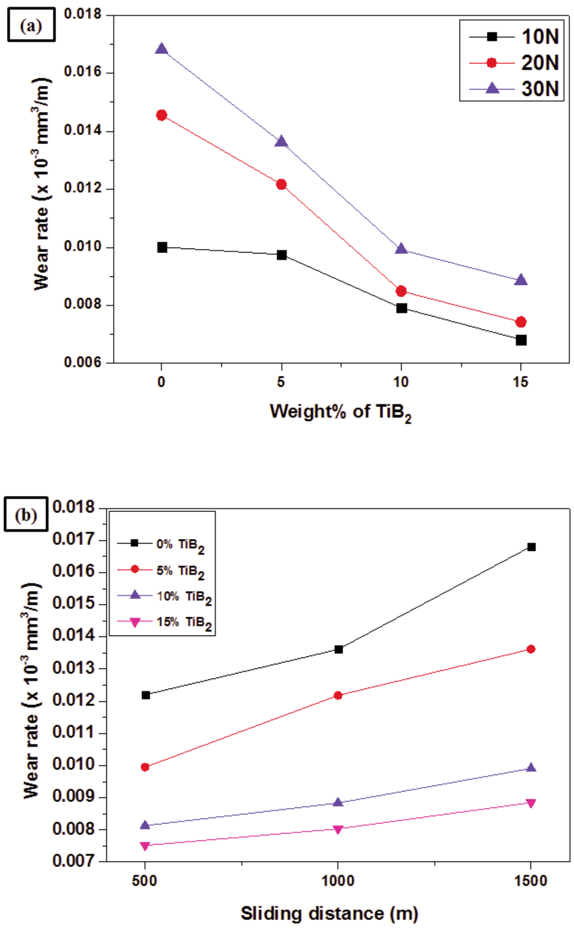

Figure 8. *(a-b) Variation of wear rate with varying content of* TiB_2 *particle*

Figures 9a-c show the worn surface morphology of the composites. From Fig. 9, it is clearly observed that the nature of the wear is abrasion because of the addition of TiB_2 particles. The worn surface of Mg/Cu+TiB_2 composites exhibit the parallel grooves - like pattern without the occurrence of plastic flow is revealed in Fig. 9. It is also noticed that the wear debris is loose in nature and non-adherent with the matrix because of the enriched hardness of the magnesium matrix owing to the addition of TiB_2 particles in the Mg matrix. Hence, the abrasive wear mechanism is observed for the composite samples. The reason for the abrasive wear in the composite samples is that the presence of hard TiB_2 particles and ploughing is observed during the sliding of the pin over the sample surface. Moreover, the mechanically mixed layer can be observed in Fig. 9a and the wear track and few wear debris are seen in Figs. 9b-c. Therefore, the micrographs are evidence that the addition of TiB_2 particles reduces the wear rate of the prepared composites.

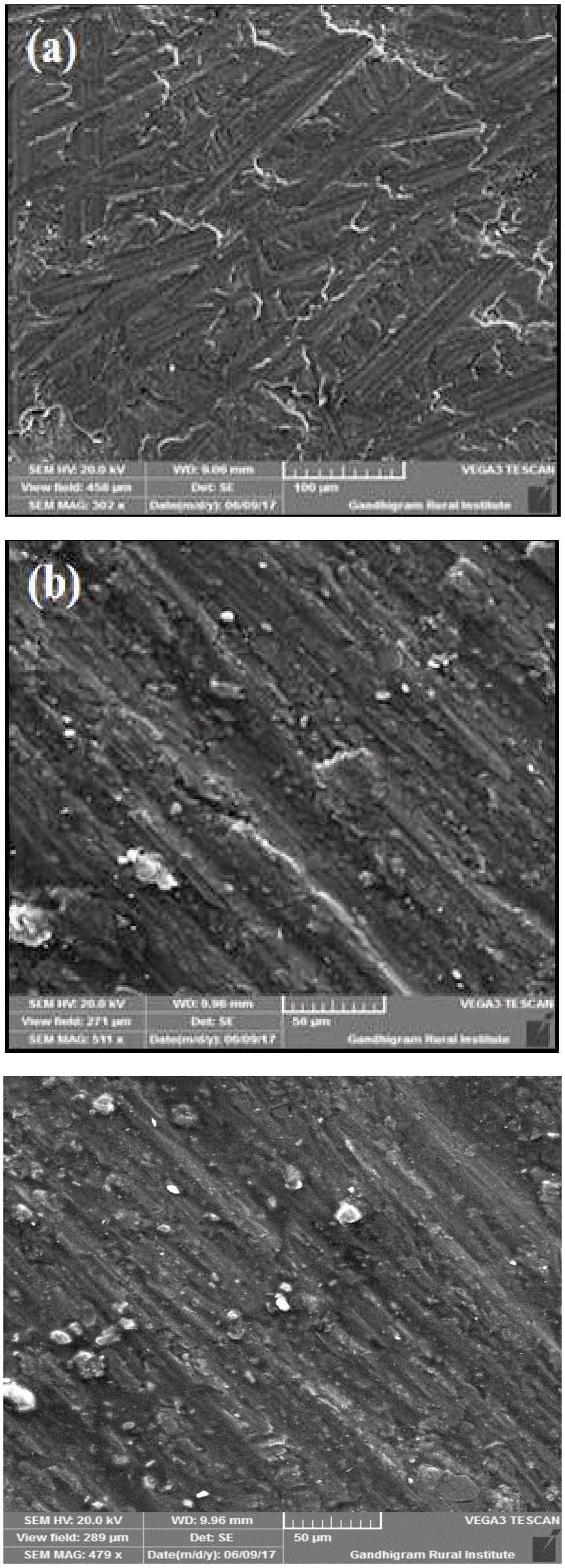

Figure 9. *Wear surface morphology of matrix alloy and Mg/Cu/TiB_2 composites (a) Mg/Cu+5%TiB_2 (b) Mg/Cu+10%TiB_2 (c) Mg/Cu+15%TiB_2*

4. Conclusions

The microstructure, mechanical properties, and wear behavior of Cu and TiB_2 reinforced magnesium matrix were fabricated via powder metallurgy route and analyzed. The main investigations of the research are as follows:

- Microstructure reveals the fine particle distribution and good interfacial bonding between the reinforcement and the matrix.
- Impact strength, compressive strength, and hardness increase with the increase in the weight percentage of TiB_2 in Mg-Cu alloy matrix.
- Wear rate is appreciably reduced, as the percentage of reinforcements increases. It shows that the wear resistance of the fabricated matrix is increased.
- The composite with 15% TiB_2 offers superior mechanical properties on all aspects.
- While analyzing all the mechanical property results and the microstructure results, it is suggested that the new composite Mg-5% Cu-15% TiB_2 by powder metallurgy route exhibits desirable properties for a new composite.
- The composite with such admirable properties is suitable for light weight and high strength requiring applications. The future work can be concentrated on producing composites which could be used to make light weight automotive chassis.

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ISPITIVANJE MIKROSTRUKTURE I MEHANIČKIH OSOBINA Mg-5WT.%Cu-TiB_2 KOMPOZITA PROIZVEDENIH METALURGIJOM PRAHA

B. Stalin [a,*], **M. Ravichandran** [b], **V. Mohanavel** [c], **L.P. Raj** [d]

[a]Ana univerzitet, regionalni ogranak Madurai, Odsek za mašinstvo, Madurai, Indija
[b] K.Ramakrišnan fakultet inženjerstva, Odsek za mašinstvo, Tiručirapali, Indija
[c] Kingston fakultet inženjerstva, Odsek za mašinstvo, Velore, Indija
[d] Čenduran fakultet inženjerstva i tehnologije, Odsek za mašinstvo, Pudukotai, Indija

Apstrakt

Kompozit sa matricom magnezijumske legure ojačan masenim udelom od 5% Cu i različitim masenim udelima od (0%, 5%, 10%, 15%) titanijum diborida (TiB_2), proizveden je metalurgijom praha. U ovom radu su ispitivane mehaničke osobine kao što su tvrdoća, udarna čvrstoća, kompresiona čvrstoća, i stopa habanja Mg-Cu legure i proizvedenog kompozita. Rezultati su pokazali da je dodavanje masenog procenta od 15% TiB2 povećalo vrednost čvrstoće oko 58.56 HV usled boljeg vezivanja između Mg-Cu i TiB_2. Uz to, udarna čvrstoća i kompresivna čvrstoća su poboljšane kako je maseni procenat TiB_2 rastao. Ravnomerna distribucija ojačanih čestica povećala je udarnu čvrstoću, a efekat hladnog otvrdnjavanja poboljšao je kompresionu čvrstoću. Štaviše, stopa habanja smanjena je za 0.0112 mg dodavanjem masenog procenta od 15% TiB_2. Urađena je rendgenska difrakciona analiza (XRD) za svaki kompozit. Optička mikroskopija, skenirajuća eelektronska mikroskopija (SEM) i energetsko disperzivna spektroskopija (EDS) su korišćene za testove da bi se proučila karakterizacija osnovne legure i pripremljenog novog kompozita.

Ključne reči: *Magnezijum; Titanijum diborid; Metalurgija praha; Mehaničke osobine; Habanje*