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Influence of AZ91 alloy reinforced with nano B₄C particles on microstructural characterization, hardness and tribological properties prepared through powder metallurgy

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Abstract

In this present work, a milled B₄C nanoparticle was reinforced into the AZ91 alloy with different weight percentages (5, 10, 15, and 20%) by powder metallurgy. XRD and SEM for the crystalline behavior and morphology of the AZ91-xB₄C composite. The wear resistance on the load and Sliding Distance (SD) of the specimen has been experimented with the pin-on-disc apparatus and Vickers hardness machine to measure hardness. Wear loss decreased gradually with the addition of milled B₄C nanoparticles is identified for AZ91-xB₄C nanocomposites. Coefficients of Friction (CoF) increased with an increase in load for AZ91-xB₄C nanocomposites. Microhardness was linear with the increase in the wt. % of milled B₄C nanoparticles. The worn surface micrograph was also studied using a scanning electron microscope.

1. Introduction

Over the past few years, the research on Particulate Reinforced Metal Matrix Composite (PRMMC) owing to potential applications in the automobile, aerospace, electrical, and electronics industries. PRMMC can control grain size, increase load transfer effect, and has nucleation effect due to the thermal mismatch [1]. According to the earlier reporters [2], AZ91 is the important matrix for hybrid composite due to its inherent properties like high specific strength and corrosion. Several reinforcement particles such as TiC [3], SiC [4], MWCNT [5], and Al₂O₃ [6] have been studied in earlier days. In addition to this, B₄C can be selected as reinforcement owing to its high strength and wear resistance. Magnesium-based hybrid composites have potential applications in brake assemblies and engine blocks with high specific strength, modulus with a high wear resistance [7]. Researchers also improved the composite technology to a high-performance composite reinforced with carbides to enhance mechanical properties [8–11]. Numerous research works have been carried out for the preparation of AZ91 based composite by techniques such as Powder Metallurgy (P/M) [12], squeeze casting [13], stir casting [14], an ultrasonic method [15], and spray forming [16]. The powder metallurgy method offers many advantages to the uniform distribution with low-temperature requirements for making different composites. Mg alloy has an excellent mechanical behavior comparing to magnesium. Magnesium and its alloys have drawn various interests when reinforcing B₄C nanoparticles [17]. The wear resistance of the Mg composite enhances with increasing amounts of strengthening, which were under the higher Sliding Speeds [18].

In this regard, the present work aims to prepared the AZ91-B₄C composite by P/M. Moreover, the influence of B₄C concentration on the mechanical properties of AZ91 composite with different load and sliding distances.

2. Experimental work

2.1. Sample preparation method

Magnesium alloy AZ91 powders with a particle size of 50 μm having a purity of 98.5% as a base matrix. Boron carbide with a particle size of 10 μm from M/s. Sigma Aldrich, USA. Morphology of the purchased AZ91 alloy (figure 1(a)) indicates the curved ribbon-like flakes, and the SEM image of the purchased B₄C particle in (figure 1(b)) displays the
rhombohedral structured particles. The purchased B₄C particle was milled in a high-energy ball milling process to reduce the size of the B₄C particle; the morphology of the particle is shown in figure 1(c). The size of the milled B₄C particle is perceived in figure 1(d) by using the Zeta size analyzer, and the value is 180 nm. The mechanical alloying of AZ91-xB₄C compositions (x = 5, 10, 15, and 20 wt %) with a high-energy ball milling process. The SEM micrograph of mixed powders of the composition is in figure 2. The resultant homogenous powder was modified in the form of a pellet by a die set assembly. The compaction of the powder particles in a compression testing machine with a capability of 1000 kN; the specimen is a circular-shaped pellet with dimensions of 10 mm Φ and 20 mm height. The sample was heated in a muffle furnace at 520 °C for two hours in an argon gas atmosphere and cooled and cleaned for density, hardness, and wear testing.

2.2. Microstructural characterization
X-ray diffraction (XRD) of AZ91 nanocomposite among the diffraction angles 20°–80° with a step size of 3° min⁻¹. The microstructural investigation of powder particles and wear test samples images by using SEM.

2.3. Density and hardness
The density of the nanocomposites by Archimede’s principle as per the ASTM standard: B962-08; the specimen measured density with different compositions summarized in table 1. It reveals that the theoretical density was very close to the experimental density with a permissible range error. It indicates the good compactness of the specimen. Sintered nanocomposite specimen using emery paper to determine the micro-hardness. Micro-hardness investigated by Vickers hardness machine.

2.4. Wear test
The wear test has conducted in pin-on-disc equipment by the ASTM: G99-05. The load is 5 to 25 N with an addition of 5 N at a constant Sliding Velocity (SV) of 2.61 m s⁻¹ and SD of 1500 m. Before the test sample, and disc face dressed with acetone to remove the stains present in them. The CoF has calculated by the frictional force calibrated by the machine divided by the applied load. Worn surface morphology of Mg alloy AZ91 perceived from the SEM [3].

Figure 1. SEM image of powders, (a) Mg alloy AZ91, (b) micro B₄C, (c) milled nano B₄C and (d) particle size of milled B₄C nanoparticle.
Figure 2. SEM image of mixed composition of powders (a) AZ91-5B₄C, (b) AZ91-10B₄C, (c) AZ91-15B₄C, (d) AZ91-20B₄C.

Figure 3. XRD pattern of AZ91-xB₄C magnesium alloy nanocomposites.

Table 1. Outcomes of density and hardness.

| Sl.no | Composition | Theoretical density (g cm⁻³) | Experimental density (g cm⁻³) | Micro-hardness (HV) |
|-------|-------------|-----------------------------|------------------------------|-------------------|
| 1     | AZ91        | 1.800                       | 1.62                         | 71                |
| 2     | AZ91-5B₄C   | 1.836                       | 1.69                         | 83                |
| 3     | AZ91-10B₄C  | 1.872                       | 1.74                         | 91                |
| 4     | AZ91-15B₄C  | 1.908                       | 1.79                         | 97                |
| 5     | AZ91-20B₄C  | 1.944                       | 1.83                         | 105               |
3. Results and discussion

3.1. X-ray diffraction study

X-Ray Diffraction (XRD) pattern of AZ91-xB4C composite powders with varying reinforcement weight percentage shown in figure 3. Diffraction results of magnesium alloy AZ91 show the characteristic peaks of Mg, Al, and Zn with diffraction angles (2θ) of 36.9, 38.1, and 43.2. According to the JCPDS files (851327, 894894, and 870713), Al, Mg, Zn elements present in the plane as (101), (111), and (100), respectively. Mg element has a higher peak when compared to that of Al and Zn. The wt.% fraction reflects the intensity of diffraction peaks. As the reinforcement increases, the peaks correspond to the Mg element reduced, and the B4C particle peak is enhanced. Apart from the characteristic peaks, no other peak in the XRD results. XRD indicates no chemical reaction occurred during the composite preparation between milled B4C nanoparticles and AZ91 Mg alloy.

3.2. Microstructural analysis

The sintered samples were polished via a standard metallographic process, using an etchant solution [19]. The microstructure of the Mg alloy AZ91-xB4C nanocomposite has shown in figures 4(a)–(e). The honeycomb-like morphology with a bimodal distribution in the nanocomposite. Figure 4(a) demonstrates that the presence of voids with oxides might be due to the intrinsic nature of magnesium alloy. The micrograph exposes an

Figure 4. SEM image of (a) AZ91, (b) AZ91-5B4C, (c) AZ91-10B4C, AZ91-15B4C and (e) AZ91-20B4C.
inhomogeneous distribution of reinforced milled B$_4$C nanoparticles. The magnesium matrix with irregular mesh sizes, showing characteristic string microstructures [20]. It is evident that a formation of nanocomposites with good compatibility. The voids, microporosity in the microstructure decreased with increasing milled B$_4$C nanoparticle content. The milled B$_4$C nanoparticles effectively fill the gap in the AZ91. In this fact, AZ91-20B$_4$C presents fewer voids while comparing to all other compositions. These behaviors could reflect the density and porosity studies for the sintered specimen.

### 3.3. Density and hardness

The density and microhardness of Mg alloy in different wt. % of B$_4$C nanocomposites shown in table 1. Figure 5(a) illustrates that an increase in density of AZ91-xB$_4$C nanocomposites with increases in weight % of
reinforcement. The composite with milled B$_4$C nanoparticles is very high compared to AZ91. The discrepancies between the observed and theoretical density of the composite are associated with porosity in the specimen. The improvement of the microhardness of the composite has been increasing for the milled B$_4$C nanoparticles in figure 5(b). Comparing the hardness value of all the nanocomposites, AZ91-20B$_4$C has the highest value Mg alloy AZ91. Such an increase in microhardness values owes to the reinforcement of a phase leading to localized matrix deformation on indentation is carried out [18].

The strain, work hardening, and microhardness of the composites increase with increasing in B$_4$C powder. It can also improve the surface microhardness of the nanocrystalline microstructure with severe plastic strain. The repelling of plastic deformation has improved the microstructure to the grain boundaries and the dispersion of milled B$_4$C nanoparticles reinforcement in the matrix material [21].

3.4. Effect of reinforcement, applied load, and sliding distance on wear loss

Dry sliding wear tests were done as per the standard ASTM G99-05 using pin-on-disc apparatus. The EN31 steel disc with hardness HRC 61. The compositions were dressing with acetone before testing the pin and disk surface. The AZ91-xB$_4$C nanocomposites specimen weigh before and after test to a precision of 0.001 g electronic weighing balance apparatus to calculate the quantity of wear loss [10–23]. Wear loss for the AZ91-xB$_4$C nanocomposites with weight percentages of reinforcements in figures 6(a)–(c). The test conducted by the parameters such as SV (2.61 m s$^{-1}$), SD (1500 m), and the load of 5, 10, 15, 20, and 25 N. Figure 6(a) shows that the wear loss diminishes with increasing the wt.% of incorporating B$_4$C can act as a solid barrier on the matrix surfaces. The wear resistance of the nanocomposites rises with a rise in B$_4$C nanoparticle content. It may originate from any one of the phenomena as follows: (a) enhancing the hardness of the nanocomposites with the rise of B$_4$C particle, (b) the area of contact among the matrix of the nanocomposite and countersurface decreases with the addition of the B$_4$C nanoparticles, and (c) rising the ability to bear loads, reinforce matrix and avoid plastic deformation of B$_4$C nanoparticles with the rise of B$_4$C content [3].

Figure 6(b) demonstrates the wear loss for nanocomposites with loads of constant SV (2.61 m s$^{-1}$), SD (1500 m), 5 N load occurs a diminish in the volume of wear loss than the load to insignificant ploughing action. The chance of metal-to-metal contact raised with rising loads. The increase in wear loss of AZ91 magnesium alloy composite at high loads causes cracks on the surface. The AZ91-20B$_4$C nanocomposite
shows a lower value of wear loss when compared with another AZ91 magnesium-based alloy matrix. Figure 6(c) shows the specifics of wear loss for various composites by changing the SD at SV (2.61 m s\(^{-1}\)) and load (25N). The wear loss increased with increasing the sliding distance for all the compositions; the lubricant layer at lengthier sliding distances to the inconvenience of necessary solid barrier on the surface contact. The addition of nano B\(_4\)C will decrease the wear loss of the nanocomposites and increase the wear resistance [20].

3.5. Effect of reinforcement, applied load, and sliding distance on the coefficient of friction

Figures 7(a)–(c) shows the change of Coefficient of Friction (CoF) with the constant SV (2.61 m s\(^{-1}\)), SD (1500 m), and various loads of 5–25 N. It displays that the CoF decreased with increasing weight percent of B\(_4\)C to the improvement of hardness. The friction force in tribo systems frequently results from the grip among counterparts and samples. The literature [19], the decreasing CoF with the rise of B\(_4\)C content decreases the adhesion among the matrix and counterpart. Therefore, interface bonding among B\(_4\)C nanoparticles and matrix could avoid the particulate reinforcement from the matrix and diminish the abrasive wear [3]. The addition of milled B\(_4\)C nanoparticles diminishes the CoF results due to the improvements in hardness. Additionally, the milled B\(_4\)C nanoparticles, existing on the surface of composites as bits [21–23], shield the
matrix from severe contact with the counter surfaces. From figure 7(b) that CoF rises when the load increase for the composites. Moreover, the rise in CoF of the AZ91 alloy is higher than the nanocomposites. The adhesion of B4C nanoparticles could result in diminishing CoF of the AZ91-xB4C nanocomposites [3].

Figure 7(c) demonstrates the CoF for the composites by changing the sliding distance at an SV and applied load. For the smaller SD, the CoF is lesser and rises with the increase in sliding distance. The coefficient of friction decreased for AZ91-20B4C nanocomposite related to the AZ91 magnesium alloy matrix.

3.6. Wear mechanism
The increasing wear resistance is the superior hardness of milled B4C nanoparticles compared to magnesium alloy AZ91. The tribological properties, pin-on-disc are utilized under various parameters. The load, SD, and SV on the wear mechanisms of the tested specimen are in figures 8(a)–(c). Figure 8(a) represents the plastic deformation and cracks along the linear way of the wear test for the magnesium alloy AZ91 sample. Cracks in the core area of the magnesium matrix were visible on the surface. The wear mechanism influencing the test circumstances is the plastic deformation of the Mg alloy AZ91 sample. The worn surface of AZ91–xB4C nanocomposites on wear mechanism from the abrasive wear to delaminating wear. From figures 8(b)–(e) the worn surface layer owing to abrasion to delamination is high on the surface of the specimen. Figure 8(b) demonstrates the worn surface area of the AZ91–5B4C nanocomposites of the delaminated surface. Figure 8(c), milled B4C nanoparticle is improved during the matrix, and deep grooves on the direction of sliding on the surface of the AZ91–10B4C nanocomposites [22]. Worn surface areas are found coarser and have signs of deep grooves sliding together with the disc. In figure 8(d), there were no grooves on the surface area of the AZ91–15B4C composites, but it looks that in the specimen surface of micro pits and milled B4C nanoparticles debris. The deformation on the surface and the B4C nanoparticles act as a lubricating layer on the worn surface to reduce wear. In figure 8(e), lubricating film on the surface area of the specimen prevents high wear rate, oxide particles act as a lubricating agent and reduces the wear rate in AZ91–20B4C composites. The fragmentation and wear debris on the counter surface will protect the worn surface area due to the development of oxide layers and reduce the wear rate [23].

4. Conclusion
The present research shows that the AZ91 magnesium alloy is reinforced milled B4C nanoparticles through the powder metallurgy technique.

- The SEM and XRD confirm that the successful reinforcement of secondary particles in the matrix material.
- The density and hardness of AZ91-xB4C nanocomposites were increased owing to the incorporation of milled B4C nanoparticles.
- The higher wear resistance in AZ91-20B4C nanocomposites is due to the enhancement of the bonding strength among the matrix and reinforcement.
- The coefficient of friction has also reduced while adding secondary particles to the matrix material.
- The wear mechanism illustrates matrix material heavy delamination and ploughing of metal occurred while adding secondary particles to the matrix material the wear has prominently reduced. It palpated the secondary particles acts as a solid lubricant over the base material.

Data availability statement
The data that support the findings of this study are available upon reasonable request from the authors.

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