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Study of the Mechanical Properties of the Hybrid Nanocomposite Al/ZnO + Gr

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Abstract: Recently, the demand for hybrid aluminum matrix nanocomposites has surged due to improvements in their unique mechanical characteristics, such as low density, high strength, and corrosion resistance, making them suitable for use in a variety of industrial applications, including electronics, aerospace, and aircraft. This study examined the microstructure and mechanical characteristics of the aluminum matrix reinforced with a set amount of graphene at a rate of 0.5% by weight and different amounts of zinc oxide at rates of 2, 4, 6, 8, and 10% by weight. The present hybrid nano samples of Al/zinc oxide + graphene were made using the powder metallurgy process. Several examinations were performed on the samples, such as wear resistance and microhardness tests. The experimental results were analyzed by field-emission scanning electron microscopy and X-ray diffraction. A 5% improvement in the microhardness test and a 5% improvement in the corrosion rate test were obtained, and the highest improvement rate was obtained at 1 wt% of zinc oxide and 1% graphene.

Keywords: **powder metallurgy, nanocomposite, mechanical properties, microstructure examination, Al, hybrid, ZnO, Gr.**

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1. Introduction

Aluminum metal matrix composites possess exceptional attributes such as high toughness, superior wear resistance, and remarkable corrosion resistance. To cater to diverse applications, a wide range of manufacturing techniques have been employed in the synthesis of these composites (Li et al., 2014). In recent years, the limited strength, modulus, and ductility of aluminum (Al) have restricted its widespread application in the process industry. To overcome these limitations, numerous researchers have explored the incorporation of various reinforcements, such as particulates, short fibers, and long fibers, to enhance the mechanical properties of the material. These reinforcements include oxides (Al_2O_3, SiO_2, Y_2O_3, ZnO), borides (TiB_2, ZrB_2), intermetallic compounds (Fe_3Al, Ni_3Al), carbides (SiC, TiC, TaC), nitrides (Si_3N_4, TiN), as well as other inorganic and organic materials (fly ash, carbon fibers, CNT, SWCNT, MWCNT), which are incorporated into the Al matrix. The resulting aluminum metal matrix composites (Al-MMCs) exhibit improved strength and ductility, making them capable of providing a longer service life in various components (Bhoi et al., 2020).

Nanocomposites are a class of materials characterized by the presence of at least one component at the nanoscale. They can be broadly classified into three types: metal nanocomposites, ceramic nanocomposites, and polymer nanocomposites. Metal nanocomposites, as the name suggests, consist of a metal matrix with nanoscale components dispersed within it. Examples of metal matrix nanocomposites include Al/Al_2O_3, Al/MgO, Al/TiO_2, Al/Fe_2O_3, Al/Fe_3O_4, and Al/CNTs. Ceramic nanocomposites are composed of a ceramic matrix in which nanoscale constituents are incorporated. These nanoscale constituents can enhance the mechanical, electrical, or thermal properties of the material (Chidambaram et al., 2019).

There are three distinct types of processes used to manufacture composite materials: (a) liquid-state methods, which consist of in-situ processing, spray deposition, stir casting, and pressure infiltration; (b) techniques for semi-solid states, like compo casting; and (c) solid-state methods, including powder metallurgy. Each of these processes offers unique advantages. Liquid-state methods leverage the high interfacial energy between molten metal and reinforcements, resulting in good homogeneity of reinforcements within the matrix. On the other hand, solid-state methods enable the production of homogeneous reinforcements while avoiding the clustering of particle reinforcements (Manohar et al., 2018).

The metal oxide zinc oxide (ZnO) is essential to several disciplines, such as chemistry, physics, and materials science. ZnO is widely used in technology to produce fuel cells, sensors, microelectronic circuits, piezoelectric devices, and coatings for corrosion-resistant surface passivation (Harishanand et al., 2013).

Kumar Bhoi et al. (2020) studied a composite material consisting of aluminum and zinc oxide produced using a hybrid microwave sintering technique. Two different weight percentages (wt%) of zinc oxide nanorods, specifically 0.5% and 2%, were incorporated into the fabrication process of the composite material. Microhardness and nanoindentation tests were used to assess the composite material's mechanical properties. The microhardness results revealed that the addition of 2 wt% of zinc oxide nanorods led to a significant increase in hardness, with a hardness improvement of 27.78% compared to the pure aluminum matrix. In terms of nanohardness, the nanoindentation results indicated a further enhancement of 32.21% when 2 wt% of zinc oxide was incorporated, along with a slight decrease in the elastic modulus by 4.92%. Bhoi et al. (2022) fabricated composites of aluminum zinc oxide (Al-ZnO) nanorods with varying weight percentages of ZnO (3.5% and 5% by weight) in aluminum. The composites were produced using a combination of powder metallurgy and microwave hybrid sintering techniques. Detailed analysis of the composite material through microstructural examination and energy-dispersive spectroscopy revealed a relatively uniform distribution of reinforcements and a clear interface between the components. The composite material with 5% ZnO content demonstrated promising results in terms of mechanical properties. Specifically, it exhibited a significantly higher microhardness (approximately twice as high), nanohardness (about 3.5 times higher), and elastic modulus (approximately 1.9 times higher) compared to pure aluminum. Joshua et al. (2020) synthesized 7068/ZnO composites made via powder metallurgy. Various weight percentages of ZnO (5%, 10%, and 15%) were added to the AA7068 alloy as it was sintered. The experimental results demonstrated significant improvements in the mechanical properties of the composites. The Vickers microhardness number increased to 66 when 15% ZnO was added, which was twice the value observed for the matrix material alone. Moreover, the wear experiments conducted using a pin-on-disc wear tester revealed enhanced wear resistance in the composites with the addition of ZnO particles.

Marcu et al. (2018) fabricated aluminum-based hybrid composites, specifically Al/Al_2O_3/Gr composites, using powder metallurgy (P/M) techniques. For the

characterization of the composites, they employed specific analysis techniques (SEM-EDS) and X-ray diffraction (XRD) analyses. The microstructural characterization of the Al/Al_2O_3/Gr composites revealed a highly uniform distribution of the constituent materials. This uniform distribution played a crucial role in imparting favorable properties to the final composite material. The even distribution of the aluminum, alumina, and graphite phases within the composite matrix contributed to enhanced mechanical, thermal, and electrical properties. Iacob et al. (2015) investigated the Al/Al_2O_3/Gr hybrid composites manufactured by powder metallurgy. The analysis of the composites' microstructure revealed a relatively uniform distribution and a certain degree of grain refinement within the specimens. Additionally, it was observed that the microhardness increased with longer milling time and higher reinforcement content, primarily attributed to the presence of hard Al_2O_3 particles. Furthermore, the Al/Al_2O_3/Gr hybrid composites exhibited a low wear rate, which can be attributed to the combined effect of Al_2O_3 and Gr acting as load-bearing elements and solid lubricants, respectively. Sabry et al. (2020), Liu et al. (2016), and Sadooghi and Hashemi (2019) also showed that the manufacture of hybrid composites of graphene-reinforced aluminum and zinc oxide with other oxides or ceramic materials led to a significant improvement in mechanical properties.

The purpose of this study is to use the powder metallurgy method to investigate how graphene and zinc oxide affect the mechanical and micro characteristics of pure aluminum.

2. Materials and Methods

In this work, the following materials were used: Al powder as the matrix material; ZnO powder as a reinforcement material, added at different rates; and Gr (graphene) powder as a reinforcement material, added at a constant rate. These materials were used to fabricate an Al matrix nanocomposite material. Table 1 lists the physical characteristics of the powders used in this technique.

3. Preparation of the Specimens

All the hybrid nanocomposite samples were fabricated utilizing the method of powder metallurgy (PM). This process involved the following steps: powder mixing, compaction, sintering, and post-processing. An Al matrix nanocomposite material was fabricated by mixing the ingredients using a planetary ball mill (model QM-ISPO4). The powder mixture consisted of Al as the matrix material and ZnO as a reinforcement material, added at varying contents of 0.5, 1.5, 2.5, and 3.5 wt.%. Graphene (Gr) was added as a reinforcement material at a constant content of 0.5 wt.%, as shown in Table 2. The planetary ball mill was operated at 400 RPM for 30 minutes at room temperature to create a homogeneous powder mixture.

Table 1. Physical characteristics of such powders.

Powder	Al	ZnO	Gr
Density (g/cm^3)	2.7	5.7	2.2
Melting temperature	660.3	2014	3600
Particle size	44 µm	20nm	15nm
Purity	99.9%	99.8%	99.5%

Table 2. Weight percentages of the powders used for Al/ZnO + Gr.

Sample No.	Al (wt.%)	ZnO (wt.%)	Gr (wt.%)
Al pure	100	0	0
A	99	0.5	0.5
B	98	1.5	0.5
C	97	2.5	0.5
D	96	3.5	0.5

A hydraulic press with a uniaxial force and a pressure of 314.7 MPa was used to perform cold compaction operations in the steel mold. Figure 1 illustrates a cylindrical mold consisting of an upper punch with a diameter of 13 mm, a cylindrical cavity with 13 mm and 20 mm inside and outside diameters, respectively, and a lower base with a 13 mm diameter to produce different specimens of the functionally graded materials using the powder compaction technique. The pressed materials were sintered in an electric furnace to prevent oxidation. The sintering process was carried out at a temperature of 630 °C for a duration of 1.5 hours, as shown in Figure 2. An argon atmosphere was maintained in the furnace during the sintering process. The cylindrical specimens utilized for FESEM and XRD investigations had dimensions of 13 mm in diameter and 9 mm in height, whereas the specimens used for microhardness and wear resistance testing were 13 mm in diameter and 10 mm in height (Omran et al., 2024), as shown in Figure 3.

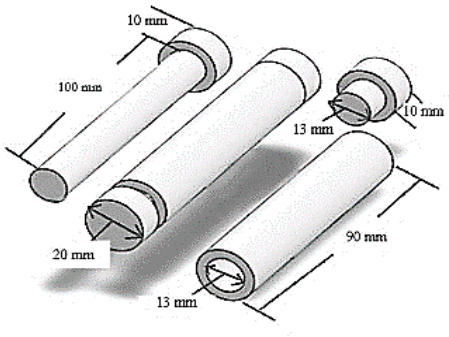


Figure 1. Powder compaction die parts: lower punch, upper punch, die chamber, and die assembly.

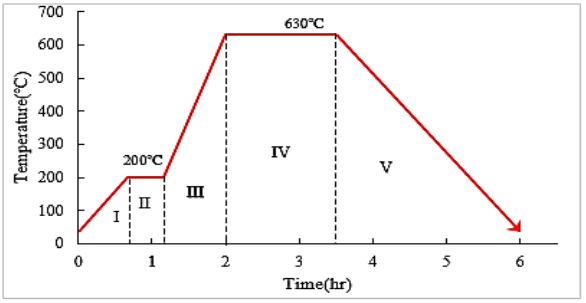


Figure 2. The sintering process.

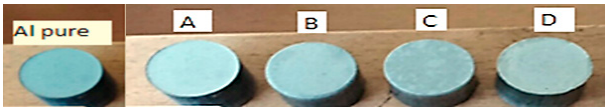


Figure 3. Specimens for wear examination and testing with varying percentages.

4 Microstructural Examination

4.1 Field Emission Scanning Electron Microscope (FESEM) Examination

FESEM (CamScan MV 2300) was used to conduct a microstructural study after the specimens were prepared. This analysis focused on the received specimens as well as those embedded with graphene (Gr) at a constant weight percentage and ZnO at variable weight percentages in an aluminum matrix. The FESEM examination aimed to assess the microstructure of the nanocomposite and to evaluate the uniform distribution of nanoparticles in the matrix. The samples were polished using a polishing cloth after being processed with grinding papers that had grit sizes between 100 and 2000 nm.

4.2 X-ray Diffraction (XRD)

The XRD-6000 (SHIMADZU Europe) was used for XRD analysis on all samples in order to identify the phases produced in the nanocomposites. Furthermore, the effect of different ZnO weight percentages on the specimens' resulting phases was investigated.

5. Mechanical Tests

5.1 Microhardness Test

The microhardness of sintered samples, including pure Al and Al/ZnO + Gr hybrid nanocomposites, was assessed using the Vickers microhardness test in accordance with the ASTM E384-99 standard. Before testing, the sintered specimens were smoothed with emery papers ranging from 100 to 2000 µm and then polished with a polishing cloth. A Vickers microhardness tester (type HVS-1000) was used to evaluate microhardness. Multiple readings were taken for every specimen, and the average value was determined.

5.2 Wear Rate Measurement

A dry sliding wear machine was utilized to evaluate the wear rate of the Al matrix with varying contents of ZnO and graphene (Gr). The calculations were based on the pin-on-disc technique using Equations (1) and (2) (Zmitrowicz, 2006). As previously mentioned, the specimens for the wear test measured 12.65 mm in diameter and 20 mm in height. These specimens were mounted against a rotating hard disc, operating at a speed of 350 rpm for a duration of 6 minutes. Different loads were applied during the test, specifically 5, 10, and 15 N.

$$\text{Wearrate} = \frac{\Delta w}{\text{S.D.}}(\text{g/cm}) \qquad (1)$$

$$\Delta w = w_1 - w_2$$

$$S.\,D. = 2\pi rn \qquad (2)$$

where

Δw is the variation in the specimen's weight (gm).

w_1, w_2 are the specimen's weights (gm) both before and after the wear test.

S.D. is the sliding distance (cm).

r is the radius of the rotating disc (cm).

n is the number of rotations per minute (rpm).

6. Results and Discussion

6.1 Microhardness

The microhardness characteristics of Al-ZnO-Gr hybrid nanocomposites are significantly affected by both the compaction process and the weight percentage of the reinforcing nanomaterials. The microhardness values for these hybrid nanocomposites have been carefully recorded in Table 3 and illustrated in Figure 4.

A clear trend emerges: as the weight percentage of ZnO increases, the microhardness values also rise. This phenomenon can be attributed to the superior microhardness of the additives compared to the aluminum matrix. As the weight percentage of the additives increases, they effectively fill the pores within the nanocomposite, reducing porosity and enhancing microhardness.

Table 3. The microhardness results for Al-ZnO-Gr.

Specimen	Wt.% of ZnO	Microhardness (HV)
Al pure	0	18.22
A	0.5	20.47
B	1.5	23.59
C	2.5	26.98
D	3.5	33.74

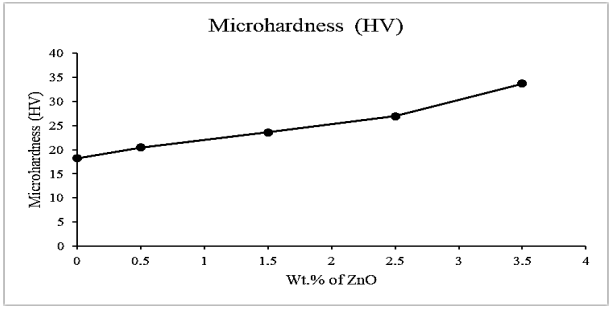


Figure 4. Microhardness values of hybrid nanocomposites

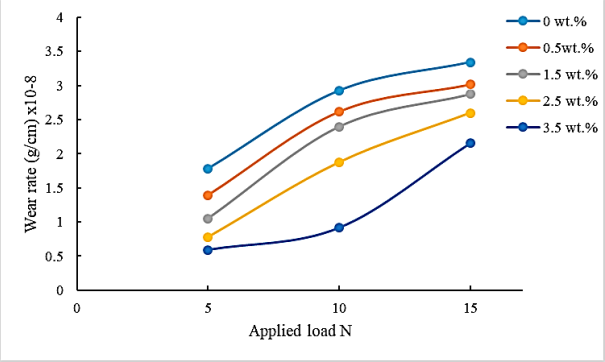


Figure 5. Al/ZnO + Gr wear rate relationship with applied loads.

6.2 Wear Rate

The wear rate of the specimens was evaluated under three different loads: 5 N, 10 N, and 15 N, while maintaining a speed of 350 rpm for a duration of 6 minutes. Figure 5 illustrates the wear rate of the Al-ZnO-Gr composites. The findings indicate that the incorporation of graphene (Gr) and an increase in zinc oxide (ZnO) content significantly reduce the wear rate compared to pure aluminum. This improvement can be attributed to the superior tribological properties of both Gr and ZnO. Consequently, these materials emerge as excellent candidates for applications where components experience wear and friction. Table 4 shows wear test results.

Table 4. Wear test results for the samples (Al/ZnO + Gr).

	Wear rate (g/cm) $x10^{-8}$				
load N	0 wt.%	0.5 wt.%	1.5 wt.%	2.5 wt.%	3.5 wt.%
5	1.781	1.394	1.053	0.782	0.594
10	2.928	2.619	.3992	1.876	.5171
15	3.344	3.017	2.874	2.598	2.155

6.3 FESEM Examination

Figure 6 shows FESEM images of pure aluminum and aluminum/zinc oxide with different zinc oxide concentrations and constant gramine content. Evidently, nanoreinforcement materials are homogeneously distributed within the aluminum matrix. As a result, the mechanical properties were improved. Also, a strong interfacial bond was formed at the interfaces between the ceramic nanoparticles and the aluminum matrix due to the correct sintering process. In addition, few pores and no cracks were observed in the samples.

6.4 XRD Analysis

XRD analysis was performed for the pure aluminum sample before adding the reinforcing materials and for the samples after adding zinc oxide at different concentrations and graphene at a fixed concentration, as shown in Figure 7. The XRD patterns show distinct peaks for all materials, and their intensity and density increase with increasing amounts of additive materials, with peaks corresponding to aluminum, zinc oxide, and graphene.

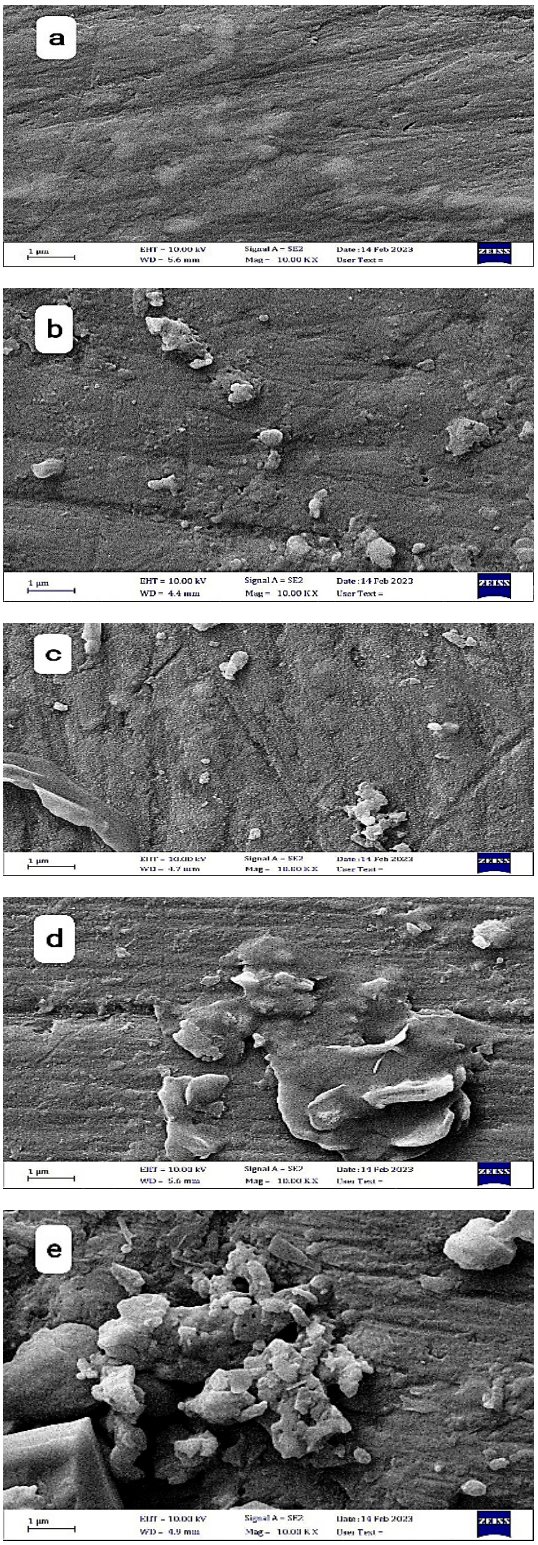


Figure 6. FESEM images: (a) pure Al; (b) Al + 0.5 ZnO + 0.5 Gr; (c) Al + 1.5 ZnO + 0.5 Gr; (d) Al + 2.5 ZnO + 0.5 Gr; (e) Al + 3.5 ZnO + 0.5 Gr.

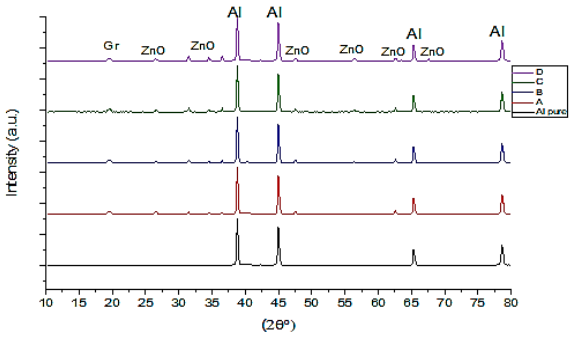


Figure 7. XRD for pure Al and Al + ZnO + Gr.

Conclusions

The powder metallurgy method was used to successfully produce the Al/ZnO + Gr hybrid nanocomposites, with aluminum reinforced by ZnO nanoparticles at several weight percentages and a fixed weight percentage of Gr. The microstructure of the samples was characterized using FESEM and XRD examinations, and mechanical properties such as microhardness and wear resistance were estimated. The results of this work showed that FE-SEM and XRD tests indicated homogeneous distribution of the reinforcement material in the aluminum matrix. The mechanical properties also showed a clear improvement, as increasing the reinforcement material increased the rate of improvement in microhardness and decreased the corrosion rate. When compared to the Al matrix, microhardness increased by 85.2% as the weight percentage of ZnO increased, but the wear rate decreased by 48.2% at the same weight percentage of ZnO.

Conflict of Interest

The authors do not have any type of conflict of interest to declare.

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