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Evaluation of the Microstructure and Mechanical Properties of Al / Fe_3O_4 Nanocomposites

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HIGHLIGHTS

- Al/Fe_3O_4 nanocomposite were successfully fabricated by powder metallurgy method.
- The microstructure examinations showed homogeneous distribution to Fe_3O_4 nanoparticles in Al matrix.
- Compared to other weight percentages, the nanocomposite with 10 wt.% of Fe_3O_4 has highest microhardness and low wear rate.
- Compared to other weight percentages, the nanocomposite with 6 wt.% of Fe_3O_4 has highest compressive strength.

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ABSTRACT

The goal of this research is to study the microstructural analysis and mechanical properties of an aluminum matrix reinforced with different amounts of nano Fe_3O_4 at (2, 4, 6, 8, and 10wt. %). Al/ Fe_3O_4 nanocomposites specimens were prepared using the powder metallurgy route. Many examinations, including Field Emission Scanning Electron Microscopy (FESEM) and X-Ray Diffraction (XRD) analysis, were performed on the specimens in this study to determine the microstructure and phases of the nanocomposites. Mechanical tests, such as compressive, microhardness, and wear tests, were also performed to assess the mechanical properties of the nanocomposites. The results of this study show that Fe3O4 nanoparticles have been homogeneously dispersed in the Al matrix by FESEM and XRD examination. While the mechanical tests show improving the compressive strength at 6 wt.% by 5.36%, the highest microhardness was at 10% by 101.6% compared with the pure Al, and improving the wear rate.

1. Introduction

Aluminum matrix nanocomposites (AMNCs) are advanced materials reinforced with soft or hard materials to produce nanocomposite materials for various engineering applications. Aluminum alloys are known for their low cost, light weight, environmental resistance, and good mechanical properties. These properties make them suitable for use in metal matrix composites (MMCs) as a matrix material [1]. Aluminum matrix nanocomposites (AMNCs) are important materials because of their high strength, wear resistance, and light weight, which enable them to be used in a variety of applications including automotive, electronics, aerospace, space shuttle components, disc brake and aero frame's structure. To improve mechanical and physical properties, various reinforcement materials are used to manufacture (AMNCs), which are used for high-tech engineering components like aerospace, automobile, and industrial shipbuilding. Due to their excellent performance, strength-to-weight and stiffness-to-weight ratios, reinforced aluminum matrix composites with nano iron oxide (Fe_3O_4) are very appealing for this reason and they are used in many applications such as aeronautical, automotive, and others [2]. Magnetic nano (Fe_3O_4) reinforced AMCs, in general, have good physical properties such as electrical and magnetic properties, allowing them to be lightweight multifunctional materials. It does not, however, have a high wear resistance [3]. A study was carried out on a composite aluminum matrix reinforced with magnetite (Fe_3O_4) and a variety of additive elements. They performed microhardness tests on AMNCs to determine their hardness and scratch strength to research their wear efficiency. Scanning Electron Microscopy was used to investigate the morphology of damaged surfaces. The aim was to assess the knowledge about nano fillers' wear resistance and mechanism [4]. Scrap aluminum containing 10% Fe_3O_4 nanoparticles was used in the experiment. The interface for the dispersion of the reinforcement and matrix was tested using Scanning Electron Microscopy (SEM) to generate AMNCs and investigate the effect of TiO_2 doping Fe_3O_4 at 2.5 wt. percent, 5 wt. percent, 7.5 wt. percent, and 10 wt. percent. Microhardness, young modulus, and density have all been measured and compared. The constituent distribution was uniform, with a close bond between the matrix and reinforcements. Wear conduct was also changed as the percentage of TiO_2 was increased [5]. New aluminum-based

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composites (Al-Zn-Si) with improved magnetic, physical, and mechanical properties were developed as part of an aeronautic research program. Different reinforcements may be used to customize these composites for applications in electrical motors and other areas of aircraft engineering. Previous research in the same field produced composites with promising properties, which were due to the reinforcement size and distribution, as well as the grain size of the matrix [6,7,8]. AA7068 is used in a variety of industries, including aerospace and automotive, medical equipment, and high-precision engineering. Powder metallurgy was used to build the AA7068/MgO composites. The particles in the composite were mixed with homogeneous dispersion, according to optical micrographs. The addition of MgO particles improved the mechanical properties of the material. With the addition of 5% MgO particles, the composite's hardness reached a limit of 68 VHN. The addition of MgO particles as a reinforcement in the AA7068 greatly improved the wear resistance [9,10]. The aim of this research is to study the effect of Fe_3O_4 nanoparticles on microstructure and mechanical properties such as microhardness, wear resistance and compressive strength of aluminum matrix nanocomposite manufactured by powder technology route, which are used in a variety of applications including automotive, electronics, aerospace, space shuttle components, disc brake, aero frame’s structure, and electrical industries.

2. Experimental Procedures

2.1 Materials

Aluminum powder with a grain size of less than 44 µm and purity of 99 percent is used in this study, which is reinforced with nano iron oxide powder (Fe_3O_4) with a mean grain size of 30 nm and purity of 99.5 percent. Table 1 shows the weight percentages of the powders used in each specimen.

Table 1: The weight percentages of powders for the specimens

No. ofspecimens	Aluminumwt.%	Magnetite (Fe3O4)
1	100	0
2	98	2
3	96	4
4	94	6
5	92	8
6	90	10

2.2 Preparation of the specimens

All nanocomposites’ specimens were prepared using the powder metallurgy (PM) method, with the mixture powders being mixed in a planetary ball mill QM-$ISPO_4$. The powders mixture in this work consists of (Al) powder as a matrix. Table 2 shows the properties of the (Al) powder that was used in this work and reinforced with magnetite (Fe_3O_4) powder in various amounts (2, 4, 6, 8 and 10 wt.%). To reduce agglomeration and achieve a homogeneous dispersion, the mixing process was carried out at 400 rpm for 30 minutes at room temperature without lubricant material. After the powders were mixed, they were cold pressed (compacted) in a uniaxial direction with a pressure of 4 tons to produce green compacts in the shape of cylindrical specimens with a diameter of 12.65mm. To avoid oxidation, the green compacts were sintered in an electrical furnace at 630°C for 1.5 hours under an argon atmosphere.

Table 2: Chemical composition and physical properties of Al

Metal matrix	Fe	Pb	As	Density	Particle size	Mesh	Purity
Al	0.5%	0.03%	0.0005%	2.7gm/cm^3	<44µm	325	99.0%

2.3 Microstructure Examination

After preparing the specimens, a microstructure analysis was performed using a Field Emission Scanning Electron Microscopy (FESEM) (cam scan MV 2300) for pure aluminum, and after embedding iron oxide with different weight percentages into an aluminum matrix. The specimens were ground with grinding papers with grit sizes from 200 to 2000 µm and polished with polishing fabric before the examination. The aim of this examination was to determine the distribution of reinforcing materials, as well as the impact of compacting pressure and sintering temperature on the microstructure of manufactured specimens.

2.4 X-Ray Diffraction (XRD)

All specimens were subjected to X-ray diffraction analysis by ((XRD-6000) SHIMADZU Europe) to determine the phases produced because of the manufactured nanocomposites. In addition, the effect of Fe_3O_4 at various weight percentages on the resultant phases of manufactured specimens was examined (AMNCs).

2.5 Mechanical properties tests

Microhardness, compressive, and wear tests were all performed on the specimens in this study. Figure 1 shows the device of mechanical properties tests.

2.5.1 Microhardness test

The microhardness of sintered specimens for pure Al and Al/Fe_3O_4 nanocomposites was determined using the Vickers microhardness test according to the ASTM E384-99 standard. The sintered specimens were smoothed with emery papers with grain sizes from 200 to 2000 µm before being polished with a polishing cloth. The microhardness of specimens was then measured using a Vickers microhardness unit, model HVS-1000 digital microhardness test, at load 0.245N for 15 seconds. For each specimen, three readings were taken, and the average value was determined.

2.5.2 Wear test

To measure the wear rate of pure Al and Al/Fe_3O_4 nanocomposite, a pin-on-disc technique was used in accordance with ASTM G99-95 standards. The specimens used in the wear test had a diameter of 12.65 mm and a length of 20 mm. The specimens for wear testing are fixed against a hardened disk that rotates at 950 rpm for 5 minutes in this test. The applied loads were varied between 5, 10, and 15N during the wear test. The following equation [11,18] was used to calculate the wear rate of the specimens:

$$\text{Wear rate} = \frac{\Delta \text{w}}{\text{SD}} \ (\text{g}/cm) \qquad (1)$$

$$\Delta \text{w} = w_1 - w_2 \qquad (2)$$

$$\text{SD} = 2\pi \, r.n.\,t \qquad (3)$$

Where: Δw: the change in the weight of the specimens (gm). w_1, w_2: the weight for the specimen before and after the wear test (gm). S.D: the sliding distance (cm). r: the radius of the rotating disc (cm). n: the number of rotations per minutes (rpm). t: the sliding time (min).

2.5.3 Compression test

Compression test was carried out and controlled by electrical, universal testing for all specimens pure Al and Al/Fe_3O_4 nanocomposite according to ASTM G1-90. The specimens used in this test had a diameter of 12.65 mm and a length of 20 mm. The young modulus can be calculated by the equation [17]:

$$Stress\ (\sigma) = \frac{Applied\ force\ on\ a\ material}{Area\ over\ which\ that\ force\ is\ applied} = \frac{P}{A}\,(MPa) \qquad (4)$$

$$\text{Strain } (\varepsilon) = \frac{\Delta L}{L} \ (\text{No units}) \qquad (5)$$

According to Hooke's Law the relation between stress and strain is as follows:

$$\mathbf{E} = \frac{stress\ (\sigma)}{strain\ (\varepsilon)} \ (\text{MPa}) \qquad (6)$$

Where: (**E**) is modulus of elasticity or Young's modulus

Figure 1: Digital Micro Hardness Tester, Dry sliding wear machine,Compressive test machine

3. Results and Discussion

3.1 FESEM

Figure 2 shows images by Field Emission Scanning Electron Microscopy FESEM for pure Al, and Al/Fe_3O_4 with various Fe_3O_4 concentrations. It can be observed that the reinforcement materials are diffused at the interfaces between Fe_3O_4 with Al matrix. Because of the thermal reaction and diffusion mechanism used in the sintering process, strong interfacial bonding has formed at the interfaces between ceramic Fe_3O_4 nanoparticles and aluminum matrix. Because of aggregations in a specific region, a uniform dispersion of Fe_3O_4 nanoparticles in the aluminum matrix cannot be achieved in most specimens for various Fe_3O_4 weight percentages. As a result, the mixing process will achieve a very homogeneous distribution of nanoparticles, resulting in improved mechanical properties.

3.2 X- Ray Diffraction

Figure 3 depicts X-Ray diffraction (XRD) results for specimens before and after Fe_3O_4 addition. For the manufactured specimens in this study, the XRD patterns consist of peaks that deal with the phases of Al and Fe_3O_4. XRD analysis of specimens reinforced by Fe_3O_4 and integrated into Al matrix revealed that increasing the weight percentage of Fe_3O_4 leads to the appearance of Fe_3O_4 phase peaks. In fact, XRD results show that increasing the weight percentage of additive materials causes the intensity of the diffraction peaks to increase. Furthermore, since no other intermetallic inclusions are present, the reaction between Al and Fe_3O_4 is most likely in a steady state thermodynamically [12].

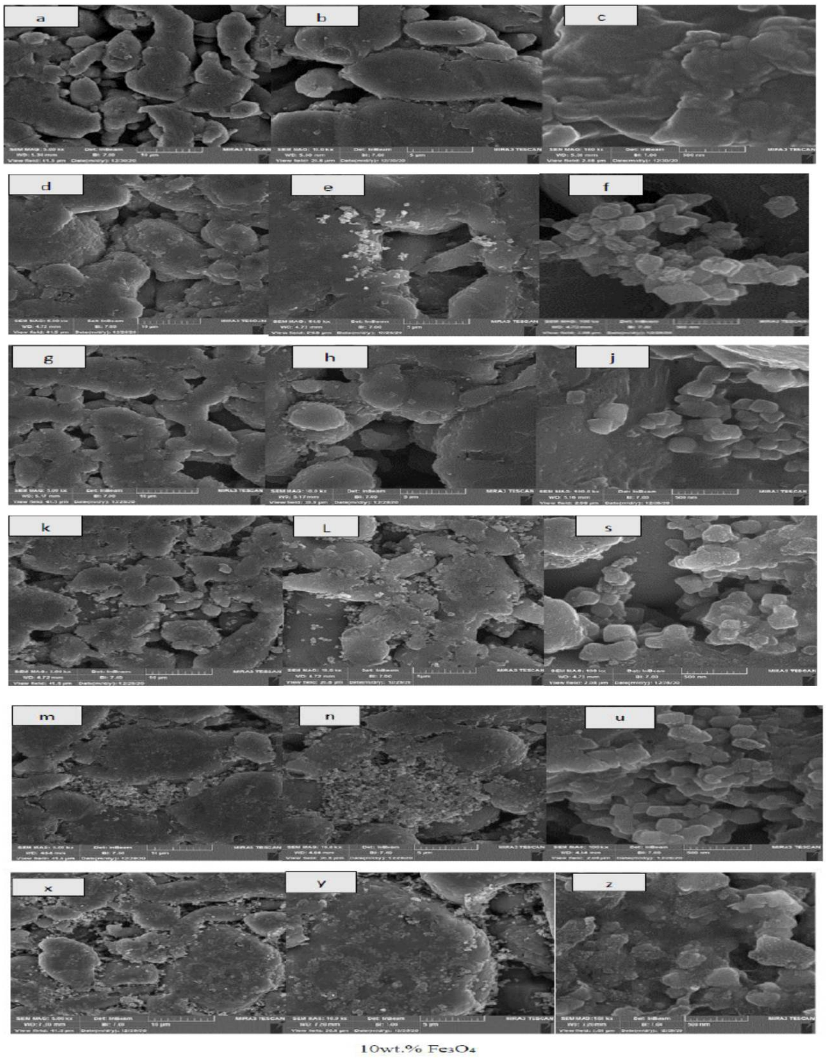

Figure 2: FESEM images of the specimens of pure Al and Al/Fe_3O_4 nanocomposites with different percentages of Fe_3O_4: (a, b, c) pure Al, (d, e, f) 2 wt. % Fe_3O_4 (g, h, j) 4 wt. % Fe_3O_4 (k, L, s) 6 wt. % Fe_3O_4 (m, n, u) 8 wt. % Fe_3O_4 and (x, y, z) 10 wt. % Fe_3O_4

3.3 Mechanical properties

3.3.1 Hardness results

Vickers microhardness findings for pure Al and Al/ Fe_3O_4 specimens are shown in Table 3. Microhardness values of Al/ Fe_3O_4 nanocomposites are typically affected by compacting strain, sintering temperature, and reinforcement nanomaterial weight percentages. The microhardness increases as the weight percentage of Fe_3O_4 increases. This is because Fe_3O_4 has a higher hardness than that of the Al matrix. Finally, the microhardness test is a valuable method for determining the microstructure and mechanical properties of nanocomposites [13]. It is clear from Table 3 that the maximum value of microhardness is for the nanocomposite containing 10wt.% Fe_3O_4. The microhardness is increased with increasing wt.% of Fe_3O_4 [19] by 101.6% for 10wt.% of Fe_3O_4 compared with the pure Al.

Table 3: The microhardness for the specimens Al/ Fe_3O_4

Sample No.	Fe3O4 Wt.%	Microhardness
1	0	18.14
2	2	23.93
3	4	24.90
4	6	32.59
5	8	33.19
6	10	36.57

3.3.2 Wear results

Figure 5 shows the wear rate for Al/ Fe_3O_4 nanocomposites with different loads of 5, 10, and 15N and a steady sliding velocity of 950 rpm versus Fe_3O_4 weight percentages. A strong interfacial bonding at the interfaces between Fe_3O_4 ceramic nanoparticles and the Al matrix will improve the wear resistance of Al/ Fe_3O_4 nanocomposites. Table 4 shows that as the weight percentage of Fe_3O_4 nanoparticles increases, the wear rate decreases. On the other hand, strain fields form around Fe_3O_4 nanoparticles due to the different thermal expansion coefficients of Fe_3O_4 and Al. The dislocations piled up because of the strain field, preventing the cracks from propagating. The Orwan mechanism is strengthened by the homogeneous dispersion of Fe_3O_4 nanoparticles in the Al matrix [14]. As a result, Al/ Fe_3O_4 nanocomposites have better wear resistance. Fe_3O_4 nanoparticles strengthen the tribological properties of the nanocomposite. This is confirmed with [15]. The wear rate decreases by 47.2% at 10 wt.% Fe_3O_4 compared with the pure Al, and the best value of the wear rate is 2.37×10^{-8} (g/cm) at 10wt.% Fe_3O_4.

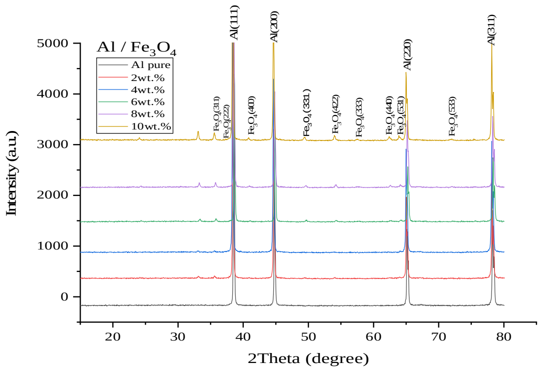

Figure 3: XRD diffraction peaks for the specimens of Al / Fe_3O_4 nanocomposite

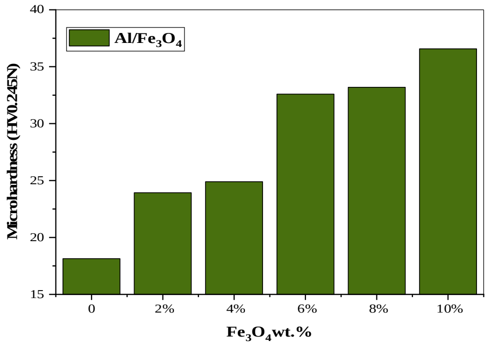

Figure 4: Relationship between microhardness and wt.% Fe_3O_4

Table 4: The results of wear test for the specimens Al/ Fe_3O_4

Applied Load N	**Wear rate (g/cm)× 10^{-8}**					
	0%	2%	4%	6%	8%	10%
5	4.90	4.59	4.43	3.56	3.43	2.37
10	5.52	4.92	4.54	3.71	3.51	2.47
15	6.44	5.77	5.31	4.69	4.38	3.40

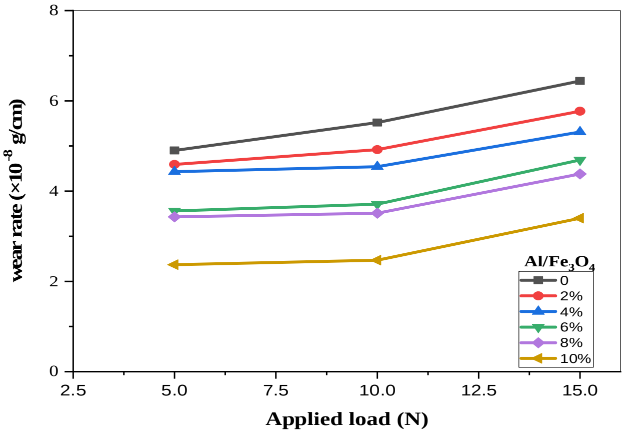

Figure 5: Relationships between applied loads and wear rate for different percentages of Fe_3O_4 nanoparticles

Table 5: The result for compressive test specimens Al/ Fe_3O_4

Sample	Wt. %	Load kN	Compressive MPa	Y models MPa
1	0	7.41	56	2750
2	2	3.91	31	1800
3	4	4.36	34	2000
4	6	7.58	59	3140
5	8	2.84	22	1750
6	10	3.42	27	1850

3.3.3 Compressive results

Figure 6 shows stress strain curves of the compressive test for pure Al and Al/Fe3O4 nanocomposites with different weight percentages of Fe3O4 as (2,4,6,8, and 10) wt.%. This graph shows that increasing the weight percentage of Fe3O4 causes to increase the compressive strength until 59 MPa at 6 wt.% Fe3O4. This may be because homogenous distribution for Fe3O4 nanoparticles allows the structure to retain its high ductility [16]. The increase in compressive strength can be explained by the fact that the addition of Fe3O4 slowed grain growth by pinning grain boundaries and stabilizing the grains. Table 5 shows the results of the compressive test for Al / Fe3O4 nanocomposite. Increasing the resistance of weight can increase brittleness while decreasing compressive strength, and then it will be restricted at the elastic deformation region and limited fracture at a specific area on the surface. Nanocomposites is clearly linked with the addition of a hard ceramic material of Fe3O4 greater than 8% by weight, which can increase brittleness while decreasing compressive strength, and then be it will be restricted at the elastic deformation region and limited fracture at a specific area on the surface.

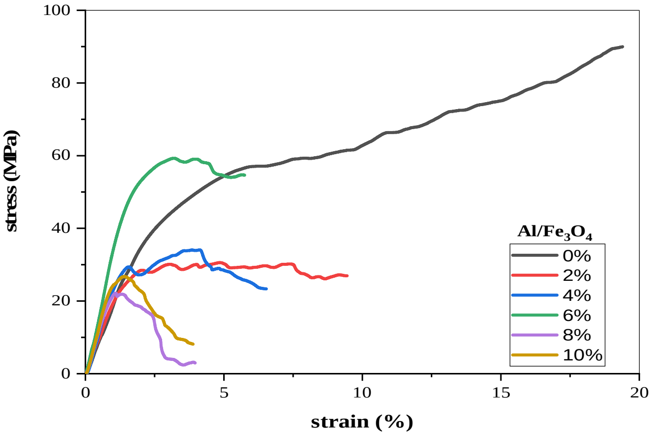

Figure 6: Stress strain curves of compressive test for pure Aluminium and Al/Fe$_3$O$_4$ nanocomposites

4. Conclusions

The (Al/Fe$_3$O$_4$) nanocomposites were successfully produced by the powder metallurgy method. As a result of the advanced process, the conclusion of the obtained results can be summarized as follows:

1) The microstructure was characterized using Field Emission Scanning Electron Microscopy (FESEM) and X- ray diffraction (XRD) exams.
2) The results of FESEM and XRD show that nanoparticles in various weight percentages are well dispersed in the aluminum matrix.
3) Microhardness, wear, and compressive tests were used to quantify mechanical properties. The experimental results show that the mechanical properties have improved.
4) Mechanical properties will improve as the proportion of reinforcement nanoparticles increases for Al/ Fe3O4 nanocomposites.
5) The microhardness is increased with increasing wt.% of Fe3O4 by 101.6%, for 10 wt.% of Fe3O4 compared with the pure Al.
6) The wear rate decreases with increasing of wt.% of Fe3O4 by 47.2% for 10 wt.% for Al / Fe3O4 compared with the pure Al.
7) The addition of Fe3O4 nanoparticles increases the compressive strength until 59 MPa at 6 wt.% of Fe3O4, with an increase by 5.36% compared with the pure Al.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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