Evaluation the microstructure, physical, magnetic and electrical properties of Al / Fe_3O_4 + Ni hybrid nanocomposite

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Abstract. This paper aims to study the physical, electrical, magnetic properties and microstructural analysis of aluminum matrix incorporated with different amounts of Fe_3O_4 at (2, 4, 6, 8, and 10wt.%) with a constant amount of Ni at 2wt.%. Al / Fe_3O_4 + Ni hybrid nanocomposites specimens were prepared using the powder metallurgy method. Aluminum matrix nanocomposites (AMNCs) are important alloys because of their high strength, wear resistance, and lightweight, which enables them to be used in different thermal environments in a variety of applications including automotive, electronics, and aerospace. Many examinations, including Field Emission Scanning Electron Microscopy (FESEM) analysis, were performed on the specimens in this study to determine the microstructure and phases of the nanocomposites, and study important properties such as density, porosity, magnetic and electrical properties, to evaluate these properties of the hybrid nanocomposites. FESEM analysis revealed that Fe_3O_4 and Ni nanoparticles were homogeneously distributed in the Al matrix in this study. The results of these tests showed that increasing the weight percentage of Fe_3O_4 and constant weight percentage of Ni nanoparticles decreases the porosity and increases the density and increases saturation magnetization (Ms) as well as improved electrical properties.

Keywords: hybrid nanocomposites, powder metallurgy, Fe_3O_4, Ni, microstructure, physical, electrical, and magnetic properties

1. Introduction

Metal Matrix Nanocomposites (MNCs) have been studied extensively in recent years due to the promising properties that make them suitable for a wide range of functional and structural applications [1]. Aluminum matrix nanocomposite materials (AMNCs) are a type of advanced composite material that allows engineers to change the material's properties to meet the demands of modern technology. These composites are used in a variety of high-tech structural and functional
applications, such as aerospace, defense, automotive, and thermal management. When the reinforcement process is reduced to the nanoscale, particle-dislocation interactions become more important, which, when combined with other strengthening effects found in traditional MMCs, results in a major improvement in mechanical properties [2–5]. Powder metallurgy worldwide success stems from its ability to manufacture complex shapes with precise measurements at a high production rate and low cost [6–9]. Because of their excellent stiffness-to-weight and strength-to-weight ratios, reinforced (with Fe₃O₄, iron oxide) aluminum matrix composites are very suitable in structural applications (automotive, aeronautical, etc.) [10–12]. E.Bayraktar et al. used powder metallurgy to create an aluminum matrix composite with nano iron oxide (Fe₃O₄) to enhance the magnetic properties of aluminum. Previous research has found that adding Fe₃O₄ nanoparticles to a material improves soft magnetic properties [7]. Furthermore, adding iron oxide (Fe₃O₄) reinforcement to increase the magnetic permeability of these composites is very appealing, resulting in a strong synchronization between the thermal and electrical conductivities as well as the magnetic permeability [8–12]. The powder metallurgy process was used to successfully fabricate the hybrid reinforcements (Fe₃O₄+SiC) novel composite. The addition of Fe₃O₄ nanoparticles and SiC hybrid reinforcements to the aluminum matrix improved the magnetic permeability as well as the thermal properties of the composites without causing mechanical degradation [13]. To fabricate hybrid aluminum matrix composites, several studies were conducted on composites with two or more reinforcements. Hybrid aluminum matrix composites (HAMC) are the next generation of composites that can replace single-reinforcement composites while also adding new features to increase the material's performance [14, 15]. An aeronautic research program resulted in the development of new aluminum-based composites (Al-Zn-Si) with improved magnetic, physical, and mechanical properties. These composites can be customized with different reinforcements for applications in electrical motors and other areas of aircraft engineering. Previous work in the area had yielded composites with promising properties, which were caused by the size and distribution of the reinforcements, as well as the grain size of the matrix [16,17,18]. The objective of this study is to see how Fe₃O₄ and Ni nanoparticles affect the magnetic, electrical, and physical properties and microstructure of aluminum matrix manufactured using the powder metallurgy method.

2. Experimental procedures

2.1. Materials

Aluminum powder with a grain size of less than 44µm and purity of 99 % is used in this study, which is reinforced with nano iron oxide Fe₃O₄(Magnetite) with a grain size of 20 nm and purity of 99.5 percent and nickel (Ni) nanoparticles with a grain size of 30 nm and purity of 99.9%. Table 1 shows the weight of the powders used in each specimen.

| No. of specimen | Aluminum Wt.% | Magnetite (Fe₃O₄) wt.% | Nickel (Ni) wt.% |
|----------------|---------------|------------------------|-----------------|
| 1              | 100           | 0                      | 0               |
| 2              | 96            | 2                      | 2               |
| 3              | 94            | 4                      | 2               |
| 4              | 92            | 6                      | 2               |
| 5              | 90            | 8                      | 2               |
| 6              | 88            | 10                     | 2               |

Table 1. The weight percentages of powders for specimens.
2.2. Preparation of the specimens

All specimens of hybrid nanocomposites, were prepared by powder metallurgy (PM) route, mixing process for mixture powders was carried out in planetary ball mill QM-ISPO4. In this work, the powders mixture consists of (Al) powder as a matrix, magnetite (Fe₃O₄) powder with a different percentage (2,4,6,8 and 10) wt.% and constant weight percentage of Ni(2wt.%) powder as an additive material. To reduce agglomeration and achieve a homogeneous dispersion, the mixing process was done at 400 rpm for 30 minutes at room temperature without lubricant materials. 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.

2.3. Microstructure Examination

2.3.1. a Field Emission Scanning Electron Microscopy (FESEM)

After preparing the specimens, a microstructure analysis was performed using a Field Emission Scanning Electron Microscopy (FESEM) (cam scan MV 2300) for pure Al, and Al/Fe₃O₄+Ni hybrid nanocomposite. The specimens were smoothed with grinding papers with grit sizes of 300 to 2000µm before being polished with polishing fabric. 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.

2.3.2. X-Ray Diffraction (XRD) analysis

All specimens were subjected to X-ray diffraction analysis ((XRD-6000) SHIMADZU Europe) to determine the phases produced as a result of the manufactured nanocomposites. In addition, the effect of Fe₃O₄ with various weight percentages and a constant weight percentage of Ni on the resultant phases of the manufactured specimens was investigated (AMNCs).

2.4. Physical properties tests

2.4.1. Density test

Density was measured geometrically for the specimens before and after sintering by equation (1).

\[ \rho = \frac{\text{mass}}{\text{volume}} \]  

Where:
\[ \rho \]: the actual density (g/cm³)

2.4.2 Porosity test

Porosity for a given specimens is calculated depending on actual (measured) and the theoretical densities of the specimen and as given in equation (2) [19].

\[ \text{Porosity} \% = [1 - \frac{\text{Actual density}}{\text{Theoretical density}}] \]  

While theoretical density is calculated by the following equation.

\[ \rho_{th} = \sum_{i=1}^{n} w_{t \times p_{i}} \]  

Where:
\[ \rho_{th} \]: the theoretical density of the specimen (g/cm³).
\[ n \]: no. of elemental powders.
2.5. Electrical test

Based on their electrical properties, materials are known as conductors, super conductors, semiconductors, and insulators. The conductivity and resistivity of material are important properties that characterize a variety of applications. The amount of resistance a longitudinal unit of material has to an electrical current is described by its resistivity ($\rho$), It is measured in ohms per meter and is determined by the area of the material segment and its length. The resistivity ($\rho$) is calculated by equation (4) [20].

$$\rho = R \cdot \frac{A}{l} \text{ (}\Omega\cdot\text{m)}$$

Where:
- $R$: is the electrical resistance of specimen
- $l$: is the length of the specimen
- $A$: is the cross-sectional area of the specimen

The electrical conductivity ($\sigma$) of a material is a measure of its ability to conduct electricity [21]. Equation (5) gives the electrical conductivity ($\sigma$).

$$\sigma = \frac{1}{\rho} \text{ (}\Omega\cdot\text{m})^{-1}$$

The specimens examined are cylindrical in shape, with a height of 6 mm and a diameter of 12.65 mm. The electrical test is carried out by (Keithley source meter 2401) device shown in figure 1.

![Keightley source meter 2401 device](image)

**Figure 1.** Keightley source meter 2401 device.

2.6. Magnetic testing

In composite materials, the magnetic properties are important which is studied in a variety of industrial applications, including electric motors, transformers, and generators electrical and electronic devices, computers, audio, and video equipment. The amount of magnetization induced by an external magnetic field is defined as the magnetization susceptibility of metal, which is calculated by equation (6)[22].

$$\chi_m = \frac{M}{H}$$

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Wt.: weight percentage (%)

$\rho_n$: density of elemental powder (g/cm³).
where:
- \( M \): is the magnetization, (emu/g).
- \( H \): is magnetic field, (G).

The magnetic properties of all specimens, pure Al and Al/Fe\(_3\)O\(_4\) + Ni, are measured at a constant frequency using a vibrating sample magnetometer (VSM) system model (Meghnatis Daghigh Kavir Co., Iran). The test was carried out at room temperature to measure magnetization in the presence of a magnetic field. Figure 2 shows: a- Vibrating sample magnetometer device VSM b- VSM schematic.

3. Results and Discussion

3.1. FESEM Results

Figure 3 show FESEM images with different weight percent of Fe\(_3\)O\(_4\) and a constant weight percent of Ni, a hybrid aluminum matrix nanocomposite Al/Fe\(_3\)O\(_4\)+Ni. It can be shown that the reinforcement materials are dispersed uniformly, with a little amount of porosity and no cracks. At the interfaces between Fe\(_3\)O\(_4\) and Ni with Al matrix, the additive materials Fe\(_3\)O\(_4\) and Ni diffuse. In fact, the function of the Ni element is to form new phases with Al grains and to diffuse at the Al and Ni interfaces. Furthermore, by reducing porosity and improving mechanical properties, the dispersion becomes more reliable.
3.2. X-Ray Diffraction analysis Results

Figure 4 displays X-Ray diffraction (XRD) results for specimens before and after Fe3O4 + Ni addition. The XRD study of specimens reinforced by Fe3O4+ Ni integrated into the Al matrix revealed that increasing the weight percentage of Fe3O4 with a constant amount of Ni increases the intensity of phase peaks Fe3O4 and Ni. In fact, XRD results show that increasing the weight percentage of additive materials causes the strength of the diffraction peaks to increase. Furthermore, since no other intermetallic inclusions are present, the reaction between Al and Fe3O4+ Ni is most likely in a steady-state thermodynamically [23].

![XRD diffraction peaks for the specimens.](image)

3.3 Physical Properties Results

3.3.1. Density and Porosity Results

Figure 5 shows the evolution of density for the manufactured nanocomposites Al/ Fe3O4 +Ni with 2wt% Ni nanoparticles before and after sintering. Ni nanoparticles cause more densification and plastic deformation, resulting in increased strain hardening and density in the hybrid nanocomposites.
The density and porosity values for Al/ Fe₃O₄+Ni specimens before and after sintering are shown in Tables 2 and 3.

**Table 2. Density and Porosity of Al/ Fe₃O₄+Ni specimens before sintering.**

| Sample No. | Fe₃O₄ wt.% | Theoretical density (g/cm³) | Experimental density (g/cm³) | Porosity ratio% |
|------------|-------------|------------------------------|------------------------------|-----------------|
| 1          | 0           | 2.7                          | 2.084                        | 22.82           |
| 2          | 2           | 2.872                        | 2.274                        | 20.81           |
| 3          | 4           | 2.920                        | 2.316                        | 20.68           |
| 4          | 6           | 2.968                        | 2.403                        | 19.04           |
| 5          | 8           | 3.016                        | 2.502                        | 17.04           |
| 6          | 10          | 3.064                        | 2.623                        | 14.39           |

**Table 3. Density and Porosity for the specimens Al/ Fe₃O₄+Ni after sintering.**

| Sample No. | Fe₃O₄ wt.% | Theoretical density (g/cm³) | Experimental density (g/cm³) | Porosity ratio% |
|------------|-------------|------------------------------|------------------------------|-----------------|
| 1          | 0           | 2.7                          | 2.189                        | 18.92           |
| 2          | 2           | 2.872                        | 2.527                        | 12.00           |
| 3          | 4           | 2.920                        | 2.585                        | 11.46           |
| 4          | 6           | 2.968                        | 2.683                        | 9.61            |
| 5          | 8           | 3.016                        | 2.762                        | 8.42            |
| 6          | 10          | 3.064                        | 2.909                        | 5.06            |

**Figure 5.** Relationship between density before and after sintering and wt.% of Fe₃O₄.

The Al particles close together and coalesce during the sintering process, forming necks and increase volume shrinkage, consequently, the density would rise. Figure 6 illustrates how increasing Fe₃O₄ and
2wt. Ni nanoparticles reduces porosity by filling voids. Furthermore, porosity reduction is highly dependent on sintering conditions such as temperature and time [7].

![Figure 6](image_url)

**Figure 6.** Relationship between porosity before and after sintering and wt.% of Fe$_3$O$_4$.

### 3.4. Electrical Properties Results

Keithley Source Meter 2401 was used to measure resistance and then calculated resistivity and conductivity for all specimens, including pure Al and Al/Fe$_3$O$_4$+Ni with different Fe$_3$O$_4$ weight percentages (2,4,6,8, and 10%) and constant ratio of Ni at (2wt.%). Table 4 shows the values of resistivity and conductivity for hybrid nanocomposite Al/Fe$_3$O$_4$+Ni.

**Table 4.** The electrical conductivity and resistivity value for the specimens with different Fe$_3$O$_4$ percentage. (Al/Fe$_3$O$_4$ +Ni).

| Fe$_3$O$_4$ wt. % | $\rho$ ($\Omega$.m) $\times 10^6$ | $\delta$ ($\Omega$. m)$^{-1}$ |
|------------------|--------------------------------|----------------------------|
| 0                | 61.11                          | 16364                      |
| 2                | 63.78                          | 15679                      |
| 4                | 65.95                          | 15163                      |
| 6                | 68.33                          | 14635                      |
| 8                | 70.23                          | 14239                      |
| 10               | 73.12                          | 13676                      |

The results show that for hybrid nanocomposite, resistivity increased with increasing Fe$_3$O$_4$ weight percentages and conductivity decreased with increasing Fe$_3$O$_4$ weight percentages, which agrees with [24]. Figures 7 and 8 demonstrate the relationship between Al/Fe$_3$O$_4$+2wt.% Ni hybrid nanocomposite resistivity, conductivity, versus Fe$_3$O$_4$ weight percentages.
3.5. Magnetic Properties Results

For the manufactured hybrid nanocomposites Al/Fe$_3$O$_4$+Ni with a different weight percentage of Fe$_3$O$_4$ nanoparticles, Table 5 show hysteresis parameters, saturation magnetization (Ms), remanence magnetization (Mr), coercive field (Hc), the relationship between Mr and Ms, and magnetization susceptibility (Xm), the magnetic properties were checked, at room temperature. The saturation magnetization (Ms) of hybrid nanocomposite increases as the weight percentage of Fe$_3$O$_4$ increases between (2.- 8wt. percent) and then decreases at 10wt. percent, which may be due to nonuniform distribution or locally agglomerated reinforced (Fe$_3$O$_4$+Ni) in the aluminum matrix [25].

Table 5. Hysteresis parameters of the Al/Fe$_3$O$_4$+2wt.% Ni Hybrid nanocomposite.

| Specimens | Ms emu/g | Mr emu/g | Hc (Oe) | Mr/Ms | Xm          |
|-----------|----------|----------|---------|-------|-------------|
| 0         | 0.1402   | 0.00123  | 6.5318  | 0.0088 | 5.394×10$^{-8}$ |
| 2         | 0.1635   | 0.00165  | 6.3125  | 0.0101 | 6.509×10$^{-8}$ |
| 4         | 0.3454   | 0.00191  | 3.5114  | 0.0055 | 2.472×10$^{-7}$ |
| 6         | 63.1512  | 26.7147  | 585.9585| 0.4230 | 2.709×10$^{-7}$ |
| 8         | 67.9034  | 27.5198  | 587.6809| 0.4053 | 2.904×10$^{-7}$ |
| 10        | 62.8463  | 24.9394  | 589.1835| 0.3968 | 2.681×10$^{-7}$ |

The value of Xm for a hybrid nanocomposite increases as the reinforced material is increased (Fe$_3$O$_4$), the highest value at 8wt.% and decrease at 10 wt.%. It may be attributable to same reason above as shown in Table 5 [25]. Figure 9 show magnetic properties and hysteresis curve for all specimens with different Fe$_3$O$_4$ percentage for pure Al and (Al/Fe$_3$O$_4$+Ni).
Figure 9. magnetic properties and hysteresis curve for all specimens.
4. Conclusions
The conclusion of the advanced processes' obtained findings can be summarized as follows:

1. Powder metallurgy route was used to successfully fabricate Al/Fe$_2$O$_3$+Ni hybrid nanocomposite.
2. The results of the (FESEM) examination revealed that the dispersion of the reinforcement materials is homogeneous in the aluminum matrix with a small amount of porosity and no cracks, as seen in the images of specimens for Al/Fe$_2$O$_3$+Ni hybrid nanocomposite.
3. The results of the XRD analysis show that increasing the weight percentage of additive materials causes the strength of the diffraction peaks to increase for Fe$_3$O$_4$ and Ni.
4. The results of density show, as compared to pure Al, the density of the Al/Fe$_2$O$_3$+Ni hybrid nanocomposite increases by 25.9% before sintering and 32.9% after sintering at 10 wt. percent Fe$_3$O$_4$ and 2 wt. % Ni.
5. When comparing pure Al to Al/Fe$_2$O$_3$+Ni, the porosity decreases by 36.9% before sintering and by 73.3% after sintering at 10wt% Fe$_3$O$_4$ and 2wt% Ni.
6. The electrical properties results show for Al/Fe$_2$O$_3$+Ni, the resistivity increased with increasing Fe$_3$O$_4$ weight percentages, while the conductivity decreased with increasing Fe$_3$O$_4$ weight percentages and constant Ni weight percentage.
7. According to the magnetic properties results, the saturation magnetization Ms and magnetization susceptibility Xm for Al/Fe$_2$O$_3$+Ni hybrid nanocomposite improved with increasing weight percentages of Fe$_3$O$_4$ nanoparticles and constant weight percentages of Ni.

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