Compression strength, dielectric and magnetic properties of new aluminium matrix hybrid nanocomposites

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Abstract. Nanocomposites are materials fabricated from two or more materials with different mechanical and electrical properties. Combining these materials produces a new designed material with new and better properties compared to the individual components. In recent years nanocomposites have been developed and employed almost in all industries. The current study deals with fabricating a hybrid nanocomposite (when there are a minimum of three materials, the composite is called as hybrid composite). Pure aluminium as the base matrix. Iron oxide Fe₂O₃ (alpha) and aluminium oxide Al₂O₃ (alpha alumina). Fe₂O₃ weight percentage (wt.%) is varied (1.5 , 2.5 and 5 % by weight) and the wt % of Al₂O₃ is held constant (2 wt.%). The new designed nanocomposite was produced using Powder Metallurgy (P/M) method. This method has been widely used for fabricating aluminium matrix nanocomposites (AMNCs), due to it is low costs and gives high accuracy as well as the ease of using. The matrix used was aluminium powder with an average particle size of (60µm) with 99.6% purity and Fe₂O₃ (99% purity and 30 nm particle size) and Al₂O₃ with (99.5% purity and 14-20 nm particle size). The experimental results revealed that the microstructure images of composites showed uniformly distributed of Fe₂O₃ and Al₂O₃ in aluminium matrix. The maximum compressive strength is 152 MPa in nanocomposite containing (1.5 Fe₂O₃ + 2 Al₂O₃) wt.%. The improvement percentage was recorded to be 30% for CS. The real and imaginary components of relative permittivity decrease with increase of frequency. The hysteresis curve of nanocomposites showed that the magnetic properties were improved for all the nanocomposites but the better response of magnetic properties was found with the nanocomposite of (1.5% Fe₂O₃ + 2% Al₂O₃) content.

1.Introduction
Aluminium metal matrix nanocomposites (AMMNCs) are the most common in various industries such as automotive, aerospace, electronics and power transmission lines, because of the high ratio of strength to weight and good electrical properties. The main properties on which the applications of aluminium is based are the high mechanical strength, low density and relatively good electrical conductivity [1].

A. Tan, et al. [2] (2016), investigated the effect of the hybrid reinforced materials (micro SiC + nano TiB₂) with various content on the microstructure and mechanical properties of the aluminium using powder metallurgy method. The experimental results revealed that the mechanical properties are improved with increase in content of TiB₂ nanoparticles reinforcement. The best improvement was observed in composite including (5 TiB₂ + 10 SiC wt%). Ferreira, et al. [3, 4] (2016, 2018), designed and fabricated Al. alloy (AMCs) reinforced with different percentages of nano-Fe₃O₄
magnetic particles using powder metallurgy method. Microstructural, electromagnetic and mechanical properties were studied. Compression test results showed the effect of Fe₃O₄ on the impact resistance of the nanocomposites. In addition to, the results were observed good improvement in magnetic properties with increase wt% of Fe₃O₄ particles. S. J. Vijaya, et al. [5] (2018), studied the effect of the hybrid nanoreinforced materials (CNTs + Fe₂O₃) with various contents on the electromagnetic properties of the aluminium using modelling techniques to analyze the material properties. The results revealed significant improvement in the electromagnetic properties of hybrid nanocomposites compared to the base alloy. It was found that as the vol.% Fe₂O₃ increased, the magnetic flux density value as well as energy increased. But the magnetic field strength decreased with the increase in the vol.% Fe₂O₃ particles.

The aim of present work is to fabricate of an aluminium metal matrix nanocomposites (AMMNCs) based on powder aluminium with 60 µm using powder metallurgy (P/M) route. The microstructure, compressive strength, permittivity, dielectric loss and magnetic permeability testing are attempt to be make using different samples sizes of AMMNCs hybrid (Al₂O₃ 14-20 nm and Fe₂O₃ 30nm). The major objective of the current work is the preparation of the composites containing (0, 1.5 Fe₂O₃ + 2 Al₂O₃, 2.5 Fe₂O₃ + 2 Al₂O₃ and 5 Fe₂O₃ + 2 Al₂O₃) wt%.

2. Materials and methods

2.1. Materials and nanocomposites fabrication
In this work nano Fe₂O₃ and Al₂O₃ particles were reinforced pure aluminium. The size of nano Fe₂O₃ (α) was 30 nm while the Al₂O₃ (α) particles varies from 14-20 nm. The manufacturing of nanocomposites will be using powder technology method.

The aluminium powders were mixed with the nanoparticles to prepare nanocomposites with (1.5, 2.5, and 5) wt.% of Fe₂O₃ and 2 wt.% of Al₂O₃ for each percent reinforcement, these powders are added to alcohol to be mixed well in two steps: First, mixing by magnetic field for 5 min. Second, mixing by (ultrasonic –cleaner) for 20 min, In order to increase the homogeneity between the particles of the material. These mixed powders were dried at 70 °C for 15 min, after that the powders have been added 5% of (pva) as a lubricant to reduce friction during compressive, then leave dry at room temperature. The powder mixtures were compressed at room temperature under uniaxial press at 250 MPa. The samples were sintered at 600 °C under argon gas. By two steps, first step at 300°C for (1 h) and second step at 600 °C for (2 h). The total weight per case (added percentage) was 25 g. Table 1 shows the percentage and weight details.

| Al (g) | Fe₂O₃ (g) | Al₂O₃ (g) | Total (g) | Fe₂O₃ wt% | Al₂O₃ wt% |
|--------|-----------|-----------|-----------|-----------|-----------|
| 25     | 0         | 0         | 25        | 0         | 0         |
| 24.125 | 0.375     | 0.5       | 25        | 1.5       | 2         |
| 23.875 | 0.625     | 0.5       | 25        | 2.5       | 2         |
| 25.25  | 1.25      | 0.5       | 25        | 5         | 2         |

2.2. Experimental measurements
During microstructure analysis, proper preparation of the specimen surface requires a small sample of the nanocomposite selected, prepared and manufactured. Then polishing in addition to coating
samples with gold and palladium spray (for 135 seconds), to discover accurate content and obtain the best accuracy. After preparing the microstructure specimens, they were tested by Field Emission Scanning Electron Microscope (FESEM), using test device type (Cam Scan Mv 2300).

The mechanical properties of the manufactured specimens were obtained by using compression test machine model STM-50 according to standard ASTM E-4 with capacity 50KN. Cylindrical specimens of 14 mm height and 10 mm diameter with compression speed of 0.5 mm/min at room temperature were tested.

Base metal and composite material samples are subjected to electrical tests using (IVIUMSTAT.XR) device at room temperature with (1 V) voltage, (1 A) current avg. and frequency ranges between 10 KHz to 10 MHz. The electrical tests include dielectric loss ($\delta$), real and imaginary relative permittivity ($\varepsilon$).

The magnetic test measures the magnetic properties of powders and the composites by using a vibrating sample magnetometer (VSM) device model (Meghnatis Daghigh Kavir Co., Iran) at a constant frequency. The test measure the magnetization against the magnetic field at room temperature. As the magnetic field is varied over a limited range, the magnetization of the powders is measured by the sense coils with a lock in amplifier.

3. Experimental results and discussion

3.1. Microstructure results

Figure 1 shows the FESEM images under 200 nm or under 800000X magnification respectively. These images show the microstructure of pure aluminium and nanocomposites containing (1.5 Fe$_2$O$_3$ + 2 Al$_2$O$_3$, 2.5 Fe$_2$O$_3$ + 2 Al$_2$O$_3$ and 5 Fe$_2$O$_3$ + 2 Al$_2$O$_3$) wt.% revealing the presence of Fe$_2$O$_3$ and Al$_2$O$_3$ and homogenous dispersion in the Al matrix. The images contain Fe$_2$O$_3$, Al$_2$O$_3$ concentrated at the grain boundaries of aluminium particles because the grain boundaries energy is high, attracting foreigner particles to it. The gray light which represents the Al matrix. Microstructure of nanocomposites reveals uniform distribution of the hybrid nanomaterials and less porosity along the grain boundaries. The nano ceramic Al$_2$O$_3$ is shown as white, while the nano Fe$_2$O$_3$ is dark.
3.2. Compression results

Figure 2 shows the compression engineering stress – strain curves of as-received and different weight percentage of Fe₂O₃ + Al₂O₃ nanomaterials. It can be seen that the compression strength of the nanocomposite containing (1.5 Fe₂O₃ + 2 Al₂O₃ wt% ) is significantly enhanced compared to as-received and the other nanocomposites. The ultimate compression strength of the base metal with (1.5 Fe₂O₃ + 2 Al₂O₃ wt% ) was improved by 30 % compared with the as-received metal. This finding is agreed with the results reported by [6]. The experimental results of compression tests of the nanocomposites containing different weight percentage are listed in table 2, which shows slightly enhanced of failure strain of as-received. For analysis the compression strength and failure strain, 12 specimens were tested with 0.5 mm/min test speed and the average results of three samples for each percentage were recorded.

Figure 1. Shows the FESEM images under 200 nm.
Figure 2. Shows the compression engineering stress – strain curves.

Table 2. Experimental compression testing at room temperature (RT).

| Composites                                     | Compression strength (MPa) | Failure strain |
|------------------------------------------------|----------------------------|----------------|
| As-received                                    | 117                        | 0.3635         |
| Al + (1.5% Fe$_2$O$_3$ + 2% Al$_2$O$_3$) wt.% | 152                        | 0.6467         |
| Al + (2.5% Fe$_2$O$_3$ + 2% Al$_2$O$_3$) wt.% | 125                        | 0.5362         |
| Al + (5% Fe$_2$O$_3$ + 2% Al$_2$O$_3$) wt.%   | 90                         | 0.4853         |

Figure 2 and table 2 show that the compression strength (CS) and failure strain (FS) of the nanocomposite were improved with increasing the Fe$_2$O$_3$ + Al$_2$O$_3$ weight percentage. The CS was obtained to be 117 MPa for as-received and 152 MPa at (1.5% Fe$_2$O$_3$ + 2% Al$_2$O$_3$) nanoreinforced materials, with 30% improvement factor compared to the base metal. The main reasons for above improvement can be attributed to many factors, the incorporation of harder Fe$_2$O$_3$ + Al$_2$O$_3$ particles for strengthening the nanocomposite resulted in hard nature of nanocomposite. The proper interfacial bounding between the pure aluminium with the harder nanoparticles, in addition to that, the nanoparticles were homogeneous and uniformly distributed into the base metal. Thus, the CS and FS are improved [4].

3.3. Electrical results (Permittivity)

Electrical properties of the pure aluminium and three nanocomposites were analyzed by testing carried out on the computerized universal testing device type (IVIUMSTAT.XR). By measuring the real, imaginary and static permittivity values of the metal according to Debye Dispersion theory, the relative permittivity can be described [7]:

\[ \varepsilon_r = \varepsilon' + j\varepsilon'' \]  
… (1)

Where: $\varepsilon_r$ is the relative permittivity  
$\varepsilon'$ is real part of permittivity  
$\varepsilon''$ is imaginary part of permittivity
When: 
\[ \varepsilon' = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + \omega^2 \tau^2} \quad \cdots (2) \]

\& 
\[ \varepsilon'' = \frac{\varepsilon_s - \varepsilon_\infty}{1 + \omega^2 \tau^2} \quad \cdots (3) \]

Where: \( \varepsilon_\infty \) is Dielectric constant at high frequency
\( \varepsilon_s \) is Dielectric constant at low frequency
\( \omega = 2\pi f \), is Angular frequency of the electric field
\( \tau \) is Relaxation time.

The dielectric loss angle (\( \tan\delta \)), is taken as measure of dielectric loss and is known also as loss tangent.

\[ \tan\delta = \frac{\varepsilon''}{\varepsilon'} \quad \cdots (4) \]

Figure 3 shows the relation of real and imaginary parts of permittivity against frequency (log Hz). Figure 4 shows a comparison of dielectric loss and relative permittivity properties of hybrid nanocomposites with different amount of hybrid reinforcements (a, b, c, d). The relative permittivity (\( \varepsilon_x \)), volume resistance (\( R_x \)) and dielectric loss (\( \tan\delta \)) were measured to test the electrical properties of the hybrid nanocomposites materials. Figure 3 shows the electrical properties of as-received and hybrid nanocomposites for different amount of \( \text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3 \) against log frequency (log Hz). These properties revealed a nonlinear relation by adding the hybrid amount of nanoparticles materials.

(a) As-received  
(b) 1.5%  
(c) 2.5%  
(d) 5%

**Figure 3.** Shows the curves of the real and imaginary parts of permittivity
Figure 4. Shows the curves of relative permittivity and dielectric loss angle

Where $\varepsilon'$ (black line) is real part of permittivity and $\varepsilon''$ (blue line) is the imaginary part of permittivity in figure 3. Tan (delta) (black line) is the dielectric loss angle and $C_s/C_o$ (blue line) is the relative permittivity in figure 4.

The aluminium doped 1.5% $\text{Fe}_2\text{O}_3 + 2\% \text{Al}_2\text{O}_3$ having higher values of $\varepsilon'$, $\varepsilon''$ and $\tan\delta = \varepsilon''/\varepsilon'$ than those undoped (as-received). The electrical and mechanical properties can be improved by using one reinforced material [8].

It is observed from above curves that the relative permittivity values for as-received and nanocomposites varies in random behaviour with increase of frequency and then at high frequency it slightly decrease. This is true and agreed well with what concluded by Ref. [9]. Many attempts have been carried out on the improvements of dielectric properties using unique nanoreinforced material. Z.Li et al [10] manufactured Al. doped SiC nanopowder and they observed that the increasing in wt% of SiC leads to increasing in the dielectric loss. Aluminium – SiC nanocomposite containing different amount of SiC was fabricated and electrically tested. They deduced that the increase of electrical conductivity leads to significantly increase of dielectric loss [11].

3.4. Magnetic results

The absolute magnetic permeability ($\mu_{abs}$) is main magnetic property of a material, which is concerning to magnetic susceptibility ($\chi_m$). The magnetic susceptibility is a measurement of the amount of magnetization of a metal in reaction to an exterior magnetic field, as follows [12]:

$$\mu_{abs} = (1 + \chi_m) \mu_0 \quad ...... \quad (5)$$
\[ \chi_m = \frac{M}{H} \]  
\[ \ldots (6) \]

where : \((\mu_0)\) is vacuum permeability, equal to \(4\pi \times 10^{-7} \text{ henry/m} \).

\((M)\) is the Magnetization, ampere per meter.

\((H)\) is Magnetic field, ampere per meter.

According to equations (5 & 6), the value of \(X_m\) (magnetic susceptibility) can be obtained by applying the above equation. It is clear that the value of \(X_m\) is maximum for the nanocomposite containing 1.5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\). Table 3 shows the value of \(X_m\) and absolute magnetic permeability \(\mu_{abs}\) with magnetic field \((H)\) between (12000 to -12000 A/m) for four types of composites. Maximum magnetization \((M)\) occurred at the nanocomposite containing 1.5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\) which is equal to 0.11666 A/m. The improvement in \((M)\) value equal to 26% compared to the magnetization for pure aluminium. Figure 5 shows the variation of magnetic field \((H)\) against magnetization \((M)\).

### Table 3. Magnetic properties of nanocomposites with different hybrid nanomaterials.

| Composites                  | \(X_m\) \((\text{H/m})\) | \(\mu_{abs}\) \((\text{H/m})\) | Magnetization \((M)\) \((\text{A/m})\) |
|-----------------------------|---------------------------|---------------------------------|-------------------------------------|
| Pure Aluminium              | \(7.7 \times 10^{-6}\)   | \(1.254 \times 10^{-6}\)        | 0.0927 to -0.0927                   |
| Al + (1.5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\)) wt.% | \(9.73 \times 10^{-6}\)  | \(1.257 \times 10^{-6}\)        | 0.11666 to -0.11666                |
| Al + (2.5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\)) wt.% | \(8.95 \times 10^{-6}\)  | \(1.256 \times 10^{-6}\)        | 0.10733 to -0.10733                |
| Al + (5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\)) wt.% | \(8.79 \times 10^{-6}\)  | \(1.256 \times 10^{-6}\)        | 0.10544 to -0.10544                |

**Figure 5.** The variation of magnetic field \((H)\) against magnetization \((M)\)

Maximum \(\mu_{abs}\) was found in (1.5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\)) nanocomposite which was equal to \(1.257 \times 10^{-6} \text{ H/m}\) and reduced to \(1.256 \times 10^{-6} \text{ H/m}\) for (5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\)) nanocomposite, while this value was recorded as \(1.254 \times 10^{-6} \text{ H/m}\) for pure aluminium. The magnetization \((M)\) is an intrinsic property depends on the distribution of the nanoparticles. The experimental results also show that the value of \((M)\) for (1.5% Fe\(_2\)O\(_3\) + 2% Al\(_2\)O\(_3\)) nanocomposite is better than that of pure
aluminium and other nanocomposites. This can be attributed to relatively fine distribution of the nanoparticles for (1.5% FeO$_3$ + 2% Al$_2$O$_3$) nanocomposite [13]. The enhancement in µabs may be due to the improvement of the permeability (µabs) of the nanocomposites was due to the enhanced of dielectric loss properties. The improvement of microstructure and mechanical properties resulted in improving the µabs induced by containing the hybrid nanomaterial [14].

4. Conclusions

From the experimental results that have been obtained, the following remarks can be listed below:

- From microstructure observation it would be concluded that the hybrid nanomaterials (FeO$_3$ + Al$_2$O$_3$) could be uniformly distributed in pure aluminium. Adding FeO$_3$ and Al$_2$O$_3$ nanoparticles to the aluminum matrix significantly improved their mechanical properties. The best improvement which observed in ultimate compression strength was 30 % at (1.5% FeO$_3$ + 2% Al$_2$O$_3$) wt.%.
- Adding FeO$_3$ and Al$_2$O$_3$ nanoparticles to the matrix significantly improved their electrical properties. In addition it was observed that the real and imaginary values of relative permittivity for all nanocomposites decreased with the frequency increasing at specific range.
- The increase in weight percentage of nanoparticles FeO$_3$ and Al$_2$O$_3$ led to increase the magnetic properties of the nanocomposite studied. However, the best magnetic properties were found in nanocomposite containing (1.5% FeO$_3$ + 2% Al$_2$O$_3$) wt.%. The hysteresis curves for the three nanocomposites with pure aluminium gave the measured magnetization value was equal to 0.0927 A/m for pure aluminium, 0.11666 A/m for 1.5% FeO$_3$ + 2% Al$_2$O$_3$, 0.10733 A/m for 2.5% FeO$_3$ + 2% Al$_2$O$_3$ and 0.10544 A/m for 5% FeO$_3$ + 2% Al$_2$O$_3$.

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