Improvement of Mechanical Properties of Magnesium (Mg) Matrix Composites Reinforced with Nano Alumina (Al$_2$O$_3$) Particles

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Abstract. Nano alumina particle-reinforced magnesium was fabricated by powder metallurgy technique. Powders were mixed by ball milling (without balls) for 6 hours at rotation speed 60 rpm. Then, the powder was compacted at 550 MPa and sintered at 530˚C for 2 hours. The magnesium contained 0.25, 0.5, 1, 1.5, 3, and 5 vol. % nano alumina Al$_2$O$_3$. The physical, mechanical properties and microstructures, such as density, porosity, compressive strength, hardness, wear rate, SEM and EDX, were measured and compared with the values of unmodified pure magnesium samples. The results revealed an improvement with Al$_2$O$_3$ in Mg matrix composites. The greatest improvement in UCS and Hv values were about 197 MPa and 70 Hv at the volume fraction of 3 vol.% Al$_2$O$_3$ respectively. The wear tests showed that wear resistance was increased with the increasing of the reinforcement volume fraction of Al$_2$O$_3$.

1. Introduction
Magnesium is the eighth most abundant element on earth, making up almost 2.7% of the weight of the Earth's crust [1]. Magnesium is one of the lightest engineering materials structurally, having a density of 1.74 g/cm$^3$ [2]. It is approximately one-fifth lighter than steel and two-thirds lighter than aluminum and is attractive for use in various applications [3]. It has better ductility, vibration dampening, and noise properties than aluminum [4].

Ceramic reinforcement is often used for magnesium matrix composites. This is due to the excellent intrinsic properties of ceramic materials. Ceramic materials have excellent elastic modulus, high hardness, excellent strength, low density and very good thermal conductivity. However, they have some limitations including very low ductility, low wettability and low compatibility with magnesium matrix.

Widely-used ceramic reinforcements are Al$_2$O$_3$, SiC, TiC, Y$_2$O$_3$, SiO$_2$, etc. Among these reinforcements, Al$_2$O$_3$ and SiC are particularly well known. Even though the improvements conveyed by these mechanical properties are inferior compared to fiber reinforcements, they are considered advantageous in terms of processing, cost and other properties such as compressive strength.

2. Theoretical background
A review of the previous research makes it clear that studies and investigations of magnesium matrix composites are wide-ranging and diverse. Due to the importance of magnesium's use in commerce, transportation, industries, aerospace, defense, automotive, sports and recreation, many researchers have considered it in their studies, but there is still a need to classify different grades of Mg-MMCs based on a reliable standard.

Particulate reinforcement provides improvement in properties such as erosion and wear resistance, better damping properties, higher stiffness, and lower thermal expansion coefficient compared to unreinforced metals and alloys. [3][5].

In general, the size of the particulates used in the Mg matrix is on the micron and submicron scale [3]. Although the strength level can be raised, the main issue experienced with micron particulate
reinforcement is the lowering of the ductility of magnesium. Recently, researchers have discovered that the use of nano-particulates as reinforcement could enhance both ductility and strength of Mg [6][7].

The aim of this study is to present the experimental results of the studies regarding nano-sized alumina particles, their dispersion homogeneity, hardness, compression and wear resistance properties.

3. Experimental procedures

3.1. Raw materials
In this study, the matrix material was magnesium (Mg) powder with average particle size ~ 59.454 µm and a purity percentage of 99 %. The density of the magnesium powder was 1.738 g/cm$^3$. The nano alumina (Al$_2$O$_3$) powder had an average particle size ~ 20 nm and the purity percentage was 99.9 %. The density of alumina powder was 3.97 g/cm$^3$ where used as the reinforcement phase.

3.2. Processing
The powder metallurgy method was used to prepare both magnesium and nanocomposites (Mg /Al$_2$O$_3$) with variants containing 0.25, 0.5, 1.5, 3, and 5 vol. % of nano alumina, respectively. A ball mill was used as a roller mixer (without balls) for mixing these powders together with good dispersion, in which the mixture filled a volume of 50 % of the size of the container. 1 vol.% of stearic acid was added as a process control agent to prevent oxidation of materials, cold welding of particles reduces the possibility of Al$_2$O$_3$ agglomeration and separation during mixing [8]. The rotation speed of the cylinders was about 50 rpm for 6 hours [9]. Various homogenized powder mixtures of Mg and nano alumina were then compacted at a pressure of 550 MPa, to form a sample of 12 mm diameter and 9.6 mm height. The compacted samples were then sintered under vacuum (non-oxidizing atmosphere) starting from room temperature with an average heating rate of 10ºC/min, maintained until it reached the sintering temperature of 530 C, holding time set to be 2 hours. After switching off the furnace, the samples cooled inside the furnace gradually and slowly.

3.3. Density and Porosity Measurements
The density was determined, using Archimedes’ principle (ASTM C20-00), of sintered samples. The sample was weighed in air first (Wa), then it was suspended in distilled water and weighed again (Ww). A MonoBloc Instruments electronic balance with an accuracy of 0.001 g was used for recording the weights. The density of the composite samples was obtained using the following formula [10]:

$$\rho_{ex} = \frac{Wa}{Wa - Ww} \times \rho_{w}$$

$\rho_{w}=$ Density of water

The theoretical density ($\rho_{th}$) of the samples was calculated using the rule of mixtures as shown following formula [11].

$$\rho_{th} = [ (f_f \times \rho_f) + (f_{Mg} \times \rho_{Mg}) ]$$

where $\rho_f$ and $f_f$ density and the volume fraction of alumina, respectively $f_{Mg}$ and $\rho_{Mg}$ density and the volume fraction of magnesium

The porosity of the specimen was evaluated via density measurement according to the following formula:
\[ \text{Proosity} = \left(1 - \frac{\rho_{ex}}{\rho_{th}}\right) \times 100\% \]

3.4. Microstructure characterization
Microstructures of sintered composites have been investigated scanning electron microscopy (SEM) and X-ray spectroscopy (EDS).

3.5. Compression Test
Compressive properties of Mg and Mg/Al₂O₃ nanocomposites samples with a ratio of length to diameter equal to 0.8 (12 mm diameter and 9.6 mm length) were used and strain rate of 5x10^3 (m/m.min) according to ASTM E9-89 standard. The tests were performed with a device of 25 KN capacity load, CX M500 computerized system [12].

3.6. Micro hardness Test
Microhardness measurements were conducted on the polished Mg and Mg/Al₂O₃ samples. Vickers microhardness tests were performed by digital micro-hardness tester (TH-714) under a (25 gf = 0.245 N) test load and dwell time of 15 seconds, in accordance with ASTM E3 84-99 [13].

3.7. Wear Test
The samples were tested using the pin on the disc wear apparatus, according to ASTM G99 [14]. In this apparatus, the sample is installed into the arm which is loaded with specific weights. The sample (pin) comes into contact with the rotating stainless-steel disk surface as shown in Figure 1.

![Figure 1. Diagram of the principle (pin on disc)](image-url)

The weight method was used to calculate the rate wear of samples. The mass loss (ΔM) was divided by the sliding distance (S.D) and the wear rate calculated by using the following equation.

\[ \text{WR} = \frac{\Delta M}{S.D} \]
\[ \Delta M = M_1 - M_2 \]
\[ S.D = \omega \times r \times t \]

Where: - WR = Wear rate (g/m), ΔM = mass losses, \( \omega \) = rotating speed of the disc (rpm), r = disk radius, and t = slipping time (min).

The dimensions of the samples used in this test were 12 mm diameter and 9.6 mm length, loaded with three different weights of 5, 10, and 15 N, with different sliding distances (100 to 500 m). The rotation speed of the steel disc was 243.7 rpm.
4. Results and Discussion

4.1. Porosity and density measurements
Composite material of magnesium with nanoparticle of alumina, as shown in Table 1, appears to have a porosity higher than that of pure magnesium. The reason for this phenomenon is that magnesium has higher compressibility than its composite materials, and this will produce lower porosity than is found the composites. Also, due to the great hardness of Al₂O₃ particles will cause a reduction in the pressing capacity, and then an increase in porosity, above that of the pure magnesium. Usually, Al₂O₃ works as a preventive factor during the sintering process because of its high melting temperature in comparison with Mg, and this will cause formation of a weak network bond between Al₂O₃ and Mg, which leads to an increase in porosity to a level greater than in the pure magnesium. Moreover, with the addition of alumina ceramic particles as reinforcements, the density of magnesium composites will increase. This is not desirable, due to the lightweight applications of Mg composites [9].

| Material (vol. % Rei./ Mg) | density (g/cm³) | Relative density (%) | Porosity (%) |
|---------------------------|-----------------|----------------------|--------------|
|                           | Theoretical     | Experimental         |              |
| Magnesium                 | 1.738           | 1.691                | 97.27        | 2.73          |
| 0.25 Al₂O₃/Mg             | 1.744           | 1.692                | 97.05        | 2.95          |
| 0.5 Al₂O₃/Mg              | 1.749           | 1.693                | 96.79        | 3.21          |
| 1 Al₂O₃/Mg                | 1.760           | 1.694                | 96.25        | 3.75          |
| 1.5 Al₂O₃/Mg              | 1.771           | 1.797                | 95.77        | 4.23          |
| 3 Al₂O₃/Mg                | 1.805           | 1.705                | 94.48        | 5.52          |
| 5 Al₂O₃/Mg                | 1.850           | 1.701                | 91.99        | 8.01          |

4.2. Scanning Electron Microscopy (SEM) Test
The SEM microstructure images were taken for Mg and Mg/3 vol.% Al₂O₃ of sintered samples in order to give plentiful information about the distribution of the reinforcement particles, their agglomeration and the bonding between Mg and reinforcements.

Figure 2 shows a high degree of cohesion between magnesium particles and very few pores are present in the metal. No grain boundaries are to be seen in the magnesium sintered at 530°C: this may be caused by grain growth then grain coarsening, which will produce grain coalescence and disappearing of the grain boundaries, but the crystal structure of magnesium will not change to single crystal, the structure remains polycrystalline.

Figure 3 shows the micrographs of Al₂O₃ in the Mg matrix. It indicates that the nano alumina composite shows fewer interconnections. Although some agglomeration of Al₂O₃ particles can be observed, the distribution generally appears reasonably homogeneous, which indicates that most of the alumina particles were present in the form of agglomerates and clusters, both at the grain boundary and in the grain interior. This indicates a reasonable distribution of cluster/agglomerated alumina particles in the magnesium metal matrix. The identity of the Al₂O₃ particulates was confirmed through EDX point analysis. The nano Al₂O₃ particles are of a very small size, which made the number of Al₂O₃ particles very large and this led to the formation of clusters. The agglomerations have happened due to the tendency of nano particles to agglomerate, this phenomenon has occurred due to the higher specific surface of nano alumina in comparison with the micro Mg matrix. Obviously, the increase in the specific surface of the contacted particles will lead to a higher inter-particle friction, which is will cause a minimization of the homogeneity of particle distribution.
Figure 2. SEM image of pure magnesium

Figure 3. SEM image of (Mg + 3 n Al$_2$O$_3$) composite

4.3. Energy Dispersive X-Ray Spectroscopy (EDX)

Analysis was performed by the composition scanning energy-dispersive X-Ray (EDX), to analyze the elements and its distribution or chemical characterization of the sample.

Figure 4 EDX of pure magnesium indicates Mg element only, so there is no creation of oxidations or new phases existed in the sintered pure magnesium sample. Because the process active under high vacuum.

Figure 4. EDX of pure magnesium

The EDX elemental mappings for nano-composite samples, to evaluate the distribution of nano Al$_2$O$_3$ in Mg matrix composites, are shown in Figure 5.

In all composites that contain alumina, the detection of the retained alumina mainly depends on the overlap of the elemental mapping of Al and O (where Al and O elements cover the same area).
4.4. Compression test

Figure 6 shows the final compressive strength (UCS) of Mg-nano Al₂O₃. From the graph we can see that the addition of Al₂O₃ particles in the magnesium matrix increased the compressive strength. The best result for UCS was in the content of 3% vol. Al₂O₃. However, an additional increase in the content of Al₂O₃ resulted in a decrease in the final compressive strength of the composite.

A significant improvement in UCS with increasing percentage of nano Al₂O₃ reinforcement can be attributed as follows: 1) the effect of alumina particles which hinder the movement of dislocations in the magnesium matrix via the dispersion strengthening mechanism [15]. 2) Load bearing effects because the turnout of reinforcement load transfer relies on interfacial bonding between the matrix and the reinforcement. The hardness and strength of Al₂O₃ particles increases load-bearing capacity and effective transfer from soft matrix to solid reinforcement, due to good interconnection, hence results in improvement of compressive strength [16][17]. 3) Thermal stress and elastic modulus mismatch between the magnesium and Al₂O₃ particles [16].

4.5. Micro hardness test

The average micro hardnesses of (Mg/n Al₂O₃) nano composites with 0.25, 0.5, 1, 1.5, 3, and 5 Vol. % of Al₂O₃ reinforcement were found to be 46, 51, 54, 58, 70, and 65 Hv, respectively. The micro hardness of pure Mg was 39 Hv as shown in Figure 7. Microhardness values of nano composites increased markedly, compared to unreinforced Mg materials. Micro hardness data of composites shows an increasing trend of hardness, up to Mg-3% nano Al₂O₃ composite which shows an increment of 79 %. However, at Mg-5 % nano Al₂O₃ composite showed a reduced value of hardness due to the increased porosity percentage in the matrix of magnesium.
Shows the effect of alumina on the increased micro hardness of the samples with increase vol. % of alumina. This is due to 1) The reinforcement alumina’s hardness is greater than the hardness of magnesium matrix. This can be foreseen by the simplified basis of the hardness mixtures in the following equation: [15].

\[ H_c = V_m \cdot H_m + V_r \cdot H_r \]

Where: \( H \) is hardness, \( V \) indicates volume fraction, and the symbols, c, m, and r indicate composite, matrix, reinforcement, respectively.

2) The presence of hard \( \text{Al}_2\text{O}_3 \) particles, which acts as a higher constraint to localized deformation of the matrix during indentation and the alumina particles, render their inherent property of hardness to the soft matrix which significantly increases the hardness value [18].

**Figure 7.** Micro hardness data of Mg/\( \text{Al}_2\text{O}_3 \) nanocomposites

4.6. Wear test

Wear rate results are shown in two diagrams for each group and compared with the pure magnesium. The first diagram illustrates the wear rate as a function of three different applied loads (5, 10 and 15 N) in the produced composites after 300 m sliding distance. The second diagram illustrates the wear rate (g/m) as a function of sliding distance (m) in the produced composites under 10 N applied loads.

For the nano-composites shown in Figure 8 and Figure 9 it is noted that as the volume of particles increased from 0.25 % vol. to 5 vol.%, improvements were continually obtained in wear resistance and works perfectly under the increase in load where observed. The weight loss rises with the increase of the applied load and vice versa. The minimum wear was at Mg/5 vol.% \( \text{Al}_2\text{O}_3 \) composite. This may directly explain the decline in the weight loss as the nano \( \text{Al}_2\text{O}_3 \) particle, the hardness and strength of which is significantly higher than those of pure magnesium, acts as an obstacle against wear by resisting plastic flow.

Enhancements in strength and hardness could be due to a combination of various parameters caused by the introduction of hard ceramic nano-particles into the soft matrix [19].

The reduction in weight loss can also be attributed to the increased extent of magnesium oxidation as a result of higher interfacial temperature, resulting in the ability to maintain a stable and thick oxide film by the presence of alumina particles and as a result, Mg/\( \text{Al}_2\text{O}_3 \) composites show a better load bearing capacity, thereby lowering the wear rate [20].
5. Conclusions

1) The porosity in all the composites is higher than that in pure magnesium. For composites reinforced with nano Al₂O₃ particles, the highest porosity value observed (8.01) was with content of 5 vol.% nano Al₂O₃.

2) The nano composite with the volume fraction of 3 vol.% alumina has highest compressive strength, which is about 197 MPa.

3) The hardness values of Al₂O₃ reinforcements have a tangible effect on microhardness in the Mg matrix composites. The sample reinforced with 3 vol. % Al₂O₃ particles gave the highest micro hardness reach that exceeded magnesium’s by 79%.

4) An increase in volume fraction of alumina will increase the wear resistance of the Mg/Al₂O₃ composites.

6. References

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