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# Effect of Alumina (Al2O3) Particles on The Mechanical Properties of Magnesium (Mg)

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## Abstract

In the present study, magnesium-based composites reinforced with different volume fractions (3, 5, 10, and 15) vol.% of micro sized $Al_2O_3$ particulates were fabricated by powder metallurgy technique which involves mixed, compacted and sintered. Powders were mixed by ball milling (without balls) for 6 hours at rotation speed 60 rpm. Then powder was compacted at 550 MPa and sintered at 530°C for 2 hours. Microstructures of sintered composites have been investigated by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD) energy dispersive. SEM image of sinter samples exhibit good bonding between the magnesium matrix and the alumina. The microhardness and wear resistance of micro composites has been improved significantly compared to that of pure magnesium. Highest value of microhardness is 97 HV at the volume fraction of 10 vol.% $Al_2O_3$.

**Keywords:** Mechanical Properties, Magnesium, Powder Metallurgy.

## تأثير جزيئات الألومينا ($Al_2O_3$) على الخواص الميكانيكية للمغنيسيوم (Mg)

حيدر منعم عبد زيد ، عبد الرحمن نجم عبد ، هالة سلمان حسن

**الخلاصة:**

في هذه الدراسة ، تم تصنيع مركبات أساسها المغنيسيوم معززة بكسور حجمية مختلفة (3، 5، 10، 15 %) كنسبة حجمية. من جسيمات الألومينا المايكروية الحجم والتي تم تصنيعها بواسطة ميتالورجيا المساحيق والتي تتضمن الخلط ، الضغط والتلبيد. تم خلط المساحيق عن طريق خلاط الأسطوانة (بدون كرات) لمدة 6 ساعات بسرعة دوران 60 دورة في الدقيقة. تم ضغط العينات عند 550 MPa وتم تلبيدها عند 530 م° لمدة 2 ساعة. تم فحص المجهرية عن طريق مسح المجهري الإلكتروني (SEM) ، تحليل العناصر المركبة للمركب باستخدام التحليل الطيفي للأشعة السينية المشتتة (EDS) و أجري تحليل حيود الأشعة (XRD). تشير النتائج التجريبية لنتائج صورة SEM لمركبات العينات الملبدة إلى وجود ارتباط جيد بين ارضية المغنيسيوم والألومينا. تم تحسين صلادة و مقاومة البلى للمركبات بشكل ملحوظ عند مقارنةً بالمغنيسيوم النقي. أفضل القيم لتحسين 97 HV في الكسر الحجمي 10٪من $Al_2O_3$ أظهر اختبار الضغط أن مقدار للمقاومة الانضغاطية القصوى (UCS) أفضل قيم للتحسين بنسبة حجمية 5% من $Al_2O_3$ هي حوالي 179MPa .

## 1. Introduction

Magnesium is one of the lightest engineering materials structural with having a density of 1.74 g/cm$^3$ [1]. This promising material is used in many applications now because of its low density. Mg and Mg alloys have been used in many areas, making it the third widely used element of structural materials. Magnesium-based materials are attracting further attention in aerospace and automobile applications for their potential improvement of fuel provision by efficient weight decrease [2]. High strength mechanical properties and low density and of Mg makes it an excellent option for light-weight consumer electronics, sports and medical equipment in the future [3]. In recent years, Mg has also became increasingly appealing for medical applications because of its biocompatibility, biodegradability and similar density of tensile strength, elastic modulus and of human cortical bone[4].

A proper choice of reinforcement is a necessary factor to improve the properties of the matrix material. The selection of reinforcement is done based on the shape and size of the particles, processing method, manufacturing cost and required properties of the composite [5]. If the

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composite is to be used in a structural application, then the strength, modulus, and density are the primary considerable factors. Uniform distribution of reinforcements is also another important factor to improve the properties.

For Mg matrix composites, the reinforcement can be either ceramic, which is mostly used in metallic. Among these reinforcements, $Al_2O_3$ and SiC are well known. Even though the improvements of mechanical properties are inferior compared to the fiber reinforcements, they are considered to be advantageous in terms of processing, cost and some other properties like in compressive strength. Particulate reinforcement provides improvement in properties including improved erosion resistance and wear, better damping properties, higher stiffness, and lower thermal expansion coefficient compared to the un-reinforced metals and alloys [6][7].

## 2. Experimental Procedures

### 2.1 Samples preparation

In this study, the matrix material is magnesium (Mg) powder with 105 µm size and the purity percentage was 99 %. The density of magnesium powder was 1.738 g/cm$^3$ and micro-sized of alumina (Alpha $Al_2O_3$) particle with 30 µm, purity 99.1 % and density 3.97 g/cm$^3$ were used as reinforcement phase.

The powder metallurgy method was used to prepared both magnesium and micro composites (Mg/$Al_2O_3$) with difference containing 3, 5, 10, and 15 vol.% fractions of micro alumina respectively. The roller mixer (without balls) for mixing these powders together in order to get a good dispersion, In which the mixture filled a volume of 50 % from 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 $Al_2O_3$ agglomeration and separation during mixing [8].The rotation speed of the cylinders is about 50 rpm for 6 hours [9]. Various homogenized powder mixtures of Mg and micro alumina were then compacted. Uniaxial cold compaction process was implemented on the mixed material powders by stainless steel die to produce a sample with 12 mm dimension and length 9.6 mm. In order to find the appropriate pressure to be applied in pressing process, the mixture of Mg-10 vol.% micro alumina compacted with four different pressing process and they are (400, 500, 550 and 600) MPa. the compacted pressure which gives high green density will be chosen, without surface crack. Form the obtained results the pressure of (550 MPa) was appropriate for compaction process of powder and it was determined according to measured density and to avoid the negative effect of over-pressure on samples and mould. Sampling density is determined according to ASTM C20 standard [10]. The compacted sample was then sintered for sintering process electrical tube furnace under vacuum pressure 3x10$^{-6}$ bar (non- oxidizing atmosphere) starts from room temperature with average heating rate of 10ºC/min, maintained until it reaches the sintering temperature, holding time set to be 2 hours' time long, after switching off the furnace, then the samples are cooled inside the furnace gradually and slowly. Identifying the sintering temperature of the specimens based on a higher density. After sintering for four specimens with the same both volume fraction and pressing at 550 MPa under different temperatures (400, 470, 530, and 600)°C, below the magnesium melting point (649)°C, when the sample is highly dense, its sintering temperature will be chosen. The sintering temperature was chosen at 530 ° C for magnesium composites. The density variation against sintered temperatures and more details are shown in Figure 2.

### 2.2 Density and Porosity Measurements

The density was determined by using Archimedes technique (ASTM C20-00) of sintered samples, weighing the sample in air first (Wa) then suspended in distilled water and weighted again (Ww). An MonoBloc Instrument 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]:

$$\rho_{ex} = \frac{Wa}{Wa - Ww} \times \rho_w \quad \ldots(1)$$

$\rho_w$= Density of water

The theoretical densities of the samples was calculated using the rule of mixtures as shown in the following formula [11].

$$\rho_{th}=[( f_{Rei} \times \rho_{Rei})+( f_{Mg} \times \rho_{Mg})] \quad \ldots(2)$$

The porosity of the specimen was evaluated via density measurement according to the following formula:

$$prosity = \left(1 - \frac{\rho_{ex}}{\rho_{th}}\right) * 100\ \% \quad \ldots(3)$$

### 2.3 Microstructure characterization

Microstructures of sintered composites have been investigated scanning electron microscopy (SEM), X-ray spectroscopy (EDS) and X-ray diffraction (XRD) energy dispersive.

### 2.4 Compression Test

Compressive properties of Mg and Mg/ (3, 5, 10, 15) vol.% µ $Al_2O_3$ composites samples have a ratio of length to diameter equal to 0.8 (12 mm diameter and 9.6 mm length) and the subjected strain rate is 5x10$^{-3}$ (m/m. min) according to ASTM E9-89 a standard. The tests were performed with a device of 25 KN capacity load (CX M500) computerized system [12].

### 2.5 Micro hardness Test

Microhardness measurements were made on the polished Mg and Mg/$Al_2O_3$ 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].

### 2.6 Wear Test

The samples were tested using the pin on the disc wear apparatus according to ASTM G99 [14]. With this test, the sample (pin) is mounted into the holder which is loaded by specific weights. The sample (pin) comes in touch with the rotating stainless-steel disk surface.

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

$WR = \Delta M/S.D \quad \Delta M = M_1 - M_2 \quad S.D = \omega r t$

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

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

## 3. Results and Discussion

### 3.1 Measured Density and Porosity

It could be realized from Figure 1 that there is a decrease in porosity for compaction sample counting 10 vol.% micro alumina reinforcement from (28.41% to 18.13%) with the increase in the compaction pressure from 400 MPa up to a pressure range close to 550 MPa while after this amount, and with a further increase in compacting force, the decreasing in porosity were (from 18.13% to 16.82%). This reduction can be considered as a small improvement in the reduction of porosity compared to the improvement that occurred when the pressure changes from 400 MPa to 500 MPa.

It has been noted that high pressure (6oo MPa) produces cracks in the green sample and also deformation of the die. Therefore, in order to prevent an excessive load as well as increasing to a limit that might cause a die crack or die deformation and problems of high friction between the compressed powder and the stainless-steel die. A pressure of 550 MPa have been recommended as the best compacting pressure to be used in the powder compacting stage for producing green samples.

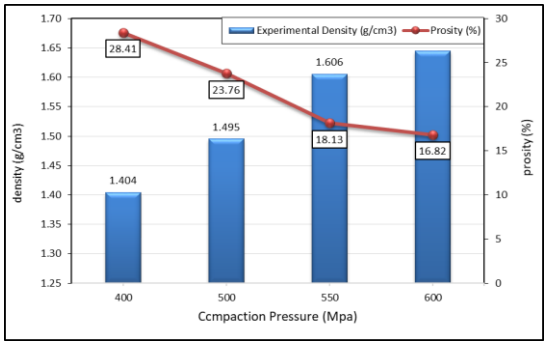


**Figure (1):** Density and porosity with different cold Compacting pressures for the green sample of 10 vol.% μ $Al_2O_3$/Mg

After the producing of the green sample at best compaction process 550 MPa, it will be sintered at a temperature less than the melting temperature of the metal matrix (magnesium).

Figure 2 presents the densities variation of the sintered samples which have been sintered to determine the best density that resulted from various temperatures. by subjecting forth different degrees of temperatures, those were 400, 470, 530 and 600°C to determine the best density that resulted from various temperatures. The temperature of 530°C have been identified as the best selected one which will provide the highest density after sintering. While with a further increase (to 600°C), the density drops dawn. For if the temperature is lowered to 530°C, then it will be not enough for the rearrangement of the particles to be able to reduce the porosity. Because high temperature leads to an increase the reaction between the composite components which increase the porosity, low sintering temperature was used (530°C). Therefore, it has been identified as the best selected temperature which will provide the highest density after sintering.

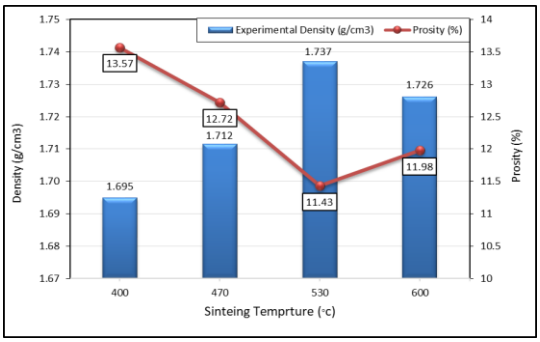


**Figure (2):** Density variation against sintered temperatures

Composite material of magnesium with micro particle of alumina as shown in Table 1 indicates that the experimental densities of the materials are lower than the theoretically calculated density. It can be seen that the porosity percentage increases with the addition amount of micro alumina particles.

The increase amount reinforcement increases porosity and this is consistent with the preceding studies [15]. It can be concluded that the difference between theoretical and experimental density is regarding to the presence of fine micropores [15] [16]. The first reason of the decreasing of density is related to the high hardness of $Al_2O_3$ reinforcement, it will work as an obstacle against the pressing capacity of samples, and this phenomenon will increase with the increase in the amount of $Al_2O_3$ and lead to an increase in porosity. The second reason is that the tremendous difference in the melting point between alumina and magnesium at the sintering stage; alumina melting point is (2045°C), which is much higher than the magnesium metal of (649°C), the mechanism of sintering process will be in the solid state. Therefore, alumina will have low tendency to make bonds with pure magnesium, and then produces a weak network formation between Mg and $Al_2O_3$ particles .The third reason is that the

particle size of micro magnesium is 105 µm, and the size of alumina is 30 µm, so the empty spaces between magnesium particles are not filled properly with the alumina reinforcement and causes an availability of residual empty spaces, which then leads to an increase in porosity [17].

**Table (1):** Results of density and Porosity of Mg-micro $Al_2O_3$ composites

| Material (vol.%/Mg) | density (g/cm³) | | Porosity (%) |
|---|---|---|---|
| | Theoretical | Experimental | |
| Mg | 1.738 | 1.691 | 2.73 |
| 3 $Al_2O_3$ | 1.805 | 1.715 | 5.01 |
| 5 $Al_2O_3$ | 1.850 | 1.720 | 6.99 |
| 10 $Al_2O_3$ | 1.961 | 1.737 | 11.43 |
| 15 $Al_2O_3$ | 2.073 | 1.724 | 16.82 |

### 3.2 Scanning Electron Microscopy (SEM) Test

The SEM microstructure images were taken for Mg and Mg/10 vol.% $Al_2O_3$ of sintered samples in order to give a lot of information about the particles' reinforcement distribution, agglomeration and description bonding between Mg and reinforcement.

Figure 3 illustrates a high degree of the cohesion between magnesium particles and very little of 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. This will produce grain coalescence and the disappearance of the grain boundaries, but the crystal structure of magnesium will not change to a single crystal. The structure stays polycrystalline.

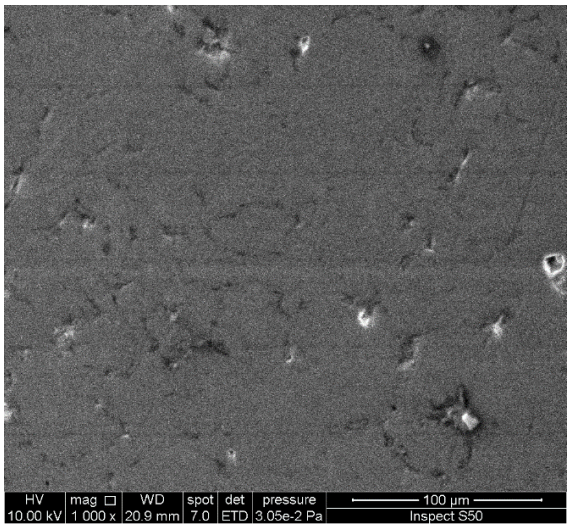


**Figure (3):** SEM image of pure magnesium

Microstructure examination as shown in Figure 4 at high magnification for micrographs 10 vol.% micro $Al_2O_3$ in Mg matrix exhibits good interface between the magnesium matrix and alumina. It can be attributed to the appropriate selection of the best compaction and sintering parameters [9]. In addition to a good compatibility between Mg and $Al_2O_3$. Alumina particles are seen individually, separated, and uniformly well distributed with a large bonding area of metal matrix. The reasonably uniform distribution of reinforcement particulates can be attributed to the adoption of suitable mixing parameters [18].

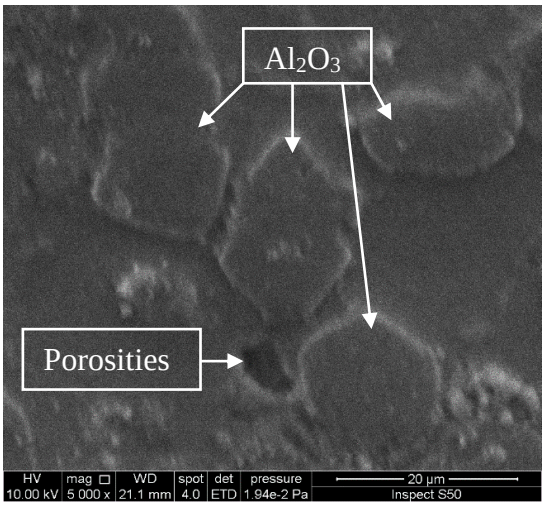


**Figure (4):** SEM image of Mg/10 vol.% $Al_2O_3$ composite

### 3.3 Energy Dispersive X-Ray Spectroscopy (EDX)

The analysis was performed by the composition scanning Energy-Dispersive X-Ray (EDX). in order to analyze the elements and its distribution or chemical characterization of the sample.

Figure 5 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 is active under high vacuum.

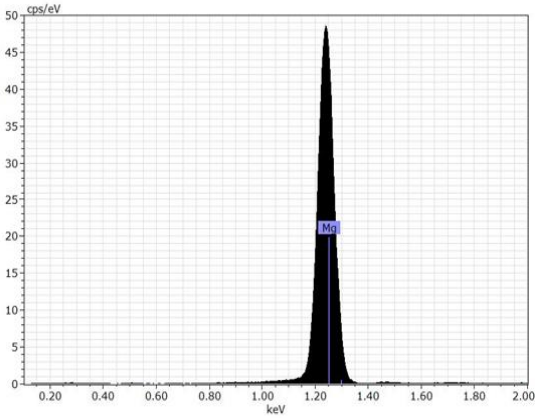


**Figure (5):** EDX of pure magnesium

EDX analysis of the 10 vol. % $Al_2O_3$ reinforced Mg matrix composite. It is clear that the peaks for magnesium, aluminium and oxygen elements as shown in Fig.6.

### 3.4 X-Ray Diffraction Analysis (XRD)

The XRD analysis was used to find out the phases present after sintering. from (Figure 7 and Figure 8) are the diffraction patterns obtained for the tested samples.

Figure 7 shows XRD results revealed that Mg only phase and did not detected any other phases at all.

As evident from Figure 8, for the formulation containing (10 vol.% micro $Al_2O_3$) from reinforcement. It can be seen that apart from the two predominant phases of Mg and $Al_2O_3$, MgO and the $Al_2MgO_4$ phase was detected only. The presence of the same phase peaks.

The presence of MgO in the structure is attributed to high chemical activity between Mg and O during the sintering process[19].Ternary compound $Al_2MgO_4$ can be attributed to be formed by inter-lapping of these three elements on the boundaries between the matrix and $Al_2O_3$particles, because of $Al_2O_3$ unstable in Mg reacting to form spinel, $Al_2MgO_4$ reaction of the reinforcement. It can severely degrade the properties of the composite [20]. Energy dispersive X-ray (EDX) analysis confirmed the presence of elements.

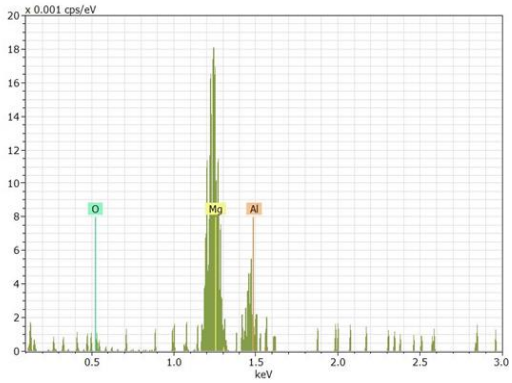


**Figure (6):** EDX of Mg/10% μ $Al_2O_3$ composite

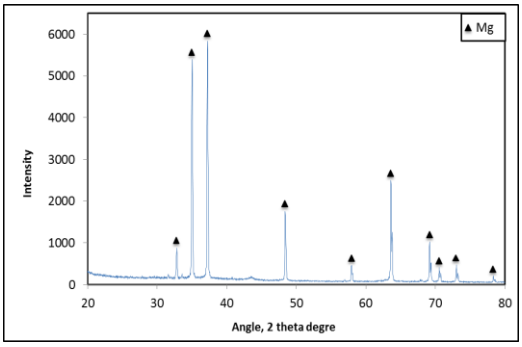


**Figure (7):** XRD analysis pattern of pure magnesium

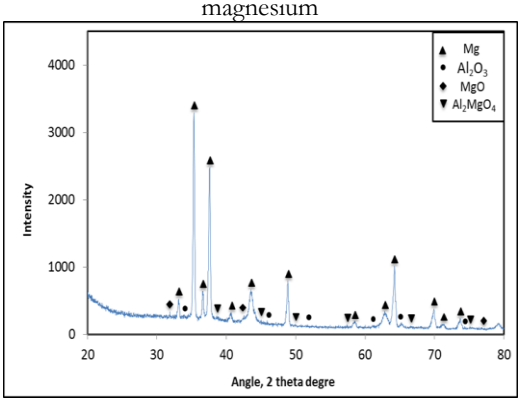


**Figure (8):** XRD analysis patern of Mg/10 vol.% $Al_2O_3$ composite

## 3.5 Compression Test

Figure 9 shows the final compressive strength (UCS) of Mg-$Al_2O_3$. From the graph, the addition of $Al_2O_3$ particles in the magnesium matrix was found to increase the compressive strength. The best result of UCS in the content of 5 vol.% $Al_2O_3$. However, an additional increase in micro alumina exceeding the 5 vol.% up to 15vol.%, compressive strength will start to minimize. This could be explained by the continuation of increasing the amount of additive after 5 vol.%. It causes the increased porosity and particle clustering which is responsible for reducing UCS, and ductility despite the beneficial influence of grain refinement [21].

A significant improvement in UCS with increasing percentage of micro $Al_2O_3$ reinforcement can be attributed as follows: 1) the effect of $Al_2O_3$ particles which are employed as a hinder to prevent the movement of dislocations in the magnesium matrix via the dispersion strengthening mechanism [21]. 2) Load bearing effects due to the turnout of reinforcement Load transfer relies on interfacial bonding between the matrix and the reinforcement. Hardness and stronger of $Al_2O_3$ particles which increases load-bearing capacity and effective transfer from soft matrix to solid reinforcement due to good interconnection. Hence the results in improvement of compressive strength [19][22]. 3) Thermal stress and elastic modulus mismatch between the magnesium and $Al_2O_3$ particles could be because of high dislocation density presence of reinforcements increases. The CUS because they cause inhomogeneous deformation and high-density dislocations .The increase in dislocation density of the composite is related to mismatch of the elastic modulus (E of Mg and $Al_2O_3$ are 44 and 472 GPa, respectively [1]) and coefficient of thermal expansion (CTE of Mg is $27.1\times10^{-6}$ $K^{-1}$ and $Al_2O_3$ particulates $7.4\times10^{-6}$ $K^{-1}$[23]) between the matrix and the reinforcement material. Therefore, this large difference in CTE and E values lead to the formation of dislocations and rise the strength [19]. And 4) The grain refinement might have an influence on Strength improvement [21].

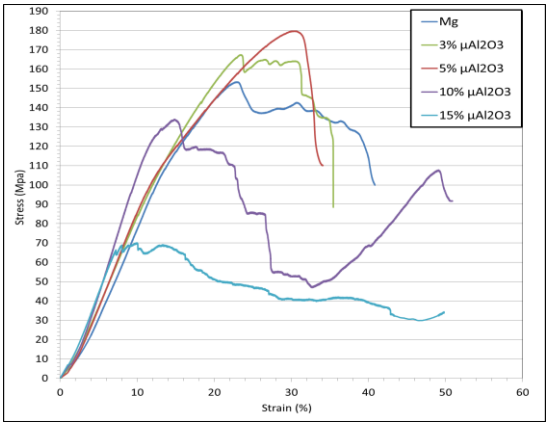


**Figure (9):** compression strength results of the Mg/μ $Al_2O_3$ composites

### 3.5 Micro hardness

The average micro hardness of Mg/$Al_2O_3$ composites with 3,5,10, and 15 Vol. % of $Al_2O_3$ reinforcement were found to be 43, 56, 97 and 61 Hv, respectively. The micro hardness of pure Mg was 39 Hv. As shown in Figure 10, Microhardness values of micro composites increased markedly compared to unreinforced Mg materials. Micro hardness data of composites shows an increasing trend of hardness up to Mg-10 % micro $Al_2O_3$ composite shows an increment of 149%. However, at Mg-15% µ $Al_2O_3$ composite has a reduced value of hardness due to the increase of the porosity percentage in the matrix of magnesium.

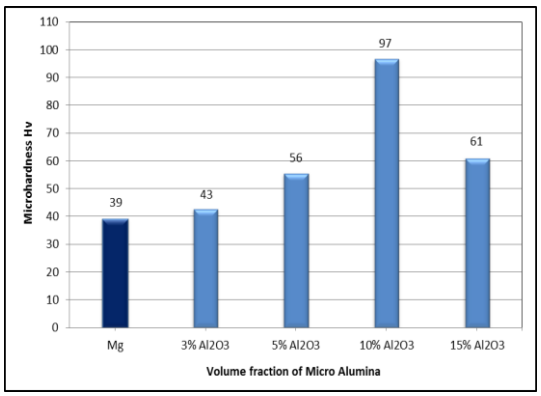


**Figure (10):** Micro hardness data of Mg/Al2O3 microcomposites

The increase in microhardness of micro composites can be attributed primarily to: a) since $Al_2O_3$ particulate is inherently much harder than mg matrix, this can be portended by the simplified basis of the hardness mixtures (Equation following) [21].

$$H_c = V_m . H_m + V_r . H_r \quad \ldots(4)$$

Where: H is hardness, V indicates volume fraction, and the symbols, c, m, and r indicate composite, matrix, reinforcement, respectively.
b) the presence of hard $Al_2O_3$ particles which acts as a higher constraint to localized deformation of the matrix during indentation [24].
c) The homogenous distribution of hard $Al_2O_3$ particulates in soft Mg matrix [19].

### 3.6 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.

Figures 11 & 12 show the improvement in wear resistance of micro-composite against the sliding distance and applied loads. It could be seen that in changing of the amount of alumina from 3 vol.% to 10 vol.%, a good improvement in wear resistance has been achieved. While as the amount of alumina increases from 10 vol.% to 15 vol.%, wear resistance is not improved in same expected way. The main reason of the decreasing in wear rate with increasing amount of alumina in the produced composites is due to the higher hardness of alumina reinforcement added which causes an increase in the hardness of fabricated composites. According to the hardness rule of mixtures, the composite hardness increases with the increase in the amount of alumina addition and reduction in particle size. The drastic reduction in wear mass rate may be attributed to the enhancement in hardness of the composite reinforced by $Al_2O_3$ particles and greater reduction of direct load contact between the Mg - $Al_2O_3$ composite surface and disc in comparison with pure Mg due to load bearing component action of hard $Al_2O_3$ particles [25] However, when there is an increase of 15 vol.% $Al_2O_3$, a decrease in wear rate could occur due to the increasing porosity and decreased hardness.

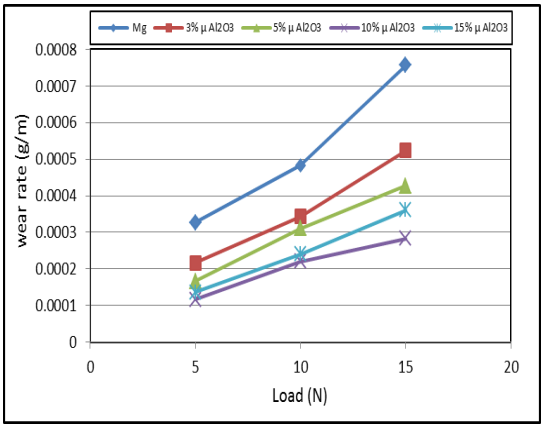


**Figure (11):** Wear rate of Mg /$Al_2O_3$ micro composite after 300 m sliding under 5, 10, and 15N applied loads

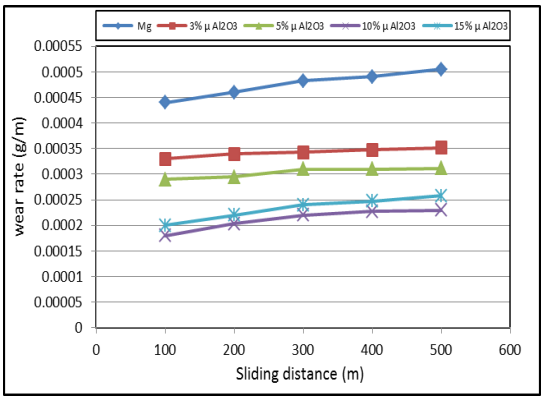


**Figure (12):** Variation wear rate with sliding distance for Mg /$Al_2O_3$ micro composite under 10 N applied load

## 4. Conclusions

The main conclusions of this study are the following:

1. Powder metallurgy (PM) method was successfully used to produce pure Mg and Mg-micro $Al_2O_3$. When applying a Compaction load at 550 MPa and sintering temperature of 530˚C for two hours is sufficient enough to fabricate a coherent composite according to the SEM analysis.

2. The porosity of the composite with 10 vol.%. micro $Al_2O_3$ is reduced from approximately 28.41% to 16.82% with compaction with increasing of compaction pressure from 400 MPa to 600 MPa respectively. It was shown that the best compaction pressure was at 550 MPa for the composite and the porosity in all the composites is higher than that in pure magnesium. The highest porosity values observed is (16.82) with content of 15 vol.% micro $Al_2O_3$.
3. The composite with 5 vol.% micro alumina has higher compressive strength which is about (179 MPa).
4. Hardness values of $Al_2O_3$ reinforcement have a tremendous effect on the microhardness in the Mg matrix composites. The reinforced with 10 vol. % $Al_2O_3$ particles gave the highest micro hardness reached that improved to 149 % as compared to magnesium.
5. The increasing volume fraction of alumina will increase the wear resistance of the Mg/$Al_2O_3$ composite the best result of when containing 10 vol. $Al_2O_3$.

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