



Mechanical characterization of micro and nano alumina-Aluminum composites produced by powder metallurgy process

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Abstract

Aluminum metal obtained great attention in the new era. It's a wide usage in automotive industries and airplanes. Also, Aluminum metal can be used for improving the mechanical properties and an intension to overcome the basic lack of aluminum/aluminum alloy which is low wear resistance are nowadays ambition. Micro sized alumina is an elderly major reinforcement additive while, the nano sized Al_2O_3 phase is recently used and studying its effects on aluminum properties which are still in need for more study. A trend has been observed in the field of aluminum based composite to employ alumina phases in micro and nano for creating composites of unique properties. A process of powder metallurgy technique has been used to fabricate, respectively, composites with an alpha micro (30 μm) and gamma nano (20 nm) sized alumina reinforcing aluminum metal matrix of 45 μm size. Also, the weight percentages of the reinforcement materials used were (5, 10 and 15wt %). Specimens have been manufactured according to ASTM standards. After the mixing of powder, the green samples have been obtained from cold axial pressing under a compaction pressure of 500MPa and sintered under 500°C for two hours in a vacuumed pressure less tube furnace.

Results revealed that as the amount of Al_2O_3 content increases, the hardness, wear resistance of the micro and nanocomposites increases significantly. Compressive strength in both of micro and nanocomposites records its maximum amount within an additive of 10wt %. Upon a comparison of the mechanical properties for the overall percentages of micro and nano composites. Nano composites presented higher wear resistance, hardness and compressive strength superior than the micro composites. As a general indication, addition of alumina reinforcement contributed in improving the mechanical properties of the pure aluminum matrix. While the nano alumina provides higher performance in the improvement of mechanical properties.

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Keywords: Metal matrix composites; Nano composite; Micro composite; Strength; Wear; Compression; Ceramic powder.

1. Introduction

Recently, as a result of the progression in technology productions, focuses have been increased on material technology science, in order to produce new suitable items for the harsh working industry. In factories, manufacturing techniques, materials with low specific weight are the most preferable for using

them in the field of defense, aerospace vehicles, rockets, building constructions, machine components, heat exchangers areas, sports and recreation. So, materials with light weight and stiffness have gotten more attention in order to increase their strength, stiffness, creep, wear resistance and a great interest was moved onto the substitution of iron-based materials by aluminum or other light metals like magnesium and titanium [1]. Improving pure aluminum metals like alloys and metal matrix composites (MMCs), are widely used in structures, non-structures, transportations and engineering fields where it is preferred to be used in these sectors due to their high performances, low cost and environmental benefits. Aluminum composites having the advantage of low fuel consumption and noise, due to their stiffness, flexibility, formability, corrosion resistance and low density, also they were used in military, aerospace and automotive components [2]. The physical and mechanical properties of composites will be an outcome and a direct result of reinforcement types. A wide range of reinforcement materials in different sizes, phases and shapes have been studied in aluminum matrix to fabricate a new AMMCs to improve the mechanical properties. Particle size and the amount of reinforcement have a pronounced effect on the mechanical properties of the metal matrix composites. Hardness, compression strength and wear resistance, of the composite is improved by proper addition of the reinforcement. Many other mechanical properties could be changed as a result of ceramic additives like the elongation of the composite may be reduced [3]. Also, reducing the particle size of the hard phase, greatly improves the strength which in turn depends on the manipulation method [4]. Particulate nanocomposites with the reinforcing particle sizes (from 10 to 100 nm) have obtained more attention because of their uniformly distributed particles, physical isotropic properties, and superior mechanical strength and ductility. Extensive work has been carried out for evaluating different nano particles as reinforcing materials such as conventional carbides SiC, B₄C, TiC, oxides Al₂O₃ [5], nitrides AlN, metallic glasses, inter metallic MoSi₂, and carbon nano tubes. In regard to ceramic materials, Al₂O₃ is the most preferred one from the other ceramics because it has no chemical reaction with the matrix material during composite processing or sintering and usually does not produce new undesired phases [6]. Metal matrix composites could be manufactured by different processes, such as solid (powder metallurgy, extrusion, forging), liquid (casting, in situ processes, squeeze casting), or gaseous state (plasma spraying). One of the most recommended is the solid state named by powder metallurgy process, this technique can be performed under low temperature and no new phases produce between the matrix phase and reinforcement. Powder metallurgy methods are based on mixing of matrix powders with reinforcement elements, axial or uniaxial cold pressing, and then followed by sintering. The powder metallurgy process on account of its simplicity, is applied widely for production of composite materials [7]. When comparing the solid phase processing (PM) with the casting process, the first one will reveal better control on the volume fraction and microstructure of the produced composite. By powder metallurgy methods, composite materials reinforced by dispersion particles, platelets, non-continuous fibers and continuous fibers could be manufactured [7, 8]. Many studies have been performed in order to improve the main lack of aluminum metal which is the low wear resistance. With the manufacturing of the new composites by impeding of hard ceramic particles an improvement in AMMCs could be achieved [9]. Many studies have been done to improve the mechanical properties of aluminum composites with the above indicated methods and different ceramic additives such as SiC, Al₂O₃, etc were used, in mechanical Behavior of Al-Al₂O₃ MMC manufactured by powder metallurgy techniques; improvements have been obtained in composite compared to matrix aluminum from 64 to 100% under the tensile and compressive loads, also stress-strain data were found to fit accordingly with the power law and the work-hardening exponents [10]. The wear behavior of Al-Al₂O₃ nanocomposites prepared by mechanical alloying and hot pressing which contains various amounts of nano sized Al-Al₂O₃ particulates are increasing linearly with sliding distance under dry condition. As well as, during the dry sliding of composite on pin on disc examination, composites with higher percentages of Al₂O₃ particles revealed in low wear losses [11]. The wear regimes with transitions in Al₂O₃ particulate-reinforced aluminum alloys and they discovered that Al₂O₃ particles reinforcement serve to delay the transition to the severe wear rate regime [12]. Fabrication of TiN reinforced aluminum metal matrix composites through a powder of metallurgical route, discovered that the incorporation of TiN particles in the aluminum matrix improves the mechanical properties and wear resistance [13]. The effects of particles sizes of the properties of Al-Al₂O₃ composites made by powder metallurgy process revealed, decreasing the alumina particle size increases the hardness also in addition, increasing the amount of alumina will produce an increase in hardness. The finer particle size of alumina will give greater yield and compressive strength, the adding of fine alumina in composite will result in high elongation rate and the addition of Alumina will reduce the wear rate of the composite [8].

The aim of this study is to characterize the effects of alumina phases (micro and nano sized) by using three different weight percentages (5, 10, and 15wt.%) in order to obtain new mechanical properties. A study of the composites wear resistance, hardness and compressive strength in each one produced, aluminum metal matrix composites as well as comparing the rate of improvement according to weight percentages, particle sizes and the phase type used.

2. Experimental procedures

2.1 Raw materials

The matrix is aluminum metal with (99.9% purity with particle size of 45 μm) the reinforcements are first micro Alpha- Al_2O_3 , 30 μm particle size, 3.97 g/cm^3 density and 99.9% purity). Then, the second nano Gamma- Al_2O_3 of 20 nm particle size, specific surface area of 230-400 m^2/g , density of 3.97 g/cm^3 , with 99.9% purity).

2.2 Mixing and compacting

Aluminum powder mixed with respectively, micro and nano alumina powder according to the weight percentages of 5, 10 and 15wt %. The powders were kept in a glass container in order to prevent any chemical reaction that might happen, and then mixed by using a roller mixer apparatus. PCA of (2 vol. % of acetone) has been added to the mixture in order to reduce segregation of alumina and prevent oxidation of aluminum during the mixing process [19], a rotational speed of the roller mixer was set to be 75 r.p.m and for six hours without interruption [3]. After distributing alumina particles in the aluminum matrix, the mixed powders were poured in a dye made of stainless steel metal with an internal diameter of 11mm, external diameter 16 mm and 70 mm length. Then, after filling the dye with the mixed powder according to the specimens dimensions, the powder was consolidated with reasonable selected compacting pressure of (500 MPa), the average rate of increasing the subjected load was 4.00 KN/second in order to provide enough time for the trapped air to release, to prevent residual stress concentration in the specimen and to obtain a green sample with less porosity and more density. The pressure was kept applying on the punch for 2 minutes for preventing the particles instantly springing back during the withdrawing process of the upper punch from the dye [3]. After, producing the green compacted sample, specimens have been interred to the pressure less vacuumed tube electric furnace in order to perform the sintering process. Vacuumed was 3×10^{-6} bar before and during the sintering process. The sintering average rate of heat increase was $20^\circ\text{C}/\text{min}$ and is maintained until it reaches the sintering temperature of 500°C , the holding time was 2 hours.

2.3 Compressive testing

The compressive tests were conducted at a room temperature on samples with a length to diameter ratio of 1.5 (11mm diameter & 16.5 length), the selected dimensions and testing procedure was according to ASTM E-9 standard. The subjected strain rate 4×10^{-3} (m/m.min)[14]. The specimen was compressed between the two jaws until splitting. The capacity of the device samples have been tested under 50KN load.

2.4 Microhardness testing

Microhardness tests can provide good indications about the hardness for the materials produced with powder metallurgy process and having porosity ASTM B933[15]. Vickers micro hardness test has been performed with 1.96 N load, and carried out on samples for 20 seconds.

2.5 Wear testing

The wear tests were conducted with a pin on disc apparatus in order to study the wear with very high precision and according to the ASTM G99 [16, 17]. The specimens with 11mm diameter and 16.5mm length have been loaded with weights of (500,1000 and 1500 grams) and were held with its axis perpendicular to the surface of a rotating steel disk in a dry friction condition. The speed of the steel rotating disc was 500 r.p.m. The specimens were located at 9 cm radial distance from the rotating axis. Four digits (0.0001g) accuracy balance (MonoBloc Model B303) used for calculating the weight loss in composite after sliding on the disc. Calculating the wear was based on the mass loss during and after the test. The mass loss of the sample was measured after each 2 minutes interval of sliding distance [16-18].

3. Results and discussion

3.1 Compressive testing

The compression test is useful to understand the mechanical behavior of the composite materials. Bonds between particles will delay the cracks initiation at the time of barreling. Figure 1 presents the ultimate compression strength (UCS) of micro and nano alumina composites which have been tested and compressed until failure.

It could be recognized from Figure 1 that at 5wt %, 10wt % and 15wt % for micro and nano alumina reinforcements, composites will bear higher force than the pure aluminum before failure initiation. This increase in ultimate strength is caused by alumina particles which are working as a hinder to prevent the movement of dislocation in the aluminum matrix via the dispersion strengthening mechanism. Upon increasing the additive from 5wt % to 10wt %, distances between alumina particles will decrease accordingly. So, higher force will be required for dislocation movements to progress among the reinforcing particles that will be required, then, an increase in compressive strength will be obtained.

Figure 1 shows that the recorded amount of ultimate compressive strengths for micro composites are (178, 260 and 200 Mpa) according to the increase in the amount of alumina which are respectively (5, 10 and 15wt%). While, the ultimate compressive strengths for nano composites are (245, 390 and 284.8 Mpa) according to the respective amount of alumina additions of (5, 10 and 15wt%). It could be seen that the strength in micro and nano composites increase when the amount of alumina increases from 5 to 10wt %. While, it continually increases the amount of alumina additives from 10wt% to 15wt.%, ultimate compressive strength will reduce in both micro and nano composites.

In the micro composite, the reduction in strength could be attributed to the increase in the amount of porosity that comes with the increase in the amount of alumina. When exceeding 10wt%, more porosity will initiate and reach a point where this increase will reduce the strength of the produced composite.

In case of the nano composite, the reason of the strength reduction is the agglomeration imperfection of nano alumina which increases with the increase in the amount of nano alumina addition. So, when exceeding the 10wt % the effect of agglomeration increased and then, a reduction in strength of the nano composite will be obtained which could be clearly seen in Figure 1.

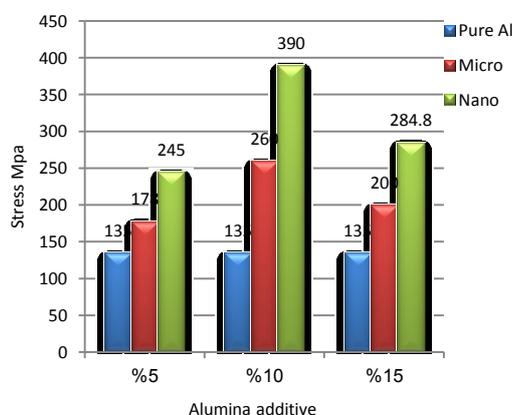


Figure 1. Compression test for pure aluminum, micro composites and nano composites.

When comparing the compressive strength of the nano composite with the micro composite at three different amounts (5, 10 and 15wt.%), it could be seen that the nano composite strength is higher than the micro composite strength. This gives an indication that UCS is in inverse relation to the particles size. These phenomena can be attributed to the criteria of decreasing the particle size which will minimize the distances between the reinforcement particles, and is shown in equation (1). Smaller particle sizes of additives will form smaller grain sizes in the composite microstructure.

$$\lambda = 4(1-f)r/3f \quad (1)$$

Where (assuming the particles as spherical); λ = the distance between the reinforcement particles, f = particles volume fraction, r = radius of particles.

A Hall-patch equation (2) shows the relation of the grain size with the flow stress at a certain plastic strain up to the ductile fracture. According to the last equation it could be concluded that as the grain size

gets smaller, the flow stresses will increase and then produces high resistance material. Figure 1 shows that the maximum amount of UCS in nano composite is 390MPa, while 260 MPa for the micro composite, the main reason of this discrepancy is due to the very high difference in their particle size. Strength of material is usually calculated from equation (2) [19]:

$$\sigma^o = \sigma_i + kD^{-1/2} \quad (2)$$

where: σ^o = flow stress, σ_i = stress opposing the movement of dislocation, k = constant, and D = grain size of material.

This improvements in compressive strength of the composites in compare with aluminum matrix are coming from the change in materials mechanical properties which caused by the amount and phases of alumina addition.

3.2 Micro hardness testing

Figure 2 illustrates the effect of the amount of reinforcement alumina on the microhardness in the produced micro and nanocomposites. Because of alumina hardness is greater than the hardness of pure aluminum matrix, therefore when the amount of reinforcement increases, micro hardness of the produced composite will increases too. The hardness of the produced composite accordingly applies with the hardness rule of mixed materials which is shown in equation (3) [8],

$$H_c = H_m F_m + H_r F_r \quad (3)$$

Where: H_c = Hardness of the produced composite, H_m = Hardness of the matrix material, H_r = Hardness of the reinforcing material, F_m = volume fraction of the matrix material, and F_r = volume fraction of the reinforcement material.

Figure 2 shows that microhardness improves with the increase in the percentage of alumina additive (5, 10 and 15wt %) in cases of micro and nano composites. These reinforcements are applicable with equation (3). Particles of alumina have higher hardness than the pure aluminum then eventually; the addition of reinforcement particles will prevent the lattice movement and reinforce aluminum structure.

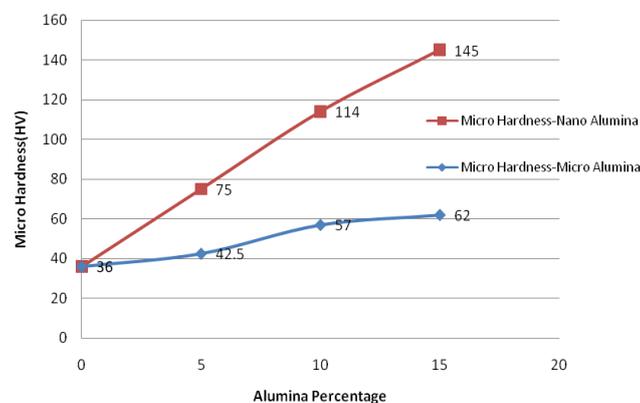


Figure 2. Microhardness results for samples in case of micro composites and nano composites.

Figure 2 shows that the amount of microhardness in a nano-composite is higher than the micro-composite. Two perspectives may explain this improvement; the first is due to the defects in coarse-grained micro particles which have higher chance to occur. The second is, because of the higher interfacial area between the hard nano alumina particles with the soft micro aluminum matrix which will definitely increase the microhardness of the nano-composite.

At the micro-composites, when increasing the amount of micro reinforcement from 10wt % to 15wt %, the improvement in micro-hardness is not relatively high in comparison with the increase from 5wt.% to 10wt.%, this could be attributed to the effect of porosity increases that accompanies the increase in the amount of micro alumina Figure 2 while the improvement is continually taking place in case of nano reinforcement.

3.3 Sliding wear testing

Figures (3 to 8) show the wear mass loss as a function of a sliding distance in the produced micro and nano composites subjected to three different loads (500,1000 and 1500g) in compare with the pure aluminum. An overall indication is obtained, the wear in micro and nano composites were less than the wear in pure aluminum matrix. Rate of mass loss is directly proportional to the sliding distance and applied load. Wear rate is relatively different from one composite to another according to the amount and phase of alumina.

In this study, the maximum weight loss in the fabricated samples were observed with the micro composite of 5wt % under 1500g were the applied load, while the minimum wear were obtained at a composite with 15wt % nano alumina reinforcement subjected to 500g load.

Main reason of the reduction in wear losses 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 micro hardness tests and the rule of mixtures, the composite hardness is increases with the increase in the amount of alumina and a decrease in particle size.

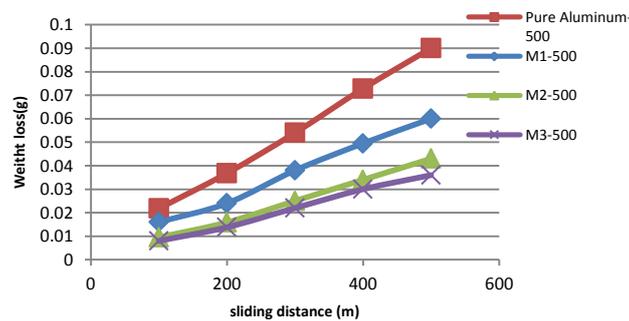


Figure 3. Weight loss for samples with micro alumina additives subjected to 500g load.

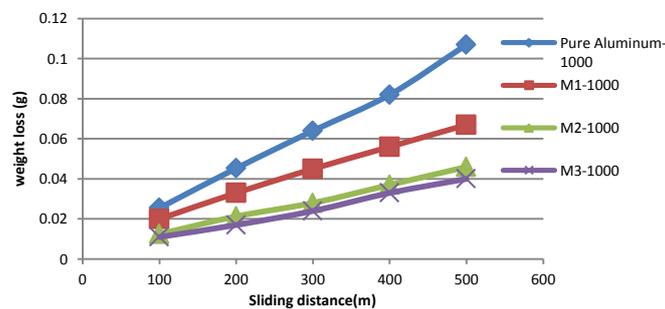


Figure 4. Weight loss for samples with micro alumina additives subjected to 1000g load.

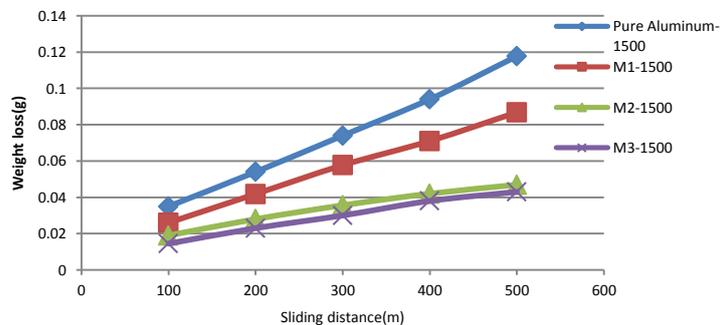


Figure 5. Weight loss for samples with micro alumina additives subjected to 1500g load.

Figures (3 to 5) shows the development in wear resistance of the micro composite against the sliding distance and applied loads. It could be seen that within the change in amount of alumina from 5wt % to 10wt % the improvement in wear resistance is higher than wear resistance of the changes from 10 wt % to 15wt %. The perspective of this phenomenon can be related to the low improvement in hardness as

shown in Figure 2, which comes from the increase in porosity after exceeding 10wt % of alumina addition.

For the case of nano composites shown in Figures (6 to 8) it could be noticed that as the nano amount of particles increase gradually from 5wt %, 10wt % to 15wt %, improvements were continually obtained in the wear resistance and works perfectly under the increase in load. This might come from two reasons, the first one is because of the reasonable increase in the relative density of nano-composites, and the second reason is, due to the increase in hardness.

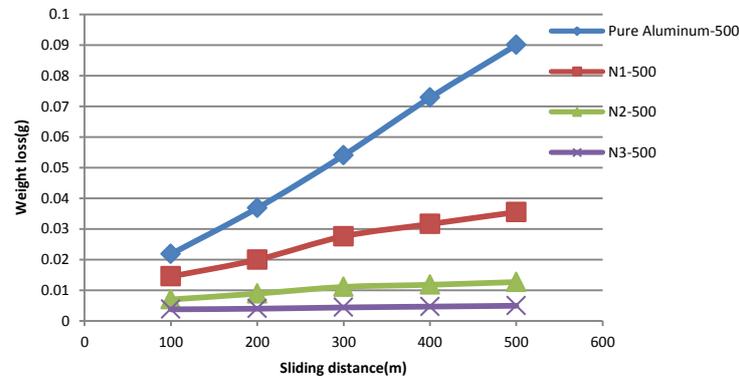


Figure 6. Weight loss for samples with Nano-alumina additives subjected to 500g load.

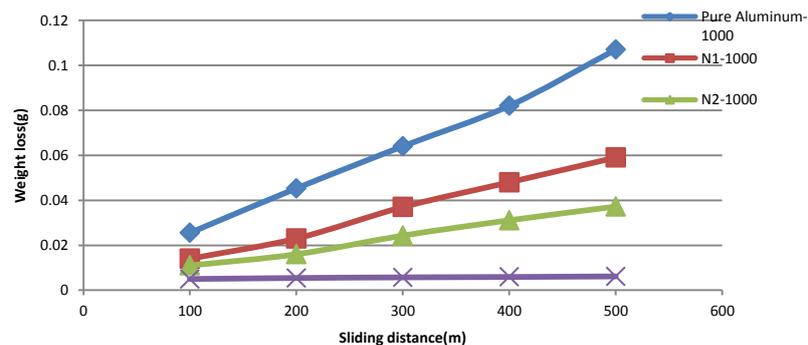


Figure 7. Weight loss for samples with Nano-alumina additives subjected to 1000g load.

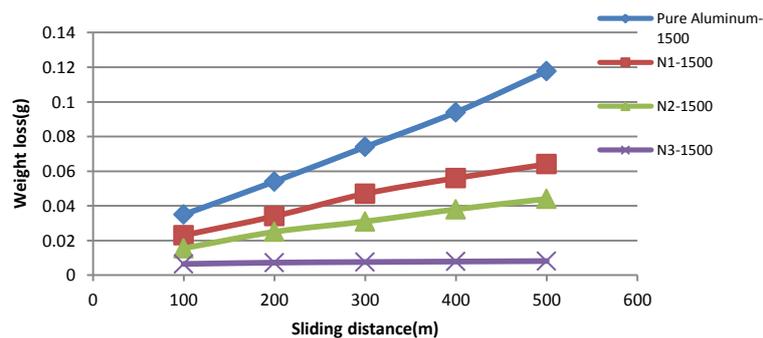


Figure 8. Weight loss for samples with Nano-alumina additives subjected to 1500g load.

Figures 9 to 17 show a comparison between each of the nano and micro-composites mass loss under the same subjected loads. In all studied cases, nano composites reveals lower mass loss than micro composite, the reason is due to the increase in hardness and density.

Also, it could be seen from Figures (9 to 17) that the less weight loss that is being obtained at 15wt % of nano-composites under the three subjected loads, the reason is due to higher observed hardness within this percentage of alumina additive. It could be recognized from Figures (15 to 17) that the weight losses in nano composite of 15wt % is approximately neglected after exceeding the sliding distance of 100m

rather than the other composites, this is due to the high hardness and density at 15wt % the nano-composite has.

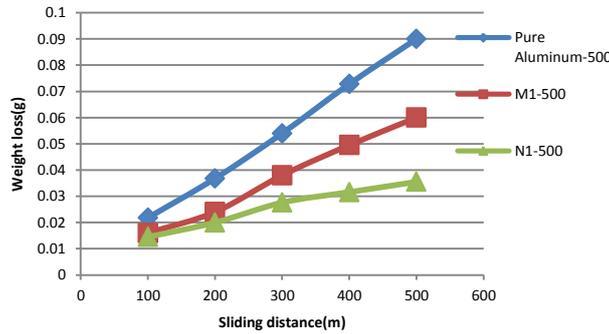


Figure 9. Weight loss for samples subjected to 500g load, 5wt % micro-alumina M1, 5wt % Nano-alumina N1 and pure aluminum.

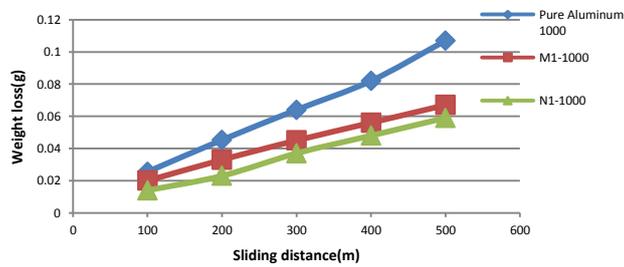


Figure 10. Weight loss for samples subjected to 1000g load, 5wt % micro-alumina M1, 5wt% Nano-alumina N1 and pure aluminum.

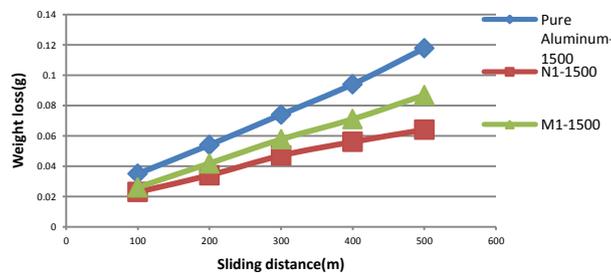


Figure 11. Weight loss for samples subjected to 1500g load, 5wt % micro-alumina M1, 5wt % Nano-alumina N1 and pure aluminum.

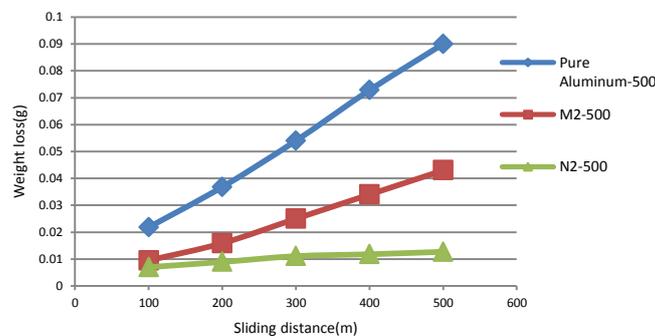


Figure 12. Weight loss for samples subjected to 500g load, 10wt % micro-alumina M2, 10wt% Nano-alumina N2 and pure aluminum.

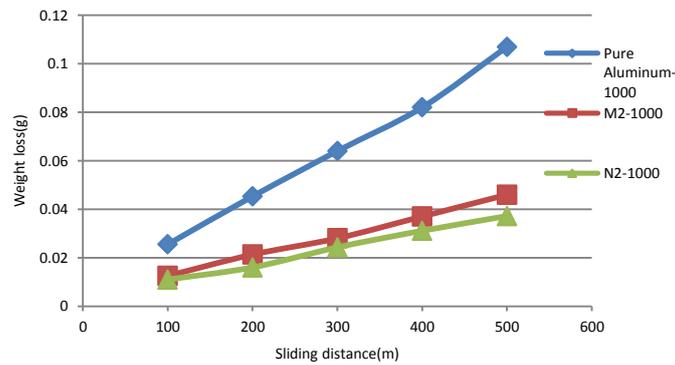


Figure 13. Weight loss for samples subjected to 1000g load, 10wt % micro-alumina M2, 10wt % Nano-alumina N2 and pure aluminum.

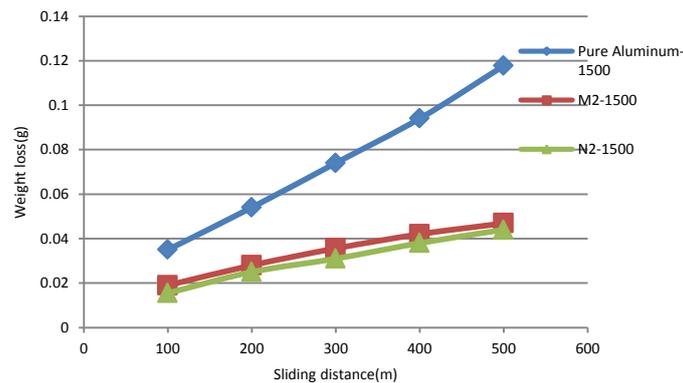


Figure 14. Weight loss for samples subjected to 1500g load, 10wt % micro-alumina M2, 10wt % Nano-alumina N2 and pure aluminum.

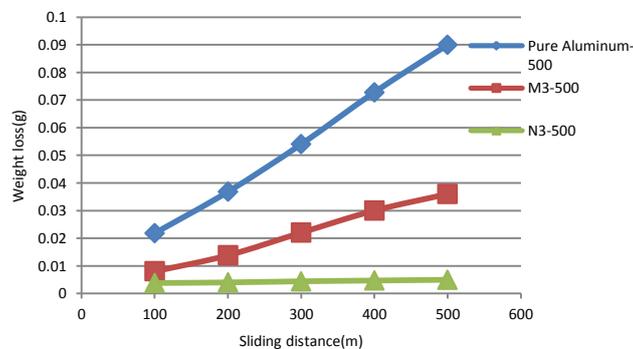


Figure 15. Weight loss for samples subjected to 500g load, 15wt % micro-alumina M3, 15wt% Nano-alumina N3 and pure aluminum.

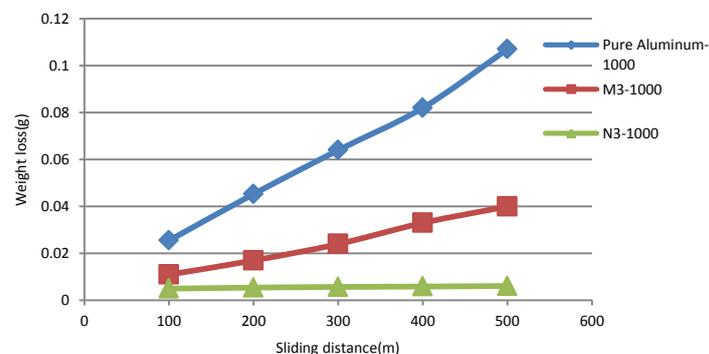


Figure 16. Weight loss for samples subjected to 1000g load, 15wt % micro-alumina M3, 15wt % Nano-alumina N3 and pure aluminum.

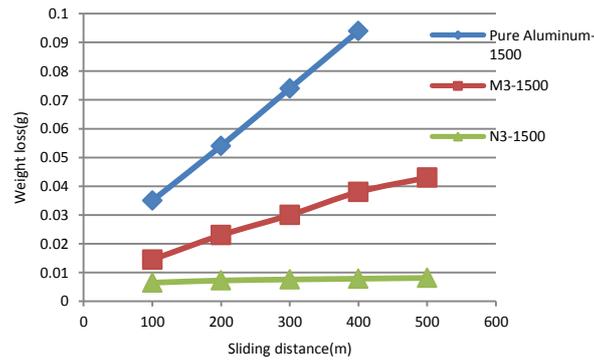


Figure 17. Weight loss for samples subjected to 1500g load, 15wt % micro-alumina M3, 15wt % Nano-alumina N3 and pure aluminum.

The Figures from (18 to 22) present the wear rate variation according to the amount of alumina changes for samples of micro and nano phases, composites subjected to 500g load and for a sequenced distances of 100, 200, 300, 400 and 500m. For all distances and the amount of alumina added, the wear rate is lower in the nano composites than in the micro composites in a fixed load and distance. Results indicated are in line with other scholars [20-22]. Wear increases within the increase in distance and inversely proportion to the amount of alumina additives.

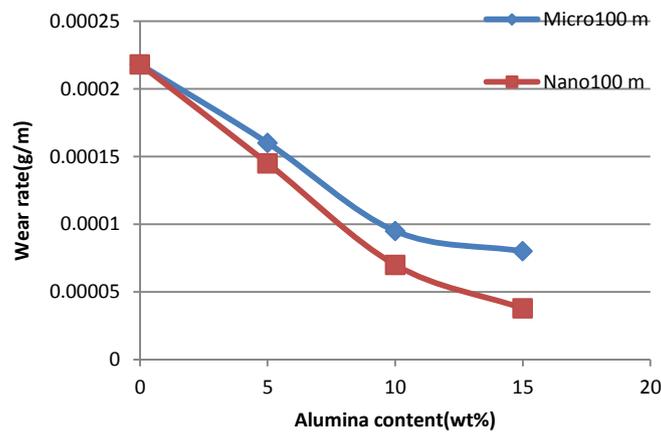


Figure 18. Wear rate at 100m sliding distance for different alumina percentage of micro composite and nano composite subjected to 500g load.

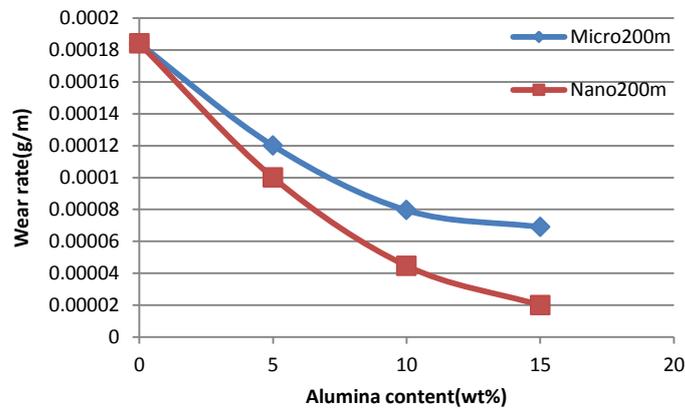


Figure 19. Wear rate at 200m sliding distance for different alumina percentage of micro composite and nano composite subjected to 500g load.

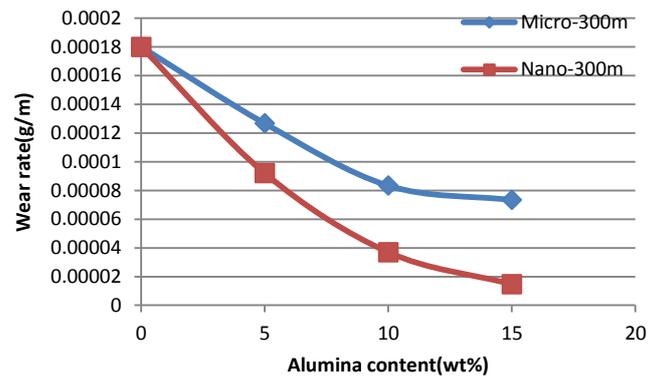


Figure 20. Wear rate at 300m sliding distance for different alumina percentage of micro composite and nano composite subjected to 500g load.

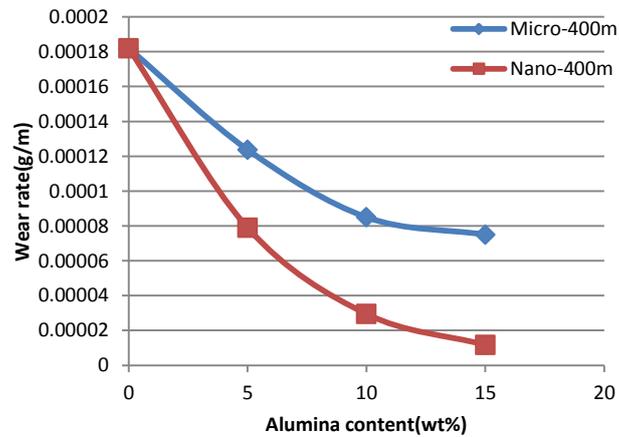


Figure 21. Wear rate at 400m sliding distance for different alumina percentage of micro composite and nano composite subjected to 500g load.

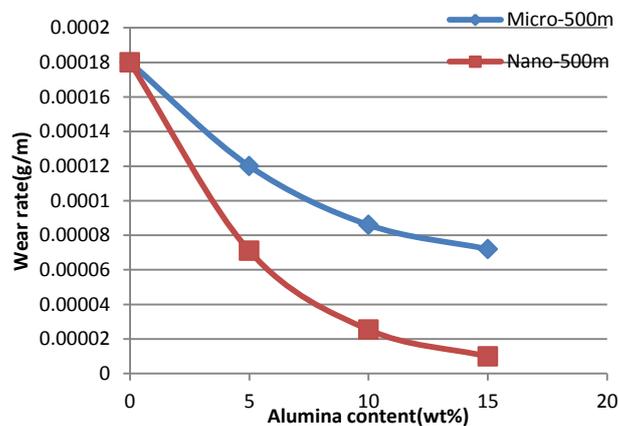


Figure 22. Wear rate at 500m sliding distance for different alumina percentage of micro composite and nano composite subjected to 500g load.

4. Conclusions

Addition of identified alumina reinforcements have reinforced the aluminum matrix. The produced composites are more flexible to use at a harsh engineering environment. The main drawback of pure aluminum metal low wear resistance can be overcome by fabricating new composites with a specified amount of alumina. In this research many conclusions and outcomes have been obtained from the results which revealed the following:

1. Compacting stress at 500Mpa for two minutes, sintering temperature at 500°C for two hours is sufficient enough to create a new composite; Mixing time for six hours in horizontal roller mixer which produces higher strength than the pure aluminum;

2. For nano and micro composites, when increasing the amount of alumina reinforcement up to 10wt %, compressive strength will increase, while increasing the amount of alumina from 10wt % to 15wt %, compressive strength will reduce. Nano composites exhibit higher ultimate compressive strength which is (390MPa) with 10wt % nano Al₂O₃. While, ultimate compressive strength was (260MPa) at the same amount of micro alumina (10wt %).
3. Hardness tests have shown that nano alumina composites are displaying higher hardness and strength comparisons with micro alumina composites.
4. Addition of alumina powder will reduce the weight loss and increase the wear resistance of the composite. Composites with nano alumina additives exhibiting a greater wear resistance than the one with micro alumina additives. At an average sliding distance of 300m and under a subjected load of 500g, the wear rate of nano composite with 5wt % was (0.000092g/m), while at the same percentage, environment, sliding distance, and loading the micro composite reveals less wear resistance which is (0.000126667 g/m).
5. Eventually, the combination between alumina and aluminum can produce an excellent balance between materials costs and mechanical properties.

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