

EFFECT OF Al₂O₃ NANOPARTICLES ON CORROSION BEHAVIOR OF ALUMINUM ALLOY (Al-4.5 wt% Cu-1.5 wt% Mg) FABRICATED BY POWDER METALLURGY

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Received 6 November 2018; accepted 23 November 2018

Abstract. In this research the effect of Al_2O_3 nanoparticles on corrosion behavior of aluminum base alloy (Al-4.5 wt% Cu-1.5 wt% Mg) has been investigated. Nanocomopsites reinforced with variable contents of 1, 3 and 5 wt% of Al_2O_3 nanoparticles were fabricated using powder metallurgy. All samples were prepared from the base alloy powders under the best powder metallurgy processing conditions of 6 hr of mixing time, 450 MPa of compaction pressure and 560 °C of sintering temperature. Density and micro hardness measurements, and electrochemical corrosion tests are performed for all prepared samples in 3.5 wt% NaCl solution at room temperature using potentiostate instrument. It has been found that density and micro hardness of the nanocomposite increase with increasing of wt% Al_2O_3 nanoparticles to Al matrix. It was found from Tafel extrapolation method that corrosion tests by potentiodynamic cyclic polarization method, it was found the pitting corrosion resistance improves with adding of Al_2O_3 nanoparticles. It was noticed that the pits disappear and the hysteresis loop disappears also from anodic polarization curve.

Keywords: powder metallurgy, nano composites, Al-Cu-Mg alloy, electrochemical corrosion.

Introduction

Aluminum matrix composites (AMCs) have been widely studied due to their low density, their good physical and mechanical properties, their good corrosion resistance, high thermal and electrical conductivity (Khichadi, Lande, & Pathan, 2016). The addition of reinforcements into the metallic matrix improves the stiffness, specific strength, wear, creep and fatigue properties compared with the conventional engineering materials (Vijaya Ramnath et al., 2014). Aluminum oxide (alumina, Al₂O₃) is currently one of the most useful oxide ceramics, as it has been used in many fields of engineering such as coatings, heat-resistant materials, abrasive grains, cutting materials and advanced ceramics. This is because alumina is hard, highly resistant towards bases and acids, allows very high temperature applications and has excellent wear resistance (Tok, Boey, & Zhao, 2006). Nanotechnology has become a key area in the development of science and engineering (Pathak, Singh, Das, Verma, & Ramachandrarao, 2002). Nanotechnology basically involves the production or application of materials that have unit sizes of about 10-100 nm. Comparing micron-sized and nano-sized alumina particles, na-

no-alumina has many advantages. A smaller particle size would provide a much larger surface area for molecular collisions and therefore increase the rate of reaction, making it a better catalyst and reactant (Wu, Zhang, Huang, & Guo, 2001). There are many ways to prepare nanocomposites including squeeze casting, vacuum infiltration or pressure infiltration, stir casting, permanent mold casting or high pressure die casting and powder metallurgy processes. The best method to prepare the nanocomposite material is powder metallurgy (PM) processes due to PM parts that can be mass produced to net shapes or near net shapes, eliminating or reducing the need for subsequent machining operation, wastes very little material - about 97% of the starting powders are converted to product, can be made with a specified level of porosity, to produce porous metal part, better control of compaction and structure and production of impossible parts (Groover, 2010).

Many corrosion studies conducted of aluminum matrix composites reinforced with micron size particles such as Al_2O_3 , SiO_2 , SiC...etc. Faris, Waheed, and Abbass (2010), Abd Alameer (2011) have been focused on the ef-

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This is an Open Access article distributed under the terms of the Creative Commons Attribution License (http://creativecommons.org/licenses/by/4.0/), which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited. fect of weight percentages of Al₂O₃ particles on the corrosion behavior and susceptibility in NaCl solution.

Alaneme and Bodunrin (2011) investigated the effect of various volume percentages of Al_2O_3 particles (9, 15, 18% vol.) and solution heat treatment on corrosion behavior of Al 6063 matrix composites in salt water, NaOH and H_2SO_4 media. They concluded that solution heat treatment improved the corrosion resistance for base alloy and composites which exhibited excellent corrosion resistance in NaCl medium than in the NaOH and H_2SO_4 media.

Majed, Mahdi, Al-Kaisy, and Abdul Maged (2014) studied the effect of adding SiO_2 particles on corrosion behavior of Al-Cu-Mg in 3.5% NaCl solution. Cyclic polarization can be the stability of passive layer for composites. Zakaria (2014) studied the effect of size and volume fraction of SiC particles on corrosion behavior of pure aluminum matrix composites in 3.5% NaCl solution and found that the corrosion rate increased with increasing the duration exposure. Abbass, Hassan, and Alwan (2015) found that adding SiC particles in 10% and 20% to Al6061 alloy matrix increased the corrosion rate in 3.5% NaCl solution.

But the effect of nanoparticles Al_2O_3 on the corrosion behavior and properties of aluminum matrix composites is not studied in details till now or very limited researches. The aim of this research is to study the effect of various percentages of Al_2O_3 nanoparticles on corrosion behavior of aluminum alloy (Al-4.5% Cu-1.5% Mg) fabricated by powder metallurgy.

1. Experimental work

1.1. Material used

The materials used for preparation of compact (Al-4.5% Cu-1.5% Mg) alloy are Al powder, Cu powder, Mg powder and types of nano particles of Al_2O_3 . The materials properties used are listed in Table 1.

Powder	Purity, %	Average particle size (µm)
Aluminum	99.98	34.56
Copper	98.50	21.28
Magnesium	99.98	44.04
α-Alumina	99.98	0.05

Table 1. The materials properties used as different powders

1.2. Sample preparation

Mixing is the most common pre–compaction step in powder metallurgy. Theoretically any composition can be prepared starting from elemental powders. In this experiment 9.4 g of aluminum powder is mixed with 0.45 g of copper and 0.15 gm magnesium powder with nano particles Al_2O_3 with particle size of 0.05 µm (50 nm) in different percentages of (1 wt%, 3 wt% and 5 wt%) with addition of zinc stearate of (0.5%) as a binder and a lubricant mixture. Mixing was carried out in a ball mill type (Jar rotating by motor) which have alumina balls. The number of balls are 20 balls with ratio 20:1 weight of powder to weight of balls for mixing time of 6 hours at speed of 650 rpm in order to get a homogeneous mixture. The homogeneous mixing will improve the sintering ability, ejection of compact and strength of the compact. Uniaxial cold compaction process was carried out on mixed powders to obtain good compaction and to produce the green billet with few porosity. The powders were pressed at (450 MPa) for all samples used in this work. A cylindrical one direction action die with 20 mm hole diameter and (20 ton) capacity of electric hydraulic press, for, incubation time (15 min) in each applied pressure was used. Sintering process was carried out in electric tube furnace type (MITI CORPO-RATION GSL) with maximum temperature of (1600 °C) with using argon of purity 99.99%. A sintering process was carried out in sintering furnace with Ar flow rate 2 L/min and at temperatures of (560) °C for one hour. The prepared sintered sample was analyzed by X-ray fluorescent (XRF) instrument type (SPECTRO X-LAP) which operated at 45 KV, 0.5 mA and energy range 25 KV. Table 2 shows the chemical composition of sintered base alloy.

Table 2. Chemical composition of sintered base alloy by XRF instrument

Element wt%	Cu	Mg	Si	Mn	Al
value	4.5	1.5	0.2	0.1	balance

2. Tests and inspections

2.1. Density and porosity measurements

The final true density and porosity for all samples were measured according to ASTM D 792-ISO 1183 standard which is based on Archimedes principle. The specific gravity of material is given by equation (1) and the porosity is given by equation (2) (Intertek Group plc., n.d.). This process was carried out at room temperature, and the auxiliary liquid used was water (density = 1 g/cm^3).

$$Sp._{Gr \text{ for sample}} = \frac{W1}{W1 - W2},$$
(1)

where: P =
$$\frac{W3 - W1}{W3 - W2}$$
, (2)

where: P – porosity of sample; W1 – weight of sample in air (g); W2 – weight of sample suspended in liquid (g); W3 – weight of wet sample i.e. weight of soaked sample air (g); Sp._{Gr for sample} – specific gravity of material.

2.2. Microstructure and microhardness

The samples were ground and then polished to a scratch free surface. Micro hardness of the specimen was measured by Vickers hardness tester (Leco Micro hardness Tester LM248AT), at an applied load of 1.96 N for an indentation period of 15 seconds. The readings were recorded here at five equivalent locations for each specimen to evaluate micro hardness average value.

2.3. Electrochemical corrosion test

In this work, to evaluate the corrosion parameters of the samples of (Al-Cu-Mg) base alloy and nanocomposities reinforced with nanoparticles Al₂O₃ in 3.5% NaCl solution at room temperature. The sample with surface area of (1 cm²) put in electrochemical cell of 1000 ml capacity which consists the three electrodes. The working electrode (W.E) is sample of the corroding metal with (1 cm^2) exposed to solution. The counter or auxiliary electrode (A.E) is generally an inert conductor, platinum rod, with (100×10) mm dimensions. The reference electrode or standard hydrogen electrode (S.H.E) is used in measuring the working electrode potential. The range of the cyclic polarization test was (-250), (+1000 mV, +1500) below and above the open circuit potential respectively. During cyclic polarization test scan rate was 5 mV/sec. The electrochemical corrosion tests by Tafel extrapolation method and cyclic polarization tests were carried out by using a WENKING MLabmulti channels and SCF-MLab. Corrosion measuring system from Bank Electronics-Intelligent controls GmbH, Germany 2007. The electrochemical cell is shown in Figure 1. Corrosion parameters are determine such as Ecorr and Icorr in order to calculate the corrosion rate (C.R) from equation as follows (Hintze & Calle, 2006).

$$CR(mpy) = 0.13 \times Icorr \times \left(\frac{EW}{\rho}\right)$$

where: CR(mpy) – corrosion rate in (mils per year); *Icorr* – corrosion current density in (μ A/cm²); *EW* – equivalent weight in (grams/equivalent); ρ – density of the corroding specimen (g/cm³).



Figure 1. Electrochemical corrosion cell used in this study

3. Results and discussion

3.1. AFM images and XRD results

The results of average particles size measurements for nano powders of Al_2O_3 after preparation one drop was put on glass slide for single nano alumina. A 50 nm of nano powder Al_2O_3 is used as shown in Figure 2 which indicates the topography, distribution of particles. The images given by AFM microscope are like topographical maps. The color of the image represents the height of the material. The lighter parts of the image are higher.





(Al-4.5% Cu-1.5% Mg) sample

Figure 3 shows XRD analysis results of sintered (Al-4.5% Cu-1.5% Mg) sample at (560 °C). In comparison to the diffraction peaks from Al powder, Cu powder and Mg powder peaks of phases (Al₂CuMg), (Mg₂Cu6Al₅) have low intensities in the diffraction pattern, which can be attributed to the low weight percentages of these phases in base alloy. These results are confirmed the diffusion of alloying elements Cu and Mg in Al matrix during sintering process and formation of Intermetallic compounds as strengthening phases. These results are matched with researchers Abbass and Sultan (2017).

3.2. Density and porosity results

The density and porosity measurement results after sintering at the temperature 560 °C for 60 min for base alloy and composites reinforced with Al_2O_3 nanoparticles are shown in Table 3.

It was seen that the density increases with an increase in the nano Al₂O₃ powder addition and porosities% decreases as compared to the base alloy. It was also shown from Table 3 that the density increases from (2.63 to 2.68) g/cm³ with addition of 5% Al₂O₃ nanoparticles while the average porosity% reaches to 12.1%. This value is within standard value (10-20%) (Groover, 2012). The density with fewer pores leads to increasing the particles bonding between micro particles of (Al-4.5% Cu-1.5% Mg) alloy and Nano Al₂O₃ particles. The mismatch in the thermal expansion coefficient between matrix of (Al-4.5% Cu-1.5% Mg) alloy and nano Al2O3 could produce residual stresses around the dispersed nanoparticles which could result in micro cracks and reduction in the mechanical properties. Moreover, it is assumed that nano Al₂O₃ existing in the grain boundaries act as barriers to prevent closing up of the grains. It has been found that nanocomposities with 5% Al₂O₃ exhibited higher densities than the base alloy. These results are in agreement with researcher Cooke, Hexemer, Donaldson, and Bishop (2012). The increase in density is connected with decrease in porosity for nanocomposities and the pores will close when increasing the addition nanoparticles. These results are in coincidence with reference (Abbass & Fouad, 2014). The addition of nanoparticles act as micro-filling and enhancement in pore filling that nano Al₂O₃ fill the voids of Al-Cu-Mg structure. The structure become denser and compact and cause densification effect which improve the microstructure of sintered alloy.

3.3. Vickers micro hardness measurements

Table 3 indicates micro hardness values for all samples at the sintering temperature (560 °C) for 60 min. It was shown that the (MMNCs) samples have significantly higher micro hardness than that of base alloy (Al-4.5 wt% Cu-1.5 wt% Mg). The hardness increases with increasing of nano Al_2O_3 powders in Al-matrix. This is due to high micro hardness of nano alumina. It was seen that the (MMNCs) sample with addition of 5 wt% Al_2O_3 has the highest micro hardness as compared with other percentages (1 wt%, 3 wt% Al_2O_3). This is due to good densification with highly density and reducing porosity. Also the nano Al_2O_3 act as a filling agent which has high diffusivity and ability to enter the inter spacing of crystal structure of aluminum phase and close the micro porosity in matrix alloy. The improvement in hardness was 38.6% for 5 wt% Al_2O_3 addition. Many researchers confirm these results in their works, as in Haleem, Zuheir, and Dawood (2012), Abbass and Fouad (2015).

3.4. Corrosion results

3.4.1. Open circuit potential-time measurements

The addition of 1 wt% nano Al₂O₃ to base alloy made the potential began at (-790.6 mV), and reached to stable value at (-790.6 mV) for composite reinforced with 1% Al_2O_3 . While the presence of 3 wt% Al_2O_3 in matrix alloy made the potential reached to (-760 mV). The addition of 5 wt% Al₂O₃ to base alloy lead to shift Eocp to (-551.2 mV) to less negative values than the base alloy (-630 mV), as shown in Figure 4 and Table 4. In the present work, the presence of Cu, Mg in Al- α phase of alloy and also intermetallic phases Al₂CuMg, Mg₂Cu6Al₅ (as indicated by XRD) act as cathodic sites which encourage the dissolution of protective oxide film and enhance the pitting corrosion. These results are confirmed by researcher Blanc, Freulon, Lafont, Kihn, and Mankowski (2006). They indicate that reactively of Cu, Mg Intermetallic compounds play in homogenous dissolution of these compounds and copper redeposition followed by local dissolution of the surrounding Al matrix according to the following equations:

$$Al \to Al^{+3} + 3e^{-}; \tag{1}$$

$$Mg \rightarrow Mg^{+2} + 2e^{-}; \tag{2}$$

$$Al_2Cu \rightarrow Cu^{\bullet} + 2Al^{+3} +; \tag{3}$$

$$Al_2Cu \rightarrow 2Al^{+3} + Cu^{+2} + 8e$$
, and $Cu^{+2} + 2e \rightarrow Cu^{\bullet}$. (4)

3.4.2. Cyclic polarization of composites reinforced with nano Al₂O₃

Table 4 shows the corrosion parameters results of composites reinforced with different wt% of nano Al_2O_3 . It was seen that the improvement in corrosion resistance was 81% for composite reinforced with 5 wt% Al_2O_3 . Figures 5, 6, 7 and 8 show the cyclic polarization curves for base alloy and composites reinforced with different wt% of nano Al_2O_3 respectively. The corrosion rate decreases with increasing Al_2O_3 nanoparticles whereas the corrosion resistance of composites reinforced with Al_2O_3 nanoparticles samples noticeable increases and become highest as compared with base alloy. It was noticed that the corrosion resistance of composite reinforced with 5% Al_2O_3 nanoparticles was higher than that of composites reinforced with (1 wt% and 3 wt%) nanoparticles.

Table 3. Density, porosity and microhardness of nanocomposities after sintering at 560 °C for 60 min.

Symbol	Sample	Density, g/cm ³	Porosity, %	HV (kg/mm ²)
А	Base alloy (Al-4.5 wt% Cu-1.5 wt% Mg)	2.63	16	89
B1	Base + 1 wt% Al_2O_3	2.64	15.3	114
B2	Base + 3 wt% Al_2O_3	2.65	14.6	132
B3	Base + 5 wt% Al_2O_3	2.68	12.1	145



Figure 4. Variation of potential – time of base alloy (Al-Cu-Mg) and composites reinforced with Al₂O₃ in seawater

Sample condition		Base alloy	1 wt% A1 ₂ O ₃	3 wt% A1 ₂ O ₃	5 wt% A1 ₂ O ₃
Parameter					
Eocp, mV		-630	-790.6	-760	-5512
Ecor, mV		-700.2	-825	-804.9	-5462
Icor, μA.cm ⁻²		177.18	146.18	106.29	33.50
Tafel slope, mV/dec	bc	-217.0	-94,9	-140.8	-179.0
	ba	80.5	158.6	106.4	73.6
C.R (mpy)		89.7	74.1	53.8	16.9
Corrosion improvement, %			17.4%	40%	81%

Table 4. Eocp values and corrosion parameters results of ba	ase
alloy and nanocomposites	





Figure 6. Cyclic polarization of composite reinforced with 1% Al₂O₃ nanoparticles



Figure 7. Cyclic polarization of composite reinforced with 3% Al₂O₃ nanoparticles



Figure 8. Cyclic polarization of composite reinforced with 5% Al₂O₃ nanoparticles



Figure 9. Micrographs image for (Al-4.5% Cu-1.5% Mg) base alloy before (a) and after corrosion (b)



Figure 10. Micrographs images for base alloy (a) and composite reinforced with 5% Al₂O₃ nanoparticles (b) after corrosion test

4. Micrographs characterization results after corrosion test

Figures 9 and 10 show the micrograph images of the sintered base alloy and composite reinforced with 5% Al_2O_3 before and after corrosion test respectively. The addition of Al_2O_3 nanoparticles reduce the number of pits and also size and shape of pit change. This is due to covering of pit by Al_2O_3 nano particle.

Conclusions

- 1. The density of nanocomposities increases with an increase of nanopowder addition to sintered base alloy (Al-4.5 wt% Cu-1.5 wt% Mg). The highest density value was with nanocomposite reinforced with 5% Al_2O_3 nanoparticales.
- 2. The nanocomposities reinforced with 5 wt% Al₂O₃ nanoparticales showed the highest micro hardness compared with other percentages.
- 3. Increasing the weight percentage of Al₂O₃ nanoparticles into the base alloy increases the pitting corrosion resistance.
- 4. The nanocomposites reinforced with 5 wt% Al₂O₃ nanoparticales have lowest corrosion rate compared with base alloy and other composites. Improvement in corrosion resistance was 81%.
- 5. From cyclic polarization results it was found the pitting corrosion resistance improved with adding nanoparticles. It was noticed that the pits disappear and hysteresis loop disappears also from anodic polarization curve for each nanocomposite.

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