Corrosion Behaviour on Powder Metallurgy Aluminium Matrix Composite Reinforced with Alumina Saffil™ Short Fibres

By

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List of abbreviations

Al	Aluminium
AlN	Aluminium nitride
Ag	Silver
Al_2O_3	Alumina
$Al\ Al_{1.67}O_4$	Aluminum oxide
$Al\ (OH)^3$	Aluminium hydroxide
AMC	Aluminium matrix composite
ASM	American Society of Materials
ASTM	American Society of Testing and Materials
Ca	Calcium
Cu	Copper
EDX	Energy dispersive X-ray spectroscopy
H_2O	Water
HV	Vickers hardness value
Ltd	Limited
Li	Lithium
Mg	Magnesium
Mn	Manganese
MMC	Metal matrix composite
Nd	Neodymium
NaCl	Sodium Chloride
P	Phosphorus
PM	Powder metallurgy
Sc	Scandium
SiN_4	Silicon nitride
Si_3Ni	Silicon nickel
SCE	Saturated colamel electrode
SEM	Scanning electron microscope
SiO_2	Silica
SiC	Silica carbide
TiH_2	Titanium hydride
TiC	Titanium carbide
TEM	Transmission electron micrograph
XRD	X-Ray Diffraction
WC	Tungsten carbide
Zr	Zirconium

List of symbols

A/cm^2	Ampere per centimetre square
wt%	Weight percent
ΔG	Change in free energy
E_{corr}	Corrosion potential
I_{corr}	Corrosion current density
mV	Micro Volt
MPa	Mega Pascal
R_p	Resistance polarization
Rpm	Rotation per minute
mg/cm^2	Milligram per centimetre square
µm/year	Micrometer per year

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1. Normah Cheman, Mohd Nazree Derman, Nur Maizatul Shima Adzli, Mohd Zaherudin Kasmuin, Shamsul Baharin Jamaludin and Noraziana Parimin (2007), *The effect of magnesium powders on PM Al-composites reinforced with short fibre alumina*, National Metallurgical Conference, 26th – 27th November 2007, Puteri Pacific Hotel, Johor.

2. Normah Cheman, Mohd Nazree Derman, Nur Maizatul Shima Adzli, Mohd Zaherudin Kasmuin, Shamsul Baharin Jamaludin and Noraziana Parimin (2007), *The fabrication and characterization of powder metallurgy aluminium – magnesium*, 6th Asean Microscopy Conference, 10th – 12th December 2007, Cherating, Pahang.

3. Normah Cheman, Mohd Nazree Derman, Nur Maizatul Shima Adzli, Shamsul Baharin Jamaludin (2008), *The fabrication and properties of PM Al-Mg composites reinforced with short fibre alumina,* Malaysian Universities Conferences Engineering and Technology, 15th - 17th March 2008, Putra Palace, Perlis.

4. Normah Cheman, Mohd Nazree Derman, Nur Maizatul Shima Adzli (2008), *Effects of alumina Saffil™ short fibres reinforcement on corrosion behaviour of Al composites prepared by powder metallurgy*, 2nd International Conference for Young Chemists, 18th – 20th June 2008, Universiti Sains Malaysia.

5. Normah Cheman, Mohd Nazree Derman, Shamsul Baharin Jamaludin, Nur Maizatul Shima Adzli (2008), *Effects of magnesium addition on corrosion behaviour of PM Al- Mg composites reinforced with alumina Saffil™ short fibres,* Malaysian metallurgical Conference, 3th – 4th December 2008, Universiti Kebangsaan Malaysia.

TINGKAH LAKU KAKISAN KOMPOSIT MATRIK ALUMINIUM YANG DIPERKUAT DENGAN GENTIAN PENDEK ALUMINA SAFFIL™

ABSTRAK

Kehadiran elemen pengaloian dan bahan penguat akan mempengaruhi tingkah laku kakisan komposit matrik aluminum. Tumpuan kajian ialah pengaruh penambahan Mg dan gentian pendek alumina Saffil™ dalam AMCs yang diperbuat melalui kaedah metalurgi serbuk terhadap tingkah laku kakisannya. Sampel untuk ujian kakisan iaitu komposit PM Al dan komposit PM Al-Mg telah diperbuat daripada serbuk Al berbentuk kepingan , serbuk Mg berbentuk butiran dan gentian pendek alumina Saffil™ yang mempunyai purata ukuran butir masing-masing ialah 70.58 µm, 273.41 µm and 16.71 µm. Parameter kajian semasa menghasilkan komposit PM Al dan komposit PM Al-Mg ialah kandungan Mg (dari 0.5% berat hingga 3.0% berat) dan kandungan gentian pendek alumina Saffil™ (dari 5% berat hingga 25% berat). Sampel diperbuat dengan menggunakan tekanan searah sebanyak 200 MPa dan disinter dalam gas argon selama 6 jam. Sampel yang telah disinter dipercirikan dengan ujian mikrostruktur, ujian ketumpatan dan keliangan dan ujian kekerasan Vickers. Dua jenis ujian kakisan iaitu ujian rendaman dan ujian elektrokimia telah dijalankan ke atas komposit yang optima. Kedua-dua ujian tersebut menggunakan larutan 3.5% berat natrium klorida (NaCl) sebagai persekitaran kakisan. Lengkung pengutuban telah dilukis berdasarkan data dari ujian elektrokimia dengan menggunakan potentiostat jenis Gamry G300 yang telah dijalankan. Ujian redaman dijalankan selama 28, 56 dan 84 hari. Graf kehilangan berat sampel ujian rendaman dilukis berdasarkan data dari ujian rendaman. Photomikrograf sampel sebelum dan selepas ujian rendaman juga dinilai dengan menggunakan mikroskop biasan elektron (SEM). Elemen komposisi yang wujud dalam sampel dianalisa dengan menggunakan tenaga penyebaran sinar-X (EDX). Kehadiran fasa oxid dalam lapisan oxid di kenalpasti denagn analisis fasa pembelauan sinar-X (XRD). Daripada penyelidikan ini, menunjukkan penambahan magnesium dalam komposit PM Al telah dapat memperbaiki kebasahan di antara gentian pendek alumina Saffil™ dan matrik Al. Komposit yang optima ialah PM Al-15% berat Al_2O_3 dan PM Al-2%berat Mg-15% berat Al_2O_3. Kadar kakisan komposit PM Al pula menurun dari 67.69 µm/tahun ke 0.66 µm/tahun apabila 2% berat magnesium dimasukkan ke dalam komposit PM Al-Mg. Micrograf SEM pula menunjukkan kakisan bopeng berlaku di kawasan tumpuan juzuk Mg yang terdapat di permukaan komposit PM Al-Mg. Fasa aluminum oxid (δ-Al $Al_{1.67}O_4$), aluminium hydroxid ($Al(OH)^3$) and aluminium oxid (α-Al_2O_3) telah dikenalpasti wujud dalam lapisan oxid. Ketumpatan arus kritikal (I_{crit}) komposit PM Al (2.0 x 10^{-1} A/cm^2) dimana ianya lebih kecil berbanding dengan ketumpatan arus kritikal (I_{crit}) komposit PM Al-Mg (4.0 x10^{-1} A/cm^2). Magnesium bertindak sebagai perencat kepada tingkah laku kakisan dalam komposit PM Al-Mg dengan menukar dan melindungi komposit dari pembentukan lapisan oksid yang stabil. Rintangan kakisan komposit PM Al berkurang kerana pembentukan lapisan oksid dipermukaan tidak sekata dengan kehadiran gentian pendek alumina Saffil™ dalam komposit dan ini menyediakan tempat permulaan kakisan berlaku pada komposit.

CORROSION BEHAVIOUR OF POWDER METALLURGY ALUMINIUM MATRIX COMPOSITE REINFORCED WITH ALUMINA SAFFIL™ SHORT FIBRES

ABSTRACT

The corrosion behaviour of PM Al composite will be affected by the presence of alloying element and reinforcing phase in the composite. This studies were focused on the influence of magnesium addition and alumina Saffil™ short fibres reinforcement on corrosion behaviour of PM AMCs. PM Al composite and PM Al-Mg composite were fabricated from flaky aluminium powder, granular magnesium and alumina Saffil™ short fibres with an average particle size of 70.58 µm, 273.41 µm and 16.71 µm, respectively. The experimental parameter were maintained the content of magnesium powder (ranging from 0.5 wt% to 3.0 wt%) and alumina Saffil™ short fibres (ranging from 5 wt% to 25 wt%) . The samples were fabricated by using 200 MPa uniaxial pressing and sintered in argon atmosphera for 6 hours. Sintered samples were characterized by using SEM, density and porosity analysis and Vickers hardness testing. The optimum samples were tested in corrosion properties by using electrochemical test and immersion test. Both experiment using 3.5 wt% NaCl as corrosion environment. Electrochemical test was carried out with apply potentiostat Gamry G300 to create Tafel plot and polarization curve. The immersion test was carried out for 28, 56 and 84 days. Weight loss curves were acquired from immersion test results. Photomicrographs of samples were examined using scanning electron microscopy (SEM). The elemental composition of sample was analyzed by Energy dispersive X-ray (EDX) analysis. The presence of an oxide phases were verified by X-ray diffraction analysis (XRD). Experimental results showed the wetability between alumina Saffil™ short fibres and Al matrix have been improved by adding magnesium in the PM Al composite. PM Al – 15 wt% Al_2O_3 and PM Al – 2 wt% Mg - 15 wt% Al_2O_3 were identified as the optimum sample. The corrosion rate of PM Al composite was decreased from 67.69 µm/year to 0.66 µm/year when 2 wt% of magnesium was added in PM Al-Mg composite. SEM photomicrograph observed that pitting was localized surrounding the magnesium constituents which occur randomly throughout the surface of PM Al-Mg composite. XRD analysis revealed that aluminum oxide (δ-Al $Al_{1.67}O_4$), aluminium hydroxide (Al $(OH)^3$) and aluminium oxide (α-Al_2O_3) phases were detected in oxide film. The critical current density (I_{crit}) of PM Al composite is lower than PM Al-Mg composite which is 2.0×10^{-1} A/cm^2 and 4.0×10^{-1} A/cm^2, respectively. Magnesium is used as inhibitor in corrosion behaviour of PM Al-Mg composite have been changed and protected with a stable formation of oxide film. The corrosion resistance of PM Al-composite was decreased with the presence of alumina Saffil™ short fibres in composite due to discontinuities in oxide film provided the corrosion initiated in sample.

CHAPTER 1

INTRODUCTION

1.1 Research introduction

Metal matrix composites (MMCs) are under attention for many applications in aerospace and automobile industries. MMCs are classified by the type of metal which is involved matrix and reinforcement. Aluminium, titanium and magnesium are widely used as matrix materials. Meanwhile, silica carbide (SiC) and alumina (Al_2O_3) are the most commonly used as reinforcement. Among MMCs, aluminium matrix composites (AMCs) are being the main interest due to the superior properties such as light weight, high strength, high specific modulus, low coefficient of thermal expansion, good wear resistance properties as compared with monolithic alloys.

Major fabrication methods used for aluminium matrix composites are powder metallurgy, casting and spray deposition. The powder metallurgy (PM) technique for manufacturing AMCs offer some advantages compared with casting. The main advantage is the low manufacturing temperature that avoids strong interfacial reaction and minimizing the undesired reactions between the matrix and the reinforcement (Torralba et al., 2003; William and Harrigan, 1998). PM technique also efficient in energy operation, less anisotropy, easy for automation and economically for certain type of products for example small components which would ordinary require a prohibitive a mount of machining and material waste.

AMCs via powder metallurgy technique is produced the complex, near net-shape and lightweight component. The demands for lightweight metals are increased tremendously especially in automotive industry. Lightweight materials are used as automotive components for reducing fuel consumption. Consequently, annual production of aluminium PM blended is expected will increase from 1200 tonnages in 1998 to 25000 tonnages within 10 years (Bishop et al., 2000).

AMCs have developed rapidly since the first appeared in 1960s. A number of researchers have attempted to enhance AMCs by adding various of alloying elements and reinforcements. The alloying elements such as magnesium, copper, silicon and manganese are usually incorporated into aluminium alloys. Meanwhile, ceramic particles and short fibres reinforcements such as SiC and Al_2O_3 are mostly used in AMCs. Indeed, short fibres reinforcements are used as engineering materials because the composites have good properties at low cost as compared to particles reinforcements.

In this research, powder metallurgy technique has been chosen to fabricate Al-Mg composite reinforced with short fibre alumina Saffil™. PM Al-Mg composite have a good properties such as near net-shape, light weight, have a low coefficient of thermal expansion and excellent mechanical properties as compared with aluminium alloys.

However, the corrosion resistance of PM Al-Mg composites are lower than pure aluminium due to magnesium alloying and short fibres alumina Saffil™ reinforcement. The previous researchers such as Tiwari et al. (2006), Bakkar and Neurbert (2007) have found that corrosion problems likely occurred in AMCs is galvanic coupling between the reinforcing phase and matrix and the selective corrosion at the reinforcement and matrix interface. Thus, the addition of magnesium and short fibre alumina Saffil™ in Al-Mg composites will be influenced the corrosion behaviour. Moreover, the porosity in composites will be arising from powder metallurgy technique also can alter the corrosion behaviour of Al-Mg composites.

1.2 **The effect of magnesium addition in Al-Mg composites on corrosion behaviour**

Magnesium is one of the lightest metallic materials available and has the lowest density. Magnesium has natural affinity for wetting or bonding to reinforcing materials. On the contrary magnesium has low stiffness, low strength, low wear resistance and high coefficient of thermal expansion.

The addition of magnesium in Al-composite and Al alloy can improve wettability. Wetting is an important aspect in AMCs synthesis for formatting strong chemical bonds at the interface between reinforcement and matrix. Asavavisithchai and Kennedy (2006) and Schaffer et al. (2001) were supported that the wettability of PM Al composite reinforced with alumina (Al_2O_3) particles and sintered Al-Mg alloy have been improved by adding the highly reactive magnesium.

However, magnesium addition in Al-Mg composites can cause galvanic corrosion because two dissimilar metal in contact with each other. Magnesium has been chosen as alloying elements in this study for reducing galvanic corrosion potential because magnesium and aluminium are closer in EMF Series as shown in Table 1.1. Magnesium also perform as good as aluminium and better than mild steel in rural and mild industrial environments but magnesium frequently suffer severe damage in wet and salt-containing environments (Eliezer and Alves, 2002).

Table 1.1: Standard Electrode Potential (EMF) series of metals (Fontana, 1987)

	Metal-metal ion Equilibrium (unit activity)	Electrode potential Vs. normal hydrogen Electrode at 25°C, volts (V vs SHE)
↑	$Au\text{-}Au^{+3}$	+1.498
	$Pt\text{-}Pt^{+2}$	+1.2
	$Pd\text{-}Pd^{+2}$	+0.987
	$Ag\text{-}Ag^{+}$	+0.799
Noble or Cathodic	$Hg\text{-}Hg_2^{+2}$	+0.788
	$Cu\text{-}Cu^{+2}$	+0.337
	$H_2\text{-}H^{+}$	
		0.000
	$Pb\text{-}Pb^{+2}$	
	$Sn\text{-}Sn^{+2}$	-0.126
	$Ni\text{-}Ni^{+2}$	-0.136
	$Co\text{-}Co^{+2}$	-0.250
	$Cd\text{-}Cd^{+2}$	-0.277
	$Fe\text{-}Fe^{+2}$	-0.403
	$Cr\text{-}Cr^{+3}$	-0.440
	$Zn\text{-}Zn^{+2}$	-0.744
Active or Anodic	$Al\text{-}Al^{+3}$	-0.763
	$Mg\text{-}Mg^{+2}$	-1.662
	$Na\text{-}Na^{+}$	-2.363
	$K\text{-}K^{+}$	-2.714
↓		-2.925

1.3 **The effect of short fibres alumina Saffil™ reinforcement on corrosion behaviour**.

The incorporation of second reinforcing phase into metal matrix composite not also enhances the physical and mechanical properties of a material, but it also can alter the material's corrosion behaviour. The presence of the short fibres alumina Saffil™ reinforcement will be accelerated the corrosion in Al-Mg composites as compared to unreinforced Al alloy. Accelerated corrosion is originated from electrochemical and chemical interaction between Al-Mg composite constituents and microstructural effects.

According to Winkler and Flower (2004), corrosion will be affected by reinforcement addition in MMCs. A reaction between the reinforcement and metal matrix occurs during the processing of metal matrix composite and formed an interfacial phase that binds the two materials together. Thus, preferential corrosion will be occurred at the interfacial phase which is strongly anodic or cathodic.

Pardo et al. (2004), also reported that thermal stress arise at the interfaces between the matrix and the reinforcement due to considerable mismatch the thermal expansion coefficient when a metal matrix composite is subjected to temperature changes. As a result a dislocation at interface is generated. The dislocation could lead to further discontinuities in the oxide film, increasing the number of sites where corrosion can be initiated and rendering the composite liable to severe attack. Meanwhile, Zhang et al. (2007), Dobrzanski et al. (2005) and Datta et al. (2004) were founds that the corrosion attacked adjacent to the fibers in MMCs.

1.4 The effect of the powder metallurgy process on corrosion behaviour

The corrosion behaviour will be affected by changing a microstructure of composites and the presence of defects from manufacturing processes. Yoshiaki et al. (1995) have found that manufacturing parameter can affect the corrosion behaviour. Bakkar and Neubert (2007), also have been reported that microstructure changes and process contaminants resulted from manufacture of the MMCs.

Powder metallurgy is one of a method to manufacture Al-Mg composites. However, processing materials through conventional powder metallurgy method will be resulted the presence of such micro pores (Chandramouli et al., 2007) and defects in Al-Mg composite were occurred during compaction and sintering process (Updhyaya, 2000). The tenacious oxide film over the aluminium particles will be ruptured during compaction process, thus resulting in metal-to-metal contact. Meanwhile, during sintering, aluminium expands more than aluminium oxide layer over particles. As a result, the hair-line cracks will be developed on the oxide layer.

The micro pores and defects will be influenced the corrosion behaviour of Al-Mg composites. Silva-Maia et al. (1999) and Hosni et al. (2008) has been reported that corrosions are closely associated to the presence of heterogeneities such as porosity, voids, or precipitates. Based on the study from Shankar et al. (2004), PM stainless steel applications were limited due to the relatively poor mechanical and corrosion properties when compared to wrought products.

According to Pardo (2004), a research on corrosion properties of aluminium matrix composites is still at development stage. Thus, the purpose of this research is to study the corrosion behaviour of PM Al-Composites reinforced with short fibres alumina Saffil^{TM}. The study will be focused on the effects of alloying magnesium and alumina Saffil™ reinforcement on corrosion behaviour of Al-Mg composites.