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Nurul Razliana Bte Abdul Razak

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LIST OF ABBREVIATIONS

Ag	Argentum
Al	Aluminium
Al ₂ O ₃	Aluminium oxide/alumina
AlB	Aluminium boride
AlB ₂	Aluminium diboride
AlN	Aluminium nitride
AMC	Aluminium matrix composite
B	Boron
B ₄ C	Boron carbide
BN	Boron nitride
C	Carbon
CMC	Ceramic matrix composite
Cu	Copper
EAA	Polyethylene-co-acrylic acid
EVA	Ethylene vinyl acetate
FCC	Fluid catalytic cracking
FDA	Food and Drug Administration
Fe-Cr	Iron-chromium
GC	Gas chromatography
GC-MS	Gas chromatograph-mass spectrometer
H ₂ S	Hydrogen sulphide

HDPE	High density polyethylene
HMS	Hexagonal mesoporous silica
HZSM	Hydrogen – zeolite Socony Mobil
ICSD	Inorganic crystal structure database
LDPE	Low density polyethylene
$\text{Li}_5\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_4$	Cobalt-doped lithium battery (Li_5FeO_4)
MCM	Mesoporous molecular sieve
Mg	Magnesium
MgFe_2O_4	Magnesium ferrite
MgO	Magnesium oxide
MMC	Metal matrix composite
MTS	Magnesium trisilicate
NiFe_2O_4	Nickel ferrite
OTC	Over the counter
Pb	Plumbum
PB	Polybutene
PCA	Process control agent
Pd	Palladium
PE	Polyethylene
PM	Powder metallurgy
PMC	Polymer matrix composite
PMMA	Poly(methyl methacrylate)
PP	Polypropylene

PS	Polystyrene
PVA	Polyvinyl acetate
rpm	Rotation per minute
SA	Stearic acid
SEM	Scanning electron microscope
Si	Silicon
Si ₃ N ₄	Silicon nitride
SiC	Silicon carbide
SiCp	Silicon carbide particulate
SiO ₂	Silicon dioxide /silica
Sn	Stannum
Ti	Titanium
TiC	Titanium carbide
W	Tungsten
XRD	X-ray diffraction
ZnO	Zinc oxide
ZrSiO ₄	Zircon

LIST OF SYMBOLS

°C	Degree Celsius
%	Percentage
°/min	Degree per minute
°C/min	Degree Celsius per minute
°2	Degree two theta
μm	Micronmeter
Å	Armstrong
cm ³	Centimeter cubic
g	Gram
kV	Kilovolt
mA	Milliampere
min	Minute
mL/min	Milliliter per minute
mm/min	Millimeter per minute
MPa	Megapascal
N	Newton
psig/min	Pound per square inch per minute
rpm	Rotation per minute
wt%	Weight percent
	Lambda

Pencirian dan Sifat-Sifat Komposit Aluminium/Zink Oksida Sebagai Pemangkin Untuk Penyahpolimeran Polietilena Ketumpatan Rendah Kepada Minyak Cecair

ABSTRAK

Kesan penambahan zink oksida (ZnO) atau magnesium trisilikat (MTS) dalam komposit matrik aluminium (Al) ke atas sifat-sifat fizikal dan mekanikal pada suhu pensinteran yang berbeza telah dikaji. Komposit Al/ZnO dan Al/MTS disediakan menggunakan kaedah metalurgi serbuk (MS). Komposit dicampur menggunakan mesin pengisar pada kelajuan 131 rpm dengan nisbah berat bola kepada berat serbuk adalah 10:1, kemudian dimampatkan pada 200 MPa selama 2 minit. Keputusan menunjukkan penambahan ZnO atau MTS ke dalam komposit Al telah meningkatkan ketumpatan dan kekerasan komposit tetapi kekuatan mampatan didapati berkurangan. Komposit Al/ZnO atau Al/MTS yang disinter pada suhu 750 °C mempunyai ketumpatan, kekerasan dan kekuatan mampatan yang lebih tinggi daripada komposit yang tersinter pada suhu 650 °C. Kesan suhu pensinteran yang berbeza memberi kesan kepada intensiti komposit. Mikrostruktur komposit Al/ZnO dan Al/MTS yang tersinter pada 750 °C menunjukkan interaksi yang lebih baik diantara pengisi dan matrik Al. Kesan asid stearik atau zink stearat kepada komposit Al/ZnO telah meningkatkan kekerasan dan kekuatan mampatan komposit tetapi ketumpatan didapati berkurang apabila disinter pada suhu 750 °C. Corak XRD komposit Al/ZnO dengan asid stearik atau zink stearat menunjukkan perubahan puncak keamatan komposit. Kehadiran asid stearik dan zink stearat telah meningkatkan pelekatan dan interaksi di antara ZnO dan matrik Al. Komposit Al/ZnO dengan zink stearat mempunyai sifat-sifat fizikal dan mekanikal yang lebih tinggi dibandingkan dengan komposit yang lain. Kegunaan komposit Al/ZnO dengan zink stearat sebagai pemangkin di dalam penyahpolimeran polietilena ketumpatan rendah (LDPE) telah menghasilkan 76.22 % minyak cecair. Produk minyak cecair mengandungi hidrokarbon ringan seperti gasolin telah dibuktikan dengan menggunakan kromatografi gas – meter spektrum jisim (GC-MS).

Characterisation and Properties of Al/ZnO Composites as Catalyst for Depolymerisation of Low Density Polyethylene to Liquid Oil

ABSTRACT

The effects of addition zinc oxide (ZnO) or magnesium trisilicate (MTS) in aluminum (Al) matrix composites on physical and mechanical properties at different sintering temperature were studied. The Al/ZnO and Al/MTS composites prepared by powder metallurgy (PM) method. The composites were mixed using a milling machine at a speed of 131 rpm with ball to powder weight ratio is 10:1, then compressed at 200 MPa for 2 minutes. The results show that the additions of ZnO or MTS in Al composites have increased the density and hardness but decrease the compressive strength of the composites. The Al/ZnO or Al/MTS composites sintered at 750 °C have higher density, hardness and compressive strength than composites sintered at 650 °C. The effect of different sintering temperature gives effect to intensity of the composites. The microstructure of Al/ZnO or Al/MTS sintered at 750 °C show better interaction between filler and Al matrix. The effect of stearic acid or zinc stearate in Al/ZnO composites has increased the hardness and compressive strength of composites but density reduced at sintered 750 °C. The XRD patterns of Al/ZnO composites with stearic acid or zinc stearate show the changed in intensity peak of composites. The presence of stearic acid and zinc stearate as binder improved the adhesion and interaction between ZnO and Al matrix composites. The Al/ZnO composites with zinc stearate have higher physical and mechanical properties compared to other composites. The applied of Al/ZnO composites with zinc stearate as catalyst in the depolymerisation of low density polyethylene (LDPE) has produced yield 76.22 % of liquid oil. The liquid oil product consists of light hydrocarbons such as gasoline was proven by using gas chromatography - mass spectrometer (GC-MS).

CHAPTER 1

INTRODUCTION

1.1 Research Background

The development of composite materials has become a necessity for modern technology due to the improved physical and mechanical properties. Metal matrix composites (MMC) have been developed in recent years, among which aluminium matrix composites have found various applications in the industry. This is due to the low density, high toughness and corrosion resistance (Kok, 2005; Rahimian et al., 2009a; Rahimian et al., 2009b). However, the drawback of these is their high production cost (Torralba et al., 2003).

Among the advance engineering materials, aluminium based metal matrix composites (MMC) show potential to develop novel lightweight high performance materials due to their remarkable properties. In addition, MMCs offer the possibility to tailor their properties to meet specific requirements, which made this type of materials quite unique in comparison to conventional unreinforced materials (Scudino et al., 2009). The family of discontinuously reinforced MMCs (e.g. particulate-reinforced composites) is particularly attractive due to their easier fabrication routes, lower costs and nearly isotropic properties compared to the continuously reinforced MMCs (Clyne & Withers, 1993). The discontinuously reinforced MMC has been successfully prepared by powder metallurgy (PM) (Slipenyuk et al., 2006; Yu et al., 2006; Lee et al., 2004; Scudino et al., 2008; Scudino et al., 2009; Schurack et al., 2008; El Kabir et al., 2008; Tang et al., 2003).

Ceramic particles are one of the most reinforcements being used in MMCs. This is due to its capability to increase strength, hardness, stiffness, chemical stability, thermal stability and thermal resistivity of the composites. MMC with ceramic reinforcement provides good combination strength attained from ceramic reinforcements and toughness due to the underlying metal matrix (Gudlur et al., 2012).

The PM is thought to be the most common production technique for MMC. One of the advantages of PM compared to casting having better control of microstructure, where better distribution of the reinforcement is possible in PM compacts. One of the advantages of this process is the ease of mixing of the different metal powders. This leads to the possibility to create new composite materials with special physical and mechanical properties in a component which cannot be produced by the melting-casting process (Yamaguchi et al., 1997).

The PM process is cost effective because it minimizes machining, produces a good surface finish and maintains close dimensional tolerances. Generally, the PM forming processes include cold compaction, sintering and finishing (Poquillon et al., 2002).

One of the best properties of composites produced by the PM method can be obtained when the reinforcement is homogeneously dispersed in the matrix, as approved by experiment (Slipenyuk et al., 2004; Kumai et al., 1996) and theoretical (Geni et al., 1998; Boselli et al., 2001; Baccino et al., 2000). Particulate reinforced MMCs improved both mechanical and physical properties and PM processing is one of the suitable methods to fabricating these composites (Sahin, 2007).

Particle size and the amount of reinforcement have pronounced effect on the mechanical properties of composites. The proper addition of reinforcements of aluminium composites has a positive effect on mechanical properties such as hardness, strength and wears resistance (Park et al., 2001; Dobrza ski et al., 2006). It has been well established

that the addition of ceramic particle to aluminium improves its strength, wear resistance and corrosion resistance (Torres et al., 2002; Sahin & Murphy, 1996).

In recent years many researches have been considerably investigated particle reinforced metal matrix composites (Miserez et al., 2004; Torralba et al., 2003; Abdizadeh et al., 2011). The manufacturing of A3561 nano Al_2O_3 , Al/nano MgO composites using the PM method were investigated by (Mazahery et al., 2009; Ansary Yar et al., 2009). The different properties of these composites affected by applied conditions have been investigated. The processing temperature, reinforcement fraction, particle size and the matrix strength are some of the factors studied and found out to be effective on the properties of the produced composites such as microstructure, mechanical behaviour, hardness and tensile strength (Shen et al., 2001; Abdizadeh et al., 2008).

Zinc oxide (ZnO) is one of the semiconducting materials with wide band gap and superior intrinsic properties such as high melting point, high hardness as well as excellent thermal stability and chemical inertness. All these features make ZnO a promising candidate to be reinforcing second phase in matrix composites. The studies on the Ag/ZnO and Cu/ZnO composites used as contact materials have been reported by researchers (Kang & Park, 1999; Wang et al., 2010).

Magnesium Trisilicate (MTS) is an inorganic compound. The function of MTS as an abrasive, absorbent, anticaking agent, bulking agent, opacifying agent and viscosity increasing agent-aqueous in cosmetic. MTS is a compound of magnesium oxide (MgO) and silicon oxide (SiO_2) with varying proportions of water. It contains oxide not less than 20 % of MgO and 45 % of SiO_2 (Wenniger et al., 2000). The investigation of the introduction of ZnO and MTS into Al has not been found in the literature yet.

However, homogeneous distribution and fine particle size are two main requirements for the reinforcement of dispersion strengthened materials. The conventional

melting and casting techniques are not useful because they are unable to give good uniform dispersions. The composites fabricated by ingot metallurgy usually present coarse grains, which would dramatically degrade the toughness (Lopez et al., 2007). Therefore the best way to fabricate this kind of materials should be the use of the PM technique. It has also been reported (Schaffer et al., 2001) that segregation and delubrication can be an important issue with the processing of PM of an appropriate binder or lubricant has not been used. This can cause preferentially unsafe surroundings with inhomogeneity and delubrication defects within the sintered product. The binder is usually composed of several kinds of polymer, wax and lubricant components (Takekawa, 1996). It was found that addition of solid binder or lubricant such as lithium stearate, and acrawax to both the premixed and prealloyed powders reasonable green densities and microstructure. Other than a binder or lubricants such as zinc stearate, stearic acid and liquid paraffin provided similar green densities but higher sintered densities and less porous microstructure (Gökçe & Findik, 2011).

The plastic materials have a significant success in the market due to multiple applications because they have good physical and mechanical properties and in many cases, replace other materials such as glass or wood. Once the plastic has finished their use, these materials from part of carbon solid waste, generating great environmental impact, because such material has very long period of biodegradation (González et al., 2011).

Polyolefin such as polyethylene (PE), polypropylene (PP) and polystyrene (PS) represents the most abundant polymers in industrial and municipal plastic waste. Their high chemical and energy content makes them interesting as potential sources of materials and energy at the end of the life (Brandrup et al., 1996).

On the other hand, world market energy consumption is projected to increase by 44 % from 2006 to 2030. This meant that crude oil consumption has to increase from 85 million billions per day in 2006 to 99 million barrels per day in 2015 and 122 million barrels per day in 2030. Consequence of the high demand for oil, its availability will decrease and its price can become excessively high. An alternative to the energy obtained from crude oil could be the energy obtained from plastic wastes (International Energy outlook USA, 2009).

The incineration can be applied for this purpose, but this alternative does not have much acceptance due to the emissions associated dioxins, greenhouse gases and discharges of ashes, as well as the attitude of the population rejected the construction of incinerations means near residential areas (González et al., 2011).

Pyrolysis is another alternative for obtaining energy from plastic waste. It is based on the principle that most organic substances are thermally unstable and they can be broken in an oxygen-free atmosphere (Miskolczi et al., 2004). The using pyrolysis process, plastic is transformed into fuel like products (Siddiqui & Redhwi, 2009b; Siddiqui & Redhwi, 2009c; Siddiqui, 2009a; Marcilla et al., 2008). This is highly endothermic process but a suitable technology for plastic waste treatment since fuel like product obtained from plastic waste have a calorific power similar to some fuel and natural gas and higher than coal. They have a high pyrolytic potential due to the energy emitted.

The two different methods of pyrolysis of plastic waste have been reported: thermal degradation (Miskolczi et al., 2004; Siddiqui & Redhwi, 2009c) and catalytic degradation (Siddiqui, 2009a; Siddiqui & Redhwi, 2009b). The different of solid catalyst can be used catalytic degradation: molecular sieve (Marcilla et al., 2007; Marcilla et al., 2008) alumina and aluminosilicate (Hayashi et al., 2007; Sakata, 1997; Aguado et al.,

1997; Aguado et al., 2002), silica gel (Uddin et al., 1998; Chaianansutcharit et al., 2007), activated carbon (Buekens & Huang, 1998) and fluid catalytic cracking (FCC) catalyst (Arandes et al., 2007; Achilias et al., 2007).

Aguado et al., (1997 & 2002) studied the use of mesoporous aluminosilicates type MCM-41 for liquid fuel production from PE wastes. Few experimental works with activated carbon catalyst have been reported. In some experiments of the materials has been used as a support for some metal such as iron, molybdenum or zinc (Buekens & Huang, 1998).

Murata et al., 2010 reported that the effect of silica-alumina catalyst on degradation of polyolefin by continuous reaction. The continuous flow reactor was operated at atmospheric pressure and polyolefin over two silica-alumina catalyst having different $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratio. The cracking effect of silica-alumina was proven by the increased amount of the gaseous product and by the decreased molecular weight of liquid product. PE, PP and PS molar rate of degradation was increased in the presence of a catalyst and affected the distributions of degradation products.

1.2 Problem Statement

Recycling plastic through the tertiary recycling process, either thermal or catalytic cracking are very attractive since it can potentially be used to convert the thermoplastic polymers into more useful end products like char, oil, and gas (Abbas-Abadi et al., 2012; Passamonti & Sedran, 2012). By using catalytic cracking, catalyst was added because it can promote the pyrolysis reaction to occur at lower temperatures which implies lower energy consumptions (Lin & Yang, 2007). Based on previous studies, there are many catalysts that were used in catalytic cracking such as molecular sieves, activated carbon,

zeolites, silica alumina and few more. According to Passamonti & Sedran (2012), catalytic cracking of low density polyethylene (LDPE) and high density polyethylene (HDPE) were commonly over an acidic catalyst such as HZSM-5, metal exchanged, zeolites, amorphous silica-alumina and crystalline mesoporous materials. All of these catalysts were produced usually by involving various chemical method and procedure which are complicated and high cost. Moreover, study of fabricating effective catalyst with low cost of production for this catalytic cracking was developing. Based on previous studies, no available study has been found in the development of composite catalyst made from zinc oxide or magnesium trisilicate in the aluminium matrix. The addition of zinc oxide or magnesium trisilicate in aluminium matrix was to reduce cost production. The new composites based on aluminium/zinc oxide and aluminium/magnesium trisilicate was applied as a catalyst in depolymerisation of low density polyethylene (LDPE). Aluminium was chosen as the matrix since it is one of the easiest metal matrixes to fabricate and have outstanding properties. Zinc oxide was selected as the filler in this due to its capability to use as catalyst especially for methanol synthesis (Andreasen et al., 2006) while magnesium trisilicate was selected because it consists of high content of silica (SiO_2) compounds which also commonly been used as a catalyst.

1.3 Research Objectives

The objectives of this research are:

1. To study the effect of different composition of the ZnO or MTS as a filler in the Al matrix composites with different sintering temperature on physical, mechanical properties, XRD and morphology of composites.

2. To study the effect of stearic acid or zinc stearate as binders on properties of Al/ZnO composites.
3. To apply the Al/ZnO as catalyst for the depolymerisation of low density polyethylene.

1.4 Scope of study

This research study focussed on the investigation of physical, mechanical and microstructure of the Al/ZnO and Al/MTS composites with and without the presence of a binder. The physical testing was carried out using a gas pycnometer to determine the density of the composites. The density of the composites was studied to determine either the composites has been densified or not with additional of different types of filler at different compositions and sintering temperature. The mechanical testing was carried using microhardness. Hardness test was carried out in order to measure the hardness of the microstructural constituents of the composites. Universal testing machine used to determine its compressive strength. The compressive strength was carried out to measure the maximum compressive strength in which indicates the capability of the composites from deformation under continuous compressive load. The morphological study of the composites was studied by using scanning electron microscopy (SEM). The purpose of the XRD analysis is to examine the phase existed when different filler was added into the composites and any chemical changes occur on the composites when different sintering was used.