EFFECT OF MAGNESIUM AND TIN AS SINTERING ADDITIVES ON MICROSTRUCTURE AND COMPRESSIVE PROPERTIES OF POROUS ALUMINUM

by

AMIRAH AHMAD HAMDI

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LIST OF ABBREVIATIONS

Ag	Silver
Al	Aluminum
Al_2O_3	Alumina
$CaCl_2$	Calcium Chloride
CYA	$C_3N_3(OH)_3$
Cu	Copper
CH_3CH_2OH	Ethanol
CH_4N_2O	Carbamide
E	Energy absorption efficiency
EDX	Energy dispersive X-ray spectroscopy
f_{Al}	Aluminum weight fraction
FESEM	Field emission scanning electron microscopy
I	Ideal energy absorption efficiency
Mg	Magnesium
$MgAl_2O_4$	Magnesium aluminate
Mg_2Sn	Magnesium stannide
NaCl	Sodium chloride
NH_4HCO_3	Ammonium bicarbonate
OM	Optical microscope
Pb	Lead
SDP	Sintering dissolution process
SEM	Scanning electron microscope

Si	Silicon
SiC	Silicon carbide
Sn	Tin
TGA	Thermal gravimetric analysis
Ti	Titanium
TiH_2	Titanium hydride
W	Energy absorption capability
W_a	Weight of dry porous Al
W_b	Weight of porous Al in water
W_c	Weight of wet porous Al
XRD	X-ray diffractometer
Zn	Zinc
ρ_{rel}	Theoretical relative density
ρ_{Al}	Density of aluminum
ρ_{liquid}	Density of water
$\rho_{space\ holder}$	Density of space holder
γ_{lv}	Surface tension of liquid-vapour
γ_{sv}	Surface tension of solid-vapour
γ_{sl}	Surface tension of solid-liquid interface
ε	Strain
ε_d	Densification strain
ε_m	Maximum strain
σ	Stress
σ_{pl}	Compressive strength

σ_{ys}	Yield strength
σ_m	Maximum stress

LIST OF SYMBOL

Å	Angstrom
atm	Standard atmosphere
°C	Degree Celsius
°C/min	Degree Celsius per minute
g/cm^3	Gram per cubic centimeter
g/L	Gram per litre
kN	Kilo Newton
L/min	Litre per minute
MJ/m^3	Mega Joules per meter cubic
MPa	Mega Pascal
mm	millimetre
nm	Nanometer
rpm	Revolutions per minutes
µm	Micrometre
vol.%	Volume percent
wt.%	Weight percent
%	Percent
Ø	Diameter

KESAN MAGNESIUM DAN TIN SEBAGAI BAHAN TAMBAH PENSINTERAN PADA MIKROSTRUKTUR DAN MAMPATAN BUSA ALUMINIUM

ABSTRAK

Objektif utama penyelidikan ini adalah untuk meningkatkan sifat mampatan busa aluminium dengan penambahan bahan tambah pensinteran; magnesium (Mg) dan timah (Sn). Penambahan Mg bersama Sn meningkatkan tindak balas pensinteran busa Al melalui mekanisma pensinteran fasa cecair. Busa Al telah dihasilkan melalui kaedah metalurgi serbuk menggunakan karbamida sebagai pemegang ruang. Proses bermula menggunakan amaun karbamida yang berbeza (20, 40 dan 60 bt.%) dalam busa Al-1.0Mg. Selepas mencapai komposisi terbaik karbamida dengan ciri-ciri dikehendaki, jumlah penambahan Mg adalah berbeza dari 0.2, 0.6, 1.0, 1.2 dan 1.6 bt.%. Proses kemudian diteruskan menggunakan amaun Sn yang berbeza (0.2, 0.6, 1.0, 1.2, 1.6, 2.0, 2.4, 3.0 and 4.0 bt.%) dalam busa Al-1.0Mg. Pencirian sifat seperti mikrostruktur, morfologi, fasa, ketumpatan, keliangan dan kekuatan mampatan dijalankan menggunakan mikroskop optik (OM), mikroskop elektron imbasan (SEM), pembelauan sinar-X (XRD), ujian ketumpatan dan keliangan dan ujian mampatan. Didapati busa Al-1.0Mg mempunyai keliangan dalam lingkungan 36.0% hingga 75.4% dengan julat ketumpatan dari 1.75 hingga 0.67 g/cm^3 dengan penambahan karbamida daripada 20 bt.% hingga 60 bt.%. Struktur berliang diperolehi dengan penyingkiran karbamida melalui proses perlarutan. Penambahan 1.0 bt.% Mg dapat mencapai kekuatan mampatan 4.20 MPa dan penyerapan tenaga 7.35 MJ/m^3. Manakala, busa Al-1.0Mg-2.4Sn menghasilkan kekuatan mampatan yang paling tinggi (17.72 MPa) dan penyerapan tenaga paling tinggi (8.67 MJ/m^3).

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ABSTRACT

The main objective of this research was to improve the compressive properties of porous aluminum by addition of sintering additives; magnesium (Mg) and tin (Sn). Addition of Mg with Sn enhance sintering response of porous Al via liquid phase mechanism. Porous Al were fabricated by powder metallurgy method using carbamide as space holder. The process begin by varies the amounts of carbamide (20 wt.%, 40 wt.% and 60 wt.%) in porous Al-1.0Mg. Then after achieving the best composition of carbamide with desired properties, amount of Mg was varied (0.2, 0.6, 1.0, 1.2 and 1.6 wt.%). The process then followed by varying the amounts of Sn (0.2, 0.6, 1.0, 1.2, 1.6, 2.0, 2.4, 3.0 and 4.0 wt.%) in porous Al-1.0Mg. Characterization such as microstructure, morphology, phases, density, porosity and compressive strength were done using optical microscope (OM), scanning electron microscope (SEM), X-ray Diffractometer (XRD), density and porosity test and compression test. It was found that porous Al-1.0Mg has porosities in the range of 36.0% to 75.4% with density ranges from 1.75 to 0.67 g/cm^3 with additions of carbamide from 20 wt.% to 60 wt.%. Porous structure was obtained with the removal of carbamide through dissolution process. The addition of 1.0 wt.% Mg was able to achieve compressive strength of 4.20 MPa and energy absorption of (7.35 MJ/m^3). Whereas, porous Al-1.0Mg-2.4Sn has produced the highest compressive strength (17.72 MPa) and the highest energy absorption (8.67 MJ/m^3).

CHAPTER ONE

INTRODUCTION

1.1 Research Background

Porous materials are cellular materials which consist of air filled pores. This pores can be either interconnected (open cell) or isolated (closed cell). Porous materials are good for functional applications such as filtration, insulation and also for structural application where weight reduction are essential such as in automotive and aerospace (Fang et al., 2017). However, for structural application, porous metals are superior to porous polymer and porous ceramic as polymer are insufficiently rigid and ceramic are too brittle. In addition, porous metals are fully recyclable and nontoxic. The most commonly used porous metals includes porous aluminum, titanium and magnesium.

Porous aluminums (Al) are in great attention with increasing applications due to its unique properties compared with dense solid aluminum, which includes high specific strength and stiffness and low weight, crushable and exhibit plateau region when compressed. These are the most important features for energy absorption (Pinto et al., 2014). Fabrication methods of porous Al have been developed and studied to tailor the required final properties to make it suitable for specific applications. Methods that commonly used to fabricate porous Al are melt gas injection, melt foaming agent, investment casting and powder metallurgy using space holder. Most of the mentioned methods either provide a limited porosity or uncontrolled porosity. However, powder metallurgy using space holder is capable of producing porous Al with in control pores size, shape and porosity. Generally, powder metallurgy method using space holder consists of mixing, compacting, dissolution and sintering process. This method can fabricate porous Al as pores created due to the removal of space holder material either by thermal decomposition or dissolved in suitable solvent. Current researcher has used several materials as a space holder includes sodium chloride (NaCl), carbamide, ammonium bicarbonate and tapioca starch (Jha et al., 2013). The size, shape and porosity of porous Al can be easily altered by changing the size, shape and amount of space holder. With increasing amount of space holder, the relative density of porous Al expected to be decreased. According to the work done by Bafti & Habibolahzadeh (2013), Cadena et al. (2016) and Michailidis et al. (2011), they found that the amount of space holder directly affected relative density and compressive properties of porous Al. In addition, physical and mechanical properties of porous Al are also reported to be influenced by the morphology and distributions of pores in porous Al. Moreover, Bafti & Habibolahzadeh (2013) who studied the effect of angular and spherical shaped of space holder found that the compressive strength of porous Al with spherical pores are significantly higher than angular pores porous Al which having the same porosities.

Aluminum powder are always covered by stable oxide film which reduces it sinterability. An addition of 0.1–1.0 wt.% of Mg has been found to reduces the oxide layers on the surface of the Al particles through the formation of a spinel phase $MgAl_2O_4$. The use of liquid phases is another alternative to solid state sintering. According to Showaiter & Youseffi (2008) the advantages of liquid phase sintering improved the densification of Al alloy. Liquid phase sintering of Al alloy has been widely investigated using Ag, Cu, Pb, Sn and Zn as sintering additives. Of them, addition of Sn with Mg is the most commonly used.

1.2 Problem statement

Porous Al produces by powder metallurgy using space holder method can be used in energy absorption application. The increasing energy absorption capability of porous Al is corresponding to the increasing porosity of the porous Al. However, porous Al with high porosity has low strength and stiffness. Hence, the challenge is to compromise between the amount of carbamide used with the compresive properties of porous Al. Porous Al have been fabricated using space holders with a small range variations of porosities. For instance, raw cane sugar has been used as space holder to obtain porosity in the range of 40.0 % to 70.0 % by Michailidis et al. (2011). Jiang et al. (2005) used carbamide as a space holder to obtain porosity in range of 50.0 % to 80.0 %. Similarly, carbamide was used to acquire the porosity in the range of 40.0 % to 85.0 % by Bafti & Habibolahzadeh (2013). Hence a systematic studies on the effect of space holder amount on the morphology and mechanical properties of porous Al-Mg with wide range of porosity to the extent of 33.5 % to 75.0 % were carried out in this work.

Magnesium (Mg) powder has previously been used in fabricating porous Al by powder metallurgy using space holder method to reduce the oxide layer of the Al particles (Hassani et al., 2012). Mg absorbs the oxygen trapped in the compact and reduces the Al oxide layer. As a consequence, more fresh metal contacts between the Al particles are formed and the Al particles are subject to less oxidation during sintering. Hence improved the sintering response. The former Sun & Zhao (2003) study revealed that adding 0.15 wt.% Mg to the compact improved the bonding between the Al particles markedly and increased significantly the static and impact energy absorbing capacities of the porous Al. An addition of 0.5 wt. % Mg was found to be sufficient for achieving good quality porous Al. In other study by Zhao et al. (2004) it was found that addition of Mg powder did not seem to induce benefit to liquid state sintering, but adding both Sn and Mg in the porous Al exhibited densified matrix. The addition of 1.0 wt.% Mg and 1.0 wt. % Sn lead to the formation of dense aluminum matrix with higher mechanical properties.

The sintering additives introduce liquid phase sintering which improved sintering respond of porous Al. However, there is limited literature available of Sn with Mg addition in porous Al fabrication. Besides, most of the porous Al fabrication are confined to the use of 1.0 wt.% Mg with 1.0 wt.% Sn and no attempt has been made so far to examine a large range of Mg and Sn contents and the influence of Sn content on sintering response and mechanical properties of porous Al. The weight percent of Sn added into the porous Al is believed to vary the amount of liquid presence during sintering process.

Energy absorption is one of the important properties of porous Al. Energy absorption of porous materials can be characterized by the means of energy absorption capability (W), energy absorption efficiency (E), and ideal energy absorption efficiency (I). However, most of the previous studies on porous Al are focused only on energy absorption capability (W). Chen et al. (2012) studied the effect of silicon carbide (SiC) additions on energy absorption properties of porous Mg alloy using energy absorption capability (W), energy absorption efficiency (E), and ideal energy absorption efficiency (I). They found that, energy absorption efficiency (E) and ideal energy absorption efficiency (I) are can be used to determine the the best energy absorber. In addition, Xie et al. (2017) who investigated the effect of calcium chloride ($CaCl_2$) as space holder on energy absorption properties of porous titanium (Ti) mentioned that the energy absorption efficiency (E) and ideal energy absorption efficiency (I) are useful to determine the suitable porous materials for different applications. For example, porous materials with highest and flatter energy absorption efficiency (E) curve have better cushioning properties. Whereas, porous materials which have constant and flat ideal energy absorption efficiency curve (I) represent uniform deformation upon force which is suitable for energy absorption applications. In this study, the energy absorption capability (W), energy absorption efficiency (E), and ideal energy absorption efficiency (I) of porous Al were evaluated.

1.3 Research objectives

The main objectives for this research are:

i. To determine the effect of different weight percent of carbamide (20, 40 and 60 wt.%) on the morphology, physical properties and mechanical properties of porous Al-1.0Mg.
ii. To investigate the effect of different weight percent of magnesium (0.2, 0.6, 1.0, 1.2, 1.6 wt.%) on the sintering respond and compressive properties of porous Al.
iii. To determine the effect of different weight percent of Sn (0.2, 0.6, 1.0, 1.2, 1.6, 2.0, 2.4, 3.0 and 4.0 wt.%) as sintering additives on sintering respond and compressive properties of porous Al-1.0Mg.

1.4 Thesis outline

The chapters of this thesis are summarized as follows:

Chapter 1 provides an overview and background of the study, problem statements and objectives of the research. Chapter 2 covers literature review of works related to porous Al. A brief introduction into porous materials and porous Al, fabrication methods, various sintering additives, type of sintering and properties of porous Al were discussed in Chapter 2. The research methodology in detailed from raw materials preparation to fabrication of porous Al, parameters investigated and characterization methods involved are described in Chapter 3. Chapter 4 presents a systematic results and discussion of the works which include the raw materials characterization, the effect of carbamide amounts, the effect of various amounts of Mg addition and the effect of various amounts of Sn on sintering respond and mechanical properties of porous Al-1.0Mg. The conclusions and recommendations for future work in porous Al fabrication are presented in Chapter 5.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

Natural cellular materials are common materials present in nature such as wood, cork, coral and bone (Cadena et al., 2016; Oana et al., 2013). Meanwhile man-made cellular materials are materials with pores intentionally integrated in their structure (Lefebvre et al., 2008). Man-made cellular materials can be produced from multiple choices of materials such as ceramics, polymers and metals. The pores made the cellular materials lightweight and have high specific strength and stiffness relative to its weight. Different production methods of porous Al have been developed to produce porous Al for specific applications either structural or functional. This study was intended to focus on porous metal and investigate the basic properties and performance of porous Al for better structural application.

2.2 Porous metals

Porous metals are defined as materials consisting of a solid metal containing a large volume fraction of gas filled pores (Kennedy, 2012). Porous metals are in great concern due to their unique properties derived from the combinations of porous structure and metallic behaviour (Banhart & Baumeister, 1998). Compared to the solid metal, porous metals possesses a high specific strength and stiffness relative to its weight, high energy absorption capability, high thermal and electrical conductivity (Ashby et al., 2002; Ramírez et al., 2014; Surace et al., 2009; Smith et al., 2012). Besides, these