SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING UNIVERSITI SAINS MALAYSIA

EFFECT OF COMPACTION PRESSURE AND ADDITION OF SILICON ON MICROSTRUCTURE AND COMPRESSIVE PROPERTIES OF POROUS MAGNESIUM

By

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled " Effect of compaction pressure and addition of Silicon on microstructure, physical and compressive properties of porous Magnesium". I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or University.

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Table of contents

Contents	**Page**

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Ar	Argon
C	Carbon
C2H4	Ethylene
CH3COOH	Ethanoic acid
CH4	Methane
CO	Carbon monoxide
CO_2	Carbon Dioxide
DSC	Differential scanning calorimetry
E	Energy absorption efficiency
f_{Mg}	Magnesium weight fraction
FCC	Face-centered cubic
GASAR	Gas-metal eutectic solidification
H_2	Hydrogen
HCP	Hexagonal close-packed
I	Ideal energy absorption efficiency
Mg	Magnesium
$Mg(Cl)_2$	Magnesium chloride
$Mg(OH)_2$	Magnesium hydroxide
MgO	Magnesium oxide
MMA	Methyl methacrylate
PMMA	Poly (methyl methacrylate)
SEM	Scanning electron microscope
Si	Silicon

TGA	Thermogravimetric analysis
W	Energy absorption per unit volume
Wa	Weight of porous Mg in air
Wb	Weight of porous Mg in water
Wc	Weight of porous Mg after immersed in distilled water
XRD	X-ray diffraction analysis
σ	Compressive stress
σm	Maximum stress
ε	Compressive strain.
εm	Maximum strain

LIST OF SYMBOLS

atm	Standard atmosphere
°C	Degree Celsius
g/cm^3	Gram per cubic centimeter
MJ/m^3	Mega Joule per meter cubic
MPa	Mega Pascal
mm	Millimetre
nm	Nanometer
µm	Micrometer
vol.%	Volume percent
wt.%	Weight percent
%	Percent
ρ	Density

KESAN TEKANAN MAMPATAN DAN PENAMBAHAN SILIKON PADA MIKROSTRUKTUR DAN SIFAT MAMPATAN BUSA MAGNESIUM BERLIANG

ABSTRAK

Magnesium (Mg) berliang telah diiktiraf sebagai bahan struktur untuk industri automotif dan aeroangkasa kerana ringan yang membawa kepada reka bentuk yang sangat jimat minyak. Eksperimen terbahagi kepada dua bahagian. Dalam bahagian pertama, Mg berliang telah difabrikasi melalui metalurgi serbuk menggunakan PMMA sebagai sebagai agen pembusaan.Tekanan pemadatan telah digunakan dari 200, 250, 300, 350 hingga 400 MPa dan disinter melalui proses pensinteran dua tahap pada suhu pensinteran 620°C. Ketumpatan Mg berliang meningkat dan keliangan berkurang dengan peningkatan tekanan pemadatan. Pencirian mekanikal menunjukkan bahawa Mg dipadat pada tekanan 400 MPa mempamerkan kekuatan mampatan, kekuatan alah mampatan yang tertinggi dan menunjukkan ideal kecekapan penyerapan tenaga yang paling optimum. Untuk bahagian kedua, kesan penambahan silikon kepada Mg berliang telah dikaji. Mg, Si dan serbuk PMMA dicampurkan di dalam nisbah 78:2:20, 76:4:20, 74:6:20, 72:8:20, 70:10:20 dan 65:15:20, dipadatkan pada 400 MPa dan disinter pada 450 °C. Tegasan alah mampatan Mg berliang pada 400 MPa adalah 33.65 MPa manakala tegasan alah mampatan yang paling tinggi bagi Mg-Si berliang pada 400 MPa adalah 36.89 MPa. Selain itu, Mg berliang mempunyai purata tenaga ideal penyerapan kecekapan 1,571 manakala Mg-Si berliang mempunyai purata ideal kecekapan penyerapan tenaga 2,261 pada 2 bt.% silikon. Secara ringkas, penambahan silikon mempunyai kesan yang besar ke atas sifat-sifat Mg berliang yang meningkatkan kekuatan alah mampatan Mg berliang dan tenaga ideal kecekapan penyerapan.

EFFECT OF COMPACTION PRESSURE AND SILICON ADDITION ON MICROSTRUCTURE AND COMPRESSIVE PROPERTIES OF POROUS MAGNESIUM

ABSTRACT

Porous Magnesium has been recognized as a promising structural material for automotive and aerospace industry due to light-weight which leads to highly fuel-efficient design. The experiment was divided into two parts. In the first part, porous Mg was fabricated by powder metallurgy using Poly(Methyl Methacrylate) (PMMA) as a space holder. Compaction pressure was varied from 200, 250, 300, 350 to 400 MPa. The green body was sintered using double sintering profile at temperature of 620°C. The porous Mg fabricated was then characterized for morphology, porosity, density, phases, compressive strength and energy absorption properties. Density of porous Mg increases, and the porosity decreases with increasing compaction pressure. The mechanical characterization indicated that porous Mg compacted at 400 MPa exhibited the highest compressive strength, compressive yield stresss and showed the most optimum ideal energy absorption efficiency. For second part, effect of addition silicon to porous Mg was studied. Mg, Si and PMMA powders are mixed in ratio of 78:2:20, 76:4:20, 74:6:20, 72:8:20, 70:10:20 and the 65:15:20, compacted at 400 MPa and sintered at 450°C. The compressive yield stress of porous Mg under 400 MPa is 33.65 MPa but the highest compressive yield stress of porous Mg-Si is 36.89 MPa. Porous Mg has the average ideal energy absorption efficiency 1.571 while porous Mg-Si have average ideal energy absorption efficiency of 2.261 at 2 wt.% of silicon. Addition of silicon has significant effect on the properties of porous Mg which increase the compressive yield strength of the porous Mg and ideal energy absorption efficiency.

CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

Foams are a class of highly porous materials with a cellular structure that possess a combination of interesting properties such as very low specific weight, high stiffness and good thermal conductivity. Commonly, porous material are organic materials and polymeric foams but there also inorganic porous materials such as porous metals have been developed for the purpose like insulation, cushioning, impact protection, catalysis, membranes, construction materials. Porous materials are the solid materials containing pores.

Structural defect of porous materials is presence of pores or cavities for traditional engineering materials because these pores will cause crack initiation and propagation,. This will lead to unfavourable impact to the mechanical properties of materials. However, presence of cavities brings benefits for porous materials such as the increase in the number of cavities to a certain extent will lead to the formation of distinctive properties. For example, it will lead to higher energy absorption, heat insulation, noise reduction and filtration in which these features make porous materials can be used in various applications. Porous materials are get great attention in several high impact sectors such as aerospace field, automotive industy and especially biomedical applications due to their lightweight structure, mechanical endurance, and biomimetic properties.

Recently, metallic foams (especially for Mg/Mg alloy foams) have attracted more and more attention. Magnesium is the lightest of all the metals, which is 35% lighter than aluminium and 4 times lighter than steel. Thus, they are draw an attention in automotive and aerospace industry for their inherent light-weight which can use to built a highly fuel save design. Magnesium not only low density, it also has high specific strength (related to low density); good castability (particularly for high pressure die casting); good weldability; and enhanced corrosion resistance.

In automotive applications, there are an estimation about 5% reduction of fuel usage with 10% reduction of weight of the vehicles. This can help to save the energy efficiencies. Thus, there is large potential for alloyed magnesium and porous magnesium used for automotive applications. Besides those advantages of magnesium, energy absorption properties of porous magnesium can be used to absorb impact. Same to aerospace industry, weight reduction is also a popular study in aerospace industry and weight reduction also can save usage of fuel and indirectly save cost. Common structural material used in aerospace industry is aluminium. However, research teams never stopped finding a better structural material to achieve most effective applications. For example, structural plastics and non-metallic materials could meet the light weight requirements but withdraw of these materials are they are non-electrical conductivity, low impact characteristics and etc, which make them not be able to be used in advance applications.Magnesium is the lightest structural metallic material, which is an ideal alternative to be used in the aerospace industry (Xu and Li, 2017).

Porous Mg can be fabricated with several methods which can be classified according to the state of the starting Mg either melts or powder. The most common methods using

Mg melts as starting materials includes melt gas injection, melt foaming agent and investment casting. However, the fabrication methods mention produces porous Mg with irregular cell size and uncontrolled pores distribution, high costs and complex process respectively.

Among all the fabrication method, powder metallurgy using space holder technique has exhibited the best method for porous Mg fabrication according to researcher (Čapek and Vojtěch, 2013). It utilizes space holder materials to produce porous Mg with interconnected pores. In addition, parameters such as pores size, pores distribution and porosity of product can be easily controlled by controlling the size and shape of starting powders (Čapek and Vojtěch, 2013). Thermal decomposition and leaching process was used to remove the space holder materials during sintering process or before sintering process. There are variety of space holder material used by current researcher includes sodium chloride (NaCl), carbamide, ammonium bicarbonate and Poly (methyl methacrylate) (PMMA).

In fabrication porous metal, the most difficult part is to obtain a good pore distribution and porosity required by the porous metal. Good distribution pores are the main factor since the properties of foam is depending on their structure. Homogeneous distributions of powder and space holder need to achieve during the milling process and compaction process in order to have uniform distributions of powder and space holder and porosity required by the porous metal. Compaction pressure and sintering process is important to ensure the compaction of powder particle and space holder remove completely to form magnesium foam with high porosity and density.

1.2 PROBLEM STATEMENT

Pure magnesium has low elastic modulus; limited cold workability, toughness; limited high strength and creep resistance at high temperatures and high chemical reactivity and in some applications limited corrosion resistance which restricted usage in automotive and aerospace applications. Thus, alloying of magnesium or adding reinforcement particles are needed to enhance the mechanical properties. In order to increase the strength of magnesium foam, several ceramic reinforcements were introduced such as Al_2O_3, SiC and CNT. However, there is a disadvantgaes of addition of reinforments, that is the interfacial reactions between the reinforcements and the matrix and poor wettability between the reinforcements and the matrix.

Moreover, reinforcing phase is constricted by the starting powder size, which is typically of the order of microns to tens of microns and rarely below 1 μm. Studied conducted by (Yong and Li, 2012), indicated that addition of silicon to magnesium alloy could substantially improve the strength of the alloy, increase the fluidity of the molten metal. Addition of Si may cause the formation of Mg_2Si phase which has high melting temperature, low density, high elastic modulus and low thermal expansion coefficient. This process can be applied to porous Mg to enhance the strength and overcome the drawback caused by reinforcement. Thus, present work was initiated to fabricate porous Mg-Si via in situ process to yield a good mechanical properties.

1.3 RESEARCH OBJECTIVE

The objectives of this research are:

I. To study the effect of different compaction pressure on mechanical properties, compressive strength of porous Magnesium

II. To study the effect of addition of silicon on microstructure and mechanical properties of porous magnesium.

1.4 THESIS OUTLINE

The chapters of this thesis are summarized as follows:

i. Chapter 1 provides an overview and background of the study, problem statement and objectives of the research.

ii. Chapter 2 covers literature review of works related to porous magnesium. This include classification of porous material, application of porous magnesium, available fabrication methods of porous magnesium, double sintering process and as well as the properties of porous magnesium.

iii. Chapter 3 describes the research methodology in detail from raw materials preparation to fabrication of porous magnesium, parameters conducted, and characterization involved.

iv. Chapter 4 presents a systematic results and discussion of the works which include raw materials characterization and the effect of different compaction pressure and addition of silicon into porous Magnesium on morphology, mechanical properties, and physical properties

v. Chapter 5 presents the overall conclusions of the research and recommendations for future work in porous magnesium fabrication.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Natural cellular materials are common materials which present in nature such as wood, cork, coral and bone. Meanwhile man-made cellular materials are materials with pores intentionally integrated in their structure. 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. Among the porous material, porous metal has a higher melting point, high-temperature stability, better weather fastness and harder ageing.

There are many benefits of the magnesium metals, such as specific strength, stiffness, high modulus elastic, good vibration which reduce performance and has been attractive to aerospace and transport industry. With these light materials incorporated in transport and aerospace fields, it can help in control environment pollution and reduction of fuel consumption which indirectly save costs. Different production methods of porous Mg have been developed to produce porous Mg 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 Mg for better structural application.

2.2 POROUS MATERIALS

Traditionally, porous materials are organic materials and porous polymeric materials. However, inorganic porous materials such as porous metals have been developed for the purpose like insulation, cushioning, impact protection, catalysis, membranes, structural materials. Porous materials are the solid materials containing pores. Structural defect of porous materials is presence of pores or cavities for traditional engineering materials because these pores will cause crack initiation and propagation, which have the unfavourable impacts on the mechanical properties of materials. However, presence of cavities brings benefits for porous materials such as the increase in the number of cavities to a certain extent will lead to the formation of distinctive proprties. For example, it will lead to higher energy absorption, heat insulation, noise reduction and filtration in which these features make porous materials can be used in various applications. Porous materials are becoming famous in several high impact sectors such aerospace, automotive especially in biomedical fields due to their lightweight structure, mechanical endurance, and biomimetic properties.

2.3 POROUS METAL

Porous metal/ metal foam is a cellular structure made up of a solid metal containing a large volume fraction of gas containing pores. These pores can be classified as closed-cell foam and open-cell foam. In an open cell, the cells are interconnected, forming continuous network of metallic struts. Whereas in closed cell, the cells are disconnected and cell walls will separate the pores. Porous metals with open cell structure are weaker and are mostly used in functional applications such as filtration, separation, heat or mass exchanger (Jiang et al. 2005). While, closed cell porous metals have good strength and mainly used in structural applications (Mohammed 2016).

Porous metals have a combination of properties which caused them famous use in many engineering applications. (Miao and Rabiei, 2018) They offer lower weight than conventional honeycomb. Hence it can be used in structural sandwich panel for some, although not all, loading configurations. Besides that, porous metal also provides significant energy absorption without generating damaging peak stresses as the capacity of porous metal undergo large strains at almost constant stress allows, this allowed them to be used as an energy-absorption devices. Due to their high thermal conductivity, high internal surface area, and interconnected pores, it can be used as heat dissipation devices.

Due to presence of structural defects, porous metals has many attractive properties, such as high impact energy absorption, outstanding physical and thermal characteristics. Due to their high strength to weight ratio and excellent energy absorption, porous metal can be used safety aspect such safety of passenger in automotive field. (Miao and Rabiei, 2018). Besides that, porous metals also can contribute its features in solar energy system. Fossil fuels must be replaced by renewable energy sources such as solar energy as fossil fuels is non-renewable sources. In such systems, high-performance is one of the essential needs. Porous materials have been introduced as one of the most efficient and affordable techniques to improve the heat transfer and energy efficiency in solar energy systems. For instance, porous silicon (PSi) has get great attention as material for solar cell and sensor applications due to it has a large surface area to volume ratio, pore sizes can be easily control, suitable surface chemistry, and the able to adjust refractive index as a function of depth. There are various types of porous metals used in industry such as steel (Costanza et al. 2016), titanium (Xie et al. 2017), magnesium (Li et al. 2016) , aluminium (Hassani et al. 2012) and copper (Eid et al. 2017).

2.3.1 Types of porous metals

Many studies have been done on development of porous metals. For instance, Costanza et al. (2016) who investigated the effect of urea space holder amount on energy absorption properties of iron porous and it was found that the increases amount of urea space holder increases the total porosity of porous iron resulting in low density and low plateau stress. Porous iron with a porosity up to 82 vol.% were successfully prepared using ammonium bicarbonate as a space holder material by Čapek et al. (2015) which show that finer iron powder have enhanced the mechanical properties of porous iron. Porous iron has been examined as an option to porous Al, because steel has better strength, better energy absorption capacity and cheaper than Al. However, iron is denser than Al and its much higher melting temperature makes a low cost production of porous iron a challenge (Lefebvre et al. 2008). Xie et al. (2017) studied the energy absorption properties of porous titanium fabricated using calcium chloride as space holder. The porous titanium having porosity between 71 to 89% reported to have plateau stress 18.1–105.6 MPa. At the same time, porous iron, titanium and magnesium have been extensively studied as biocompatible and biodegradable porous materials for orthopaedic applications (Čapek et al. 2015). Among the porous metals available, porous Mg has density of 1.74 g/cm^3 which is the lightest metal in industrial applications. Porous magnesium can absorb energy because of its specific structure.

2.4 POROUS MAGNESIUM

Magnesium is the seventh most abundant element in the Earth's crust with a average of 2.76% (Fyfe 1999). Its chemistry is intermediate between that of Be and the heavier alkali earth elements. Table 2.1 shows the physical and mechanical properties of magnesium. Magnesium is the lightest of all light metal alloys, strong, has good heat dissipation and abundant in earth which can be used extensively for engineering applications when weight is a critical design element. The use of pure magnesium is not common, it always alloyed with other metals to improve its properties. Besides that, Magnesium is very volatility at elevated temperatures and very corrosive in wet environments conditions. Therefore, the use of magnesium alloys when designing aerospace and automotive parts is critical. Besides that, nowadays, magnesium alloys are a subject of intensive research and development for applications in medicine as an osteosynthetic material. These materials able be easily decomposed and be absorbed into a human body. The main advantages of these kinds of implants would be reducing the number of surgeries (Salvetr et al., 2016).

Table 2. 1: Physical and mechanical properties of Magnesium (Nguyen,2011)

Physical Properties	**Value**	**Mechanical properties**	**Value**
Colour	Grey	**Tensile strength**	90-220MPa
Phase at STP	solid	**Compressive yield strength**	21-115 MPa

Melting point	923 K (650 °C, 1202 °F)	**Elongation**	2-15%
Boiling point	1363 K (1091 °C, 1994 °F)	**Young’s Modulus**	45GPa
Density (near r.t.)	1.738 g/cm^3	**Specific Strength**	158 kN×m/kg
when liquid (at m.p.)	1.584 g/cm^3	**Shear modulus**	17GPa
Heat of fusion	8.48 kJ/mol	**Mohs Hardness**	1-2.5
Heat of vaporization	128 kJ/mol	**Brinell hardness**	44-260MPa
Molar heat capacity	24.869 J/(mol·K	**Bulk modulus**	35.4GPa

Magnesium and its alloys have low density and high specific strength. With these properties, they can be apply in lightweight structural applications like aerospace industry (Mordike and Ebert, 2018). However, their poor formability at room temperature limited the use magnesium as a structural. The formability of magnesium is related to magnesium’s ductility which affected by it crystal structure. Mg has a hexagonal close-packed (HCP) structure and it is not as ductile as the face centred cubic (FCC) metals such as aluminium and silver. Number of operative slip systems determine the ductility of the porous Magnesium. Mg being hexagonal slips at room temperature on the base plane (0001) <11$\bar{2}$0>and secondary slip on vertical face planes (10$\bar{1}$0> in the <11$\bar{2}$0> direction. This means that ductility of magnesium is small at low temperatures. At high temperatures slip also occurs in the <11$\bar{2}$0> direction on the (10$\bar{1}$1) pyramidal planes. This deformation behaviour is also influenced by alloying, but if the structure remains hexagonal the effect is limited (Mordike and Ebert, 2018).

2.5 METAL MATRIX COMPOSITE

The increasing in demand of light and high-performance materials especially in automotive industry to save costs and fuel consumption, magnesium based MMCs is getting pay attention. For example, Mg-Al system is the good performance engineering material due to its light structure. Thus, it is excellent candidates in civic, military and aerospace applications. The potential applications of magnesium–matrix composites in the automotive industry such as disk rotors, piston ring grooves, gears, and connecting rods.

However, cost for fabrication magnesium-based composite is very expensive and its fabrication route is very complicated. It can be solved by using low cost materials which can provide a low costs route for synthesis magnesium-based composite. Various types of reinforcement materials and processing techniques are created to help in produce this class of materials. For example, the magnesium-based MMCs unidirectionally reinforced with continuous carbon fibre can readily show a bending strength of 1000 MPa with a density as low as 1.8 g /cm^3(Capelet al., 2000; Ottingeret al., 1995; Hausmannet al., 1998). The superior mechanical property can be retained at elevated temperatures of up to 350–4001C (Kagawaand Nakata, 1992; Diwanji and Hall, 1992; Ottingeret al., 1997).

Traditionally, discontinuously reinforced MMCs have been produced by several processing routes such as powder metallurgy, spray deposition, mechanical alloying (MA) and various casting techniques. Addition of ceramic reinforcements to the matrix materials either in molten or powder form is found in these techniques mentioned above. For the common MMCs, the reinforment are prepared separately before composite fabrication. This kind of MMCs fabrication is known as ex situ MMCs. In this fabrication route, reinforcment is constricted by the starting powder size, which is typically of the order of microns to tens of microns and rarely below 1 µm. Other main drawbacks that must be overcome are the interfacial reactions between the reinforcements and the matrix, and poor wettability between the reinforcements and the matrix due to surface contamination found on the reinforments (Tjong and Ma, 2000).

Properties of MMCs are governed by the size and volume fraction of the reinforcements and wettability between the reinforcements and the matrix. An ideal mechanical properties can be obtained when fine particulates are distributed uniformly in the metal matrix. This problem has led to the development of in situ MMCs in which the reinforcements are formed in a metallic matrix by chemical reactions between elements or between element and compound during the composite fabrication. Compared to the common MMCs produced by ex situ methods, the in situ MMCs exhibit the following advantages: (a) the in situ formed reinforcements are thermodynamically stable at the matrix and can withstand in high temperature services; (b) form strong interfacial bonding; (c) the in situ formed reinforcing phase are finer in size and distributed homogenously in matrix, create a better mechanical properties.

Using these routes, in situ MMCs with a wide range of matrix materials such aluminium, titanium and magnesium and second-phase particles like borides, carbides and nitrides have been produced. However, common processing features of in situ MMCs such as fabrication routes are not well understand and established.

2.5.1 Magnesium Composite Foam

Composite metal foam is a material which combination of metal and other material. The compositions of other material function act as reinforcement to the structure. By having composite metal foam, the strength and mechanical properties of composite metal foam can be increased due to strengthening and reinforcing phase method. According to Shen et al. (2016) magnesium composite have a great potential for application in aerospace, automobile, and military industries due to their low density, high specific strength and stiffness. To increase the properties of metal foam, ceramic particle can be added. The ceramic particles enhance the brittleness of metal foam and increase their strength. Moreover, when the ceramic particle added it can provide foam stability and homogeneity of cellular structure without causing the structural defects. Reinforcing materials that commonly used are alumina, silicon carbide, silicon oxide and carbon nanotubes (Banhart, 2001). This reinforcing material has the high temperature stability, high hardness and high modulus which contribute better properties of composite metal foam.

2.6 FABRICATION OF METALLIC POROUS STRUCTURES

The first metallic foam was produced by Sosnik in 1948. He creates the pores by putting mercury into molten aluminium (Sosnik ,1948). In 1956, mercury is replaced by foaming agents which will generates by thermal decomposition and until now, many researchers have developed metallic foam using this technique without the toxicity of mercury (Elliot,1956) Then in 1963, powder-compact foaming technique to manufacture metallic foams is created by Allen (Allen ,1963) Metal foams and porous metals are fabricated by the following processes.

2.6.1 Melt Gas Injection (Air Bubbling)

Metal foam cant be formed by bubbling a gas into the pure liquid metal It can be done by dischargeof liquid down the walls of the bubbles, it usually occurs too quickly to create a foam that remains stable long and able to solidify. However, in some case there are some insoluble or slowly dissolving particles such as aluminium oxide, it will raise the viscosity of the aluminium melt to a higher level. This means it will impede the drainage of aluminium melt down the wall of the bubbles and impede the stabilising of the foam. Thus, metal gas injection process is introduced and easiest to implement with aluminium and magnesium alloys as theirs's low density and do not excessively oxidised when the melt is exposed to air. Bubbles formed inside the liquid aluminium and magnesium melt can be done by variety gases. Bubbles formed by this process float to the melt surface, drain, and then start to solidify. This technique is the cheapest to apply and the foam formed with relative density in the range 0.03–0.1 (Ashby et al., 2001). Figure 2.1 shows schematic diagram process of the manufacturing system using melt gas injection

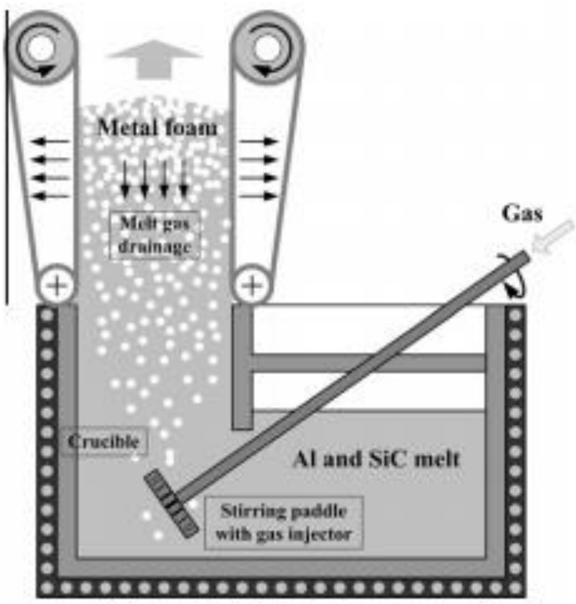

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Figure 2. 1: Schematic of the manufacturing system based on the melt gas injection method of porous Al (Ashby et al., 2001)

2.6.2 Melt Foaming Method

Melt foaming process is a cost-effective method to produce porous metallic structure. It is different from the metal gas injection method as there is no bubbling of air or gas into the melt. The method utilized the blowing agent to produce porous metal foam with closed pore structures. Under influences of heat, the foaming agent has decomposed and release gas which will responsible for the pore formation and initiate the foaming process. The most widely used foaming agent is titanium hydride (TiH_2) which decomposed in to Ti and hydrogen gas, H_2 when heated at temperature above 738K. A huge volumes of hydrogen gas can be quickly generated when addition of titanium hydride particles to aluminium melt. This will creating bubbles that can lead to a closed-cell foam, provided foam drainage is sufficiently slow, which requires a high-melt viscosity **(**Ashby et al., 2001). The cell size can be varied from 0.5 to 5 mm by altering the TiH2 content and its foaming and cooling conditions. With this method, relative densities of foam can be obtained from 0.2 to 0.07.

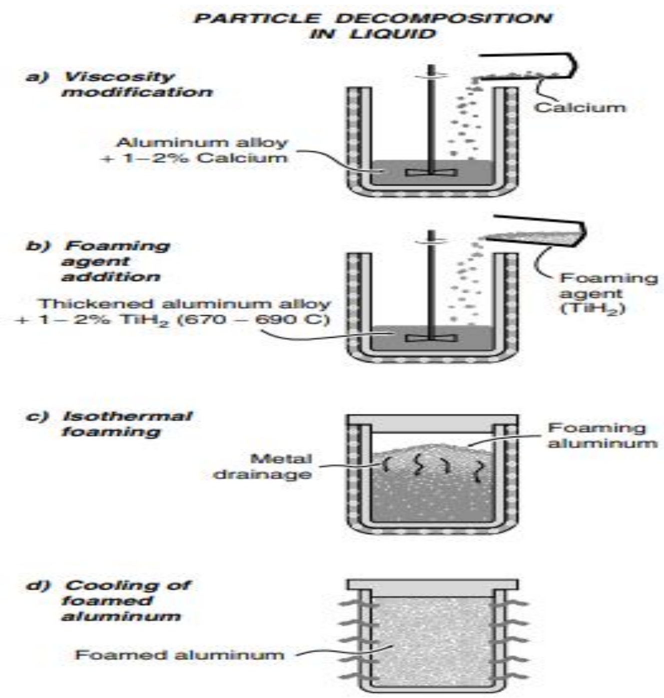

Figure 2.2: The process steps used in the manufacture of aluminium foams by gas-releasing particle decomposition in the melt (Alporas process) (Ashby et al., 2001)

2.6.3 Gas-Metal Eutectic Solidification

Gas–metal eutectic solidification can be used in metal alloy that have metal alloy–hydrogen binary phase diagrams that exhibit a eutectic. For example, Al, Be, Cr, Cu, Fe, Mg, Mn and Ni-based alloys. This process started with the metal alloy melted and saturated with hydrogen gas under pressure and then directionally solidified and progressively reduce the pressure. Solid metal and hydrogen form a gas eutectic reaction upon solidification which resulting a porous metallic structure with hydrogen filled inside the pores. This process is known as GASARs process (Nakajima, 2013). A schematic diagram of the basic approach is shown in Figure 2.3. Suitable pressure of hydrogen is used to melt the metal alloy in a furnace inside a pressure vessel. A directional solidification process happened by discharging the melt into the mould. This results in an object containing a reasonably large (up to 30%) volume fraction of pores. The pore volume fraction and pore orientation are depending on alloy chemistry, melt over-pressure, melt superheat (which affects the hydrogen solubility of the liquid metal), the temperature field in the liquid during solidification, and the rate of solidification. The method poses certain safety issues, and in its present form is a batch process. As a result, materials manufactured by this route are costly, but this process can produce a highly porous metallic structure which can function in various types of application, but it still not widely used as it is very difficult to manipulate the parameters and possess certain safety issues.

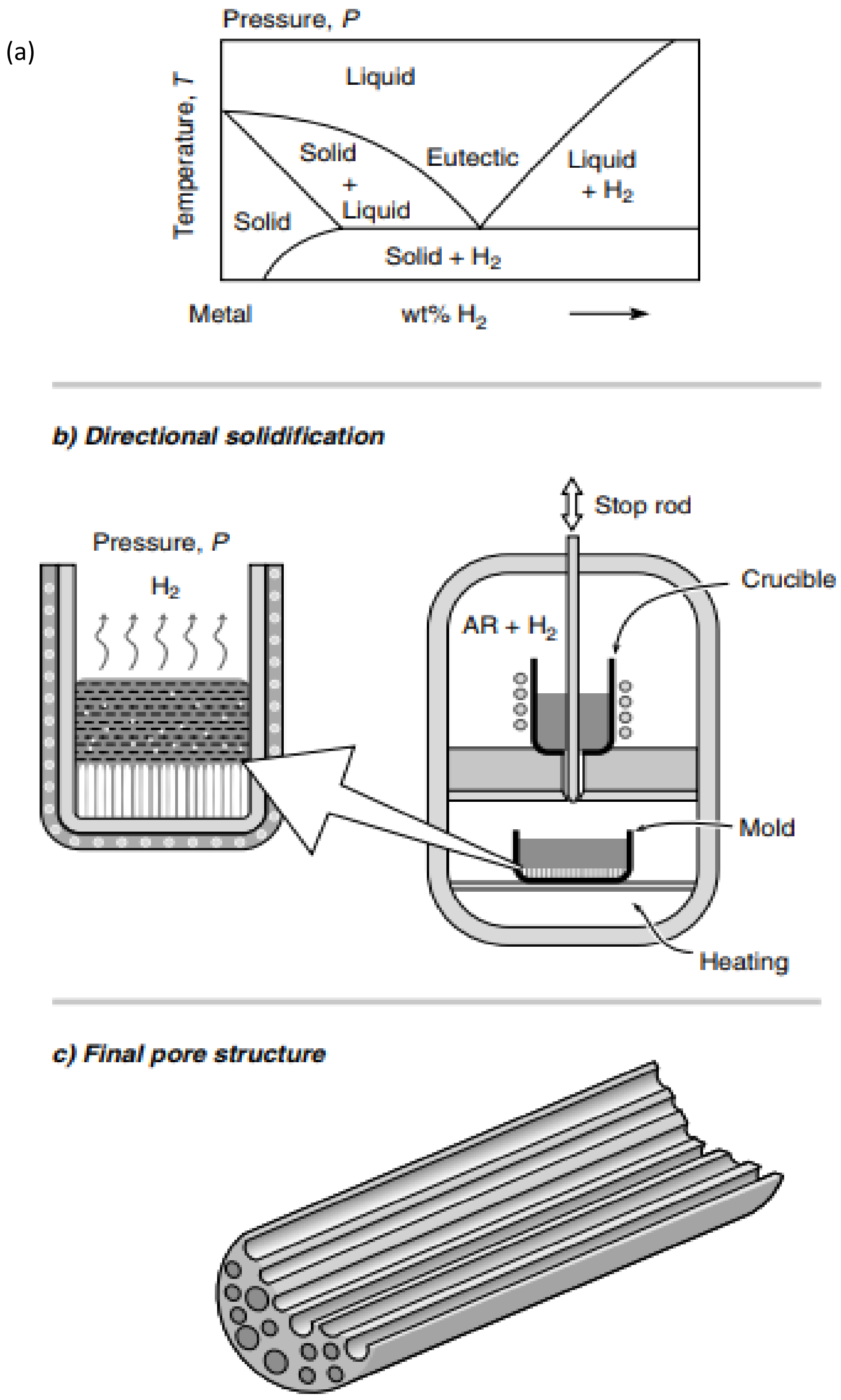

Figure 2.3: Gas–metal eutectic solidification for the manufacture of GASAR (a) Metal-hydrogen binary phase diagram (b) Directional solidification technique and (c) Final pore structure of metal foam (Nakajima, 2013)

2.6.4 Casting using a polymer or wax precursor as template

Open cell polymer foams can be used as templates to produce investment-casting molds. Variety of metal and their alloys can be casted by this method. Essentially, an open-cell polymer foam mold template with the required cell size and relative density is first choosed. This can be coated with a mold casting (ceramic powder) slurry which is then dried and embedded in casting sand. The mold is then baked both to harden the casting material. It is also caused the decomposition of polymer template and leaving behind a negative image of the foam. The metal alloy is the filled up the mold and allowed to be cooled. During melt infiltration, some liquid alloys are resist to flow, it can be solved by apply of moderate pressure. After directional solidification and the mold materials are get ridand leaved behind the metal equivalent of the original polymer foam. Metal powder slurries can also be used instead of liquid metals. The method gives open-cell foams with pore sizes of 1–5 mm and relative densities as low as 0.05. The process can be used to manufacture foams from almost any metal that can be investment cast. In a variant of the process, the precursor structure is assembled from injection-molded polymeric or wax lattices. A negative image mold can be formed by coating the lattice structure with casting slurry and then fired. Metals able cast into the mold utilising the conventional investment casting techniques **(**Ashby et al., 2001).

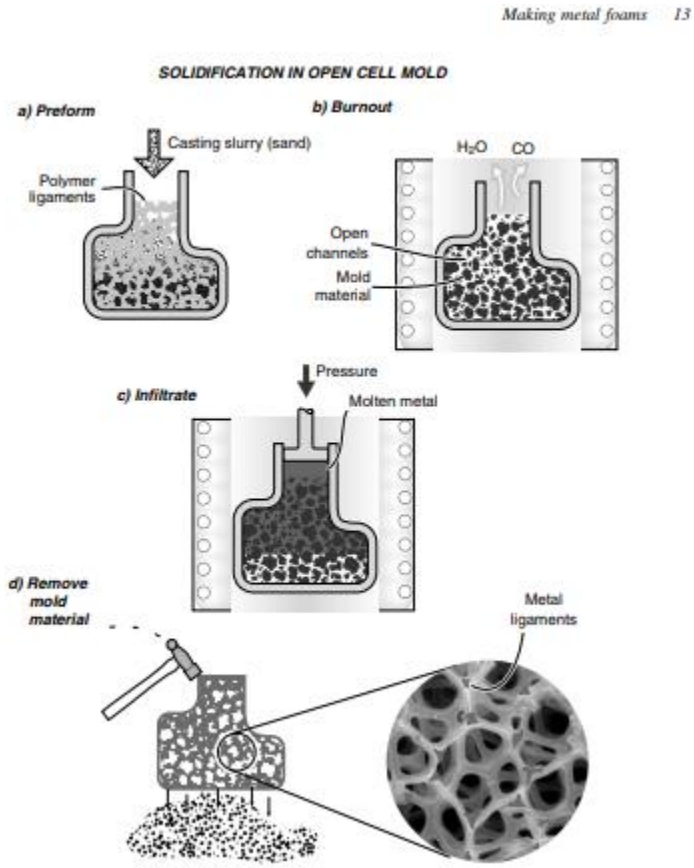

Figure 2.4: Investment casting method used to manufacture open cell foams (DUOCEL process (Ashby et al., 2001)

2.6.5 Powder Metallurgy

Powder metallurgy is a promising technique that utilized space-holder particles to fabricate the porous materials with interconnected pores.By modifying the pore size and distribution, mechanical properties of the material can be changed. Changing of pore size and distribution can affect the mechanical properties of the material and this can be done by adjusting the size and shape distibution of the powder in raw materials and its condition like sintering duration, temperature and compacting pressure. Figure 2.5 shows the fabrication of metal foam using powder metallurgy method.

Space holder technique consist of four major steps: mixing, pressing, space holder removal process and sintering as indicated in Figure 2. 5. Firstly, the space holder material such as carbamide particles ($CO(NH_2)_2$ that act as pore former are mixed and compacted together with metallic matrix powder. After that, the space holder particles are removed either before or during sintering, forming new pores in the metal matrix.

According to Hao et al., (2007), porous magnesium can be fabricated using two steps sintering. Sintering of porous magnesium was done under high purity argon atmosphere at 250°C for 3 hours to remove residual of carbamide by melting/decomposing it. Then the sample was further sintered at 630°C for 2.5 hours in order to reach metallurgical bonding among the Mg powder. Another study conducted by Bi et al. (2015) has sintered the green compact of magnesium at 400°C for 2 hours in in a Vacuum furnace under the pressure lower than 8×10^{-3} MPa with a rate of 3°C $minutes^{-1}$. Then, the green compact was fired to 550°C for 2 hours in high-purity argon with heating rate of 15°C/minutes. Lastly the sintered sample was cooled to room temperature in high purity argon.

In addition, selection of space holder material is very crucial. This is because any reaction between decomposed space holder material and metal matrix may deteriorate the mechanical properties of the resulting product (Kim et al., 2013). To minimize the negative effects on the resultant product due to contamination by the residues of space-holding particles, variety of space holders had been used by most of researchers to produce porous magnesium. This include sodium chloride (Kang et al., 2013), ammonium bicarbonate (Čapek and Vojtěch, 2013, Kowalski and Jurczyk, 2015), carbamide (Wen et al., 2001, Zhuang et al., 2008) and Poly(methyl methacrylate) (Bi et al., 2015). In this work, PMMA is choose as space holder due to very worse affinity with Mg as compared to ammonium bicarbonate and carbamide (Bi et al., 2015).

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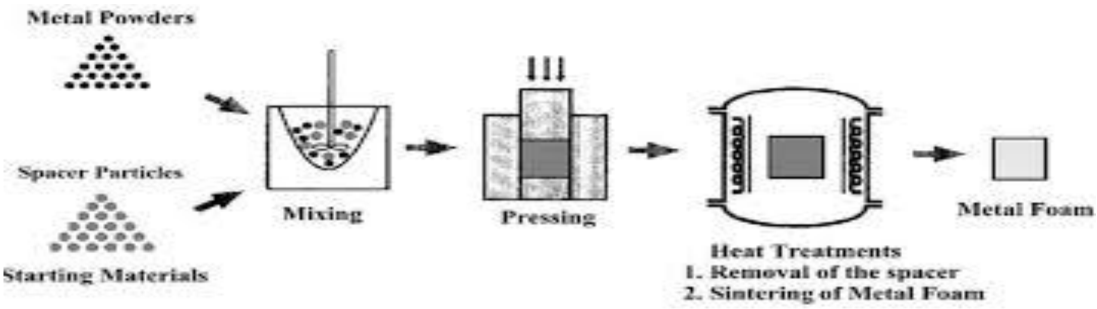

Figure 2.5: Schematic illustration of fabrication route of metallic foam with the space holder method (Arifvianto and Zhou, 2014)

2.7 Double Sintering Process

Double sintering process is one of the major step in space holder technique which divided into two process one is primary sintering and one is secondary or it is knowns as actual sintering. Primary sintering process is used to decompose the space holder material which occurred at low temperature than the secondary sintering process. Second sintering is used to enchance the strength of primary sintered foam. Parameters for sintering process must be set and controlled wisely as it will significantly affect the properties of end product.

2.7.1 Mixing process

Mixing of Mg powder with space holder material is the first process in porous Mg fabrication. Mixing process is needed to get a homogeneous mixture of two or more different materials. Homogeneity distribution of Mg powder and space holder is crucial to produce high quality porous Mg with uniform pore distribution. Hence, Mg powder and space holder must be thoroughly mixed before undergo compaction to prevent segregation and to maximize homogeneity. However, an excessive mixing was unnecessary as it may result in formation of fine powder and deformation of the powder. In addition, segregation may occur during mixing process due to huge different in size and density between Mg powder and space holder. This problem can be reduced by using appropriate binder. Binders will help to produce a sufficient binding strength between Mg particles and space holder particles and not reactive to both powders. At present, several binder have been used for the fabrication of porous magnesium, including ethanol (Kang et al., 2016, Yilong et al., 2016), hexane (Čapek and Vojtěch, 2014) and paraffin (Hao et al., 2009). In this work, a small amount of ethanol is sprayed on the PMMA during the mixing process to avoid segregation of magnesium and Poly (methyl methacrylate) (PMMA) powders. Mg powder, PMMA and silicon powder were mixed in polyethylene bottle for 1 hour utilizing alumina ball as milling media with weight ratio of powder to ball was 1:10.

2.7.2 Compaction process

Process of compacting metal powder in a die through the application of high pressures is known as compaction process. Typically, the tools are held in the vertical position with the punch tool forming the bottom of the cavity. The powder is then compacted into a shape and then ejected from the die cavity. The higher the compaction pressure, the higher the density of the green body. During compaction the powder particles rearrange themselves and filled the void between the loose powder particles which increase packing coordination. Compaction pressure is an important parameter in fabricating porous Mg. Applying too low compaction pressure resulting in insufficient strength of green compact. Whereas, by applying too high compaction