SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

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THE EFFECT OF CARBAMIDE PARTICLES SIZE ON MORPHOLOGY AND COMPRESSIVE PROPERTIES FOAM BASED MAGNESIUM

By

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DECLARATION

I hereby declare that I have conducted, complete the research work and written the dissertation entitled "**The Effect of Carbamide Particles Size on Magnesium and Magnesium Composite Foam**". 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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LIST OF ABBREVIATIONS

Al	Aluminum
Al_2O_3	Alumina
$CO(NH_2)_2$	Carbamide
˚C	Degree Celsius
$CaCo_3$	Calcium carbonate
CNT	Carbon nanotubes
DSC	Different scanning calorimetry
et al.	And others
g/cm^3	Grams/cubic centimetres
HPMC	Hydroxypropyl methyl cellulose
Mg	Magnesium
mm/min	Millimetre/minute
MPa	Megapascal
MJ/m^3	Millimetrejoule/minute cubic
NaCl	Sodium Chloride
NaOH	Sodium Hydroxide
PMMA	Polymethyl methacrylate
PSA	Particle size analyser
PVA	Polyvinyl alcohol
SDP	Sintering dissolution process
SEM	Scanning electron microscope
SM	Stereo microscope
SiC	Silicon carbide
TGA	Thermogravimetry
W	Energy absorption capability
W_a	Weight of dry sample
W_b	Weight of the sample in water
W_c	Weight of wet sample

LIST OF SYMBOLS

%	Percentage
Wt.%	Weight percent
˚C/min	Degree celcius/minute
˚C	Degree celcius
ρ	Density
σ	Stress
Ɛ	Strain
μ	Micron

KESAN SAIZ PARTIKEL KARBAMIDA TERHADAP MORFOLOGI DAN SIFAT MAMPATAN BUSA BERASASKAN MAGNESIUM

ABSTRAK

Dalam kajian ini, kesan saiz karbamida terhadap mikrostruktur busa magnesium dan busa komposit magnesium yang mengandungi 1wt.% dikaji. Busa magnesium dan busa komposit magnesium disediakan menggunakan kaedah logam serbuk melalui proses pensinteran pelarutan. Serbuk karbamida yang digunakan dalam kajian ini pelbagai saiz: 1.70-2.00 mm, 2.00-2.36 mm, 2.36-2.80 mm dan 2.80-3.36 mm. Kedua-dua dikaji melalui perincian iaitu ketumpatan, keliangan, saiz liang, morfologi, kekuatan mampatan dan tenaga penyerapan. Kesemua liang berada dalam kedudukan seragam dalam kedua-dua busa. Busa magnesium dengan karbamida bersaiz 2.00-2.36 menunjukkan kekuatan mampatan tertinggi iaitu 6.31 MPa, ketumpatan sebanyak 0.49 g/cm^3 dan tenaga serapan sebanyak 11.676 MJ/m^3 dan purata saiz liang (2.17 mm) dan keliangan sebanyak 72.4 %. Manakala, busa komposit magnesium dengan saiz karbamida 2.00-2.36 mm juga menunjukkan kekuatan mampatan tertinggi iaitu 5.094 MPa, tenaga penyerapan sejumlah 8.975 MJ/m^3 dengan ketumpatan sebanyak 0.49 g/cm^3 dan keliangan 72 %. Hasil kajian menunjukkan bahawa saiz karbamida memainkan peranan penting dalam menentukan kekuatan mampatan busa magnesium dan busa komposit magnesium melalui keseragaman taburan partikel karbamida.

THE EFFECT OF CARBAMIDE PARTICLES SIZE ON MORPHOLOGY AND COMPRESSIVE PROPERTIES FOAM BASED MAGNESIUM

ABSTRACT

In this work, the effect of carbamide particles size on morphology and mechanical properties of magnesium and magnesium composite foam containing 1 wt.% alumina were investigated. Magnesium and magnesium composite foam were prepared by powder metallurgy method using sintering dissolution process. The carbamide particle used in this experiment were varied in size; 1.70-2.00 mm, 2.00-2.36 mm, 2.36-2.80 mm and 2.80-3.36 mm. Both of magnesium foams were characterized for morphology, pore size, density, porosity, compressive strength and energy absorption using stereo microscope, densimeter and Instron test. All the pores are distributed homogenously for all the pore sizes for both magnesium and magnesium composite foam. The magnesium foam with carbamide size in the range of 2.00-2.36 showed the highest compressive strength of 6.31 MPa and energy absorption of 11.676 MJ/m^3 with an average pore size of 2.17 mm, density of 0.49 g/cm^3 and porosity of 72 %. While, magnesium composite foam with carbamide size in the range of 2.00-2.36 also showed the highest compressive strength of 5.094 MPa, energy absorption of 8.975 MJ/m^3, density of 0.49 g/cm^3 and porosity of 72%. The results showed that carbamide particles size play an important role in determining the compression properties of magnesium and magnesium composite foam by the distribution of carbamide particle size.

CHAPTER 1

INTRODUCTION

1.1 Introduction

Nowadays, materials with functional properties such as light weight, high strength, good impact energy and high permeability has attracted the attention of the industries as the material use in application automotive, aerospace, building, railways and chemical application. A new type structure and functional materials, porous materials have better properties than solid materials, which broaden the fields of research and application of foam materials. Among all available metal foams aluminum foams are the most preferred one due to their light weight. However, the use of magnesium makes foams even lighter due to its low density, 1.74 g/cm^3 compared to 2.7 g/cm^3 for aluminum. Moreover, Mg foams show greater strength than Al foams (Yilong *et al.*, 2016).

Magnesium foam is a material that satisfied the demand of having lightweight materials. Compared to aluminium foam, magnesium and magnesium alloy foams have better properties because it has long plateau stress region and the natural damping property of magnesium (Hao *et al.*, 2007). Moreover, magnesium foam has better properties in term of low melting point, stability at high temperature and high thermal conductivity. Due to its properties, magnesium has attracted the attention of industry to be used in many applications such as automotive, aerospace and construction of vehicles.

The strength of magnesium foam can be increased by strengthening/reinforcing with hard particle to form composite foam. Composite metal foam has higher strength than metal foam. Composite foam method is widely used because of the flexibility and existence of wide range of reinforcing materials. Reinforcing material that can be used to form composite are ceramic particle such as ceramic fibres, silicon carbide (SiC), ceramic nanoparticles and alumina (Al_2O_3). However Al_2O_3 is widely used because it has low density, high specific strength, stiffness and damping capacity (Braszczyńska-Malik and Kamieniak, 2017). Xia *et al.* (2015) have reported that, the addition of ceramic particle can changes the foams deformation mode from brittleness to ductile due to the reaction between ceramic particle and magnesium.

1.2 Problem Statement

In fabrication of metal foam, having good pores distribution is the most difficult part. The good pores distribution is the main factor that control the properties of metal foam. Homogeneous distribution of powder and space holder during mixing process is importance in order to have uniform distribution of powders and space holder. During mixing process, space holder has different density compared to metallic powder, which at the end of mixing it will cause different distribution. Sintering dissolution process shows that it is the best fabrication method to achieve high properties of metal foam. It is because sintering dissolution process able to completely remove the space holder from the metal to form porous metal.

Another problem that faced during fabrication of metal foam is inhomogeneity of pores shape. There is a strong relationship between morphology and mechanical properties. Basically, pores shape is reflected by the space holder shape. Morphology of pore structure is also influenced by space holder amount and size. The amount of space holder will affect the pore formation because the space holder tends to agglomerate and formed larger pores. Some of the pores are connected to each other and makes the pores become larger. In order to have good a distribution of pores in the foam structure, an appropriate selection of space holder quantity and size need to be controlled. Many researchers had investigated the effect of amount of space holder, pore shape and space holder shape, however, there is no comprehensive study on space holder size for magnesium foam.

Referring to the problem mentioned above, it can be concluded that morphology and structure of magnesium foam also depends on size of space holder. Therefore, it is important to determine the optimum size of space holder because it will affect the compressive strength and energy absorption. Thus, this study was initiated to investigate the effect of different carbamides particles on morphology and properties of magnesium and magnesium composite foam.

1.3 Objectives of Research

The objectives of the research are:

1. To fabricate magnesium and magnesium composite foam using different size of carbamide particles.
2. To investigate the effect of different carbamide particles sizes on morphology, physical properties and mechanical properties of magnesium and magnesium composite foam.

1.4 Scope of Work

Magnesium and magnesium composite foam were fabricated using powder metallurgy method with sintering dissolution process. The space holder used in this experimental is carbamide particle and it was used in different size in order to determine the optimum size for carbamide. Magnesium composite foam was added with 1 wt.% of alumina as the reinforcement and the composition of alumina was fixed for every magnesium composite foam. The magnesium and magnesium composite foam were characterized for their density, porosity, morphology, compression strength and energy absorption.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Cellular material can be defined as material with a relative density less than 0.3 (Gibson and Ashby, 1977). Cellular material is widely used because of their properties such as high stiffness, low specific weight and other properties. The application of cellular material are in various type from light weight construction, packaging, thermal insulation, vibration damping and chemical infiltration (Yu *et al.*, 1998). There are many types of cellular material such as ceramic, polymer and metal. Among them, cellular metal has large range of application because of their excellent physical and mechanical properties, as well as their relative amenability to recycling compared to polymeric foams. Cellular metal also known as metal foam was chosen for this study. Metal foam is defined as metallic cellular consisting of a solid metal containing a large volume fraction of gas-filled pores

2.2 Metal Foam

Metal foam is a porous material with many type of structure and functional materials. Metal foam have better characteristic compare to solid metal because it has good permeability, high thermal shock resistance, high temperature resistance and shape stability (Gibson, 2001). Metallic foam is used widely in brakes and shock absorber of automobiles. Metal foam also have been used as the structural material for impact energy absorbing and was reported to reduce vehicle vibration and collision safety of automobiles (Yilong *et al.*, 2016). There are many types of metal foams available in the market which is depends on the requirement of the products. Pores structure can be found in two type which is open-cell structure and closed-cell structure. An open- cell metallic foam is an interconnected pore which the pores link together without boundaries. Besides that, open cell pores make excellent heat dissipation devices due to their high thermal conductivity, high internal surface area and the connectivity of the voids. For closed-cell metallic pores it widely used for structural engineering application. It is because the properties that required for engineering application own by closed cell metal foam which are lightweight, high strength to weight ratio and good damping of noise and vibration. Figure 2.1 shows open and closed cell structures of foam.

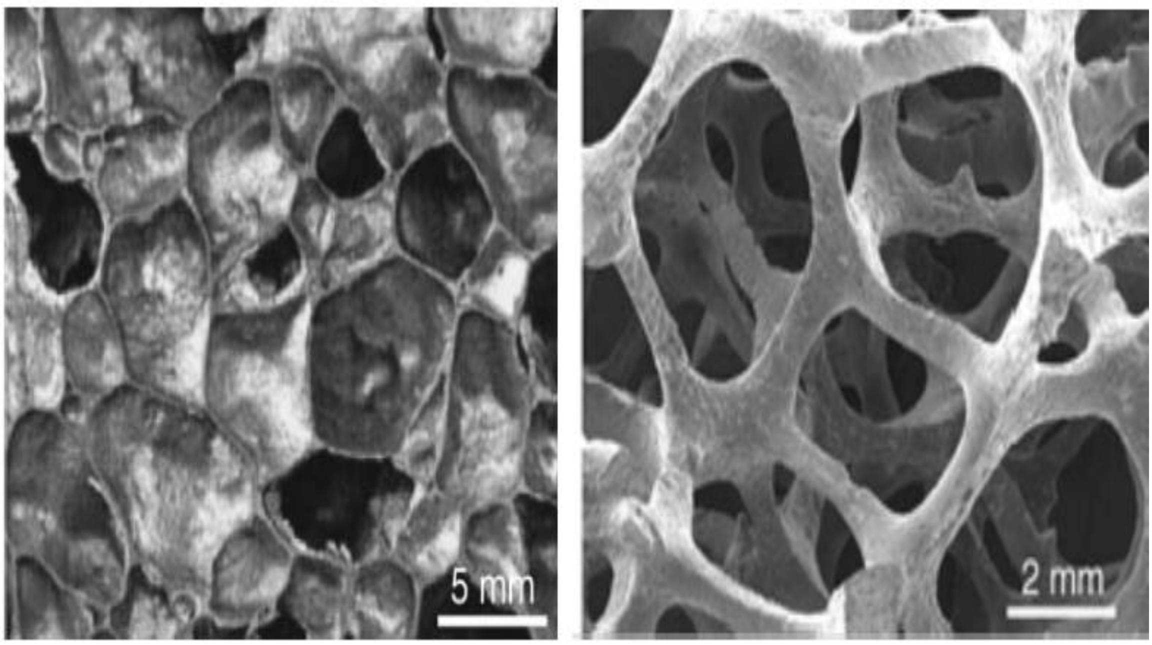

Figure 2.1: Micrographs of (a) closed cell foam and (b) open cell foam (Kennedy, 2012)

2.3 Types of Metal Foam

There are several types of metal foam such as steel foam, titanium foam, aluminium foam, magnesium foam and many more. These types of metal foams have different properties.

2.3.1 Steel Foam

Steel is an alloy metal which consists of iron and carbon as alloying element. The alloying elements in the steel contribute to increase the properties of hardness, ductility and tensile strength. With high amount of carbon content in steel, it can become harder and stronger than iron, but it also less ductile than iron. There any many applications that used steel as their material such as in construction industry which steel use in construction of road and railways. However, steel application has become limited because of heavy weight. Concerning this problem, steel foam has been fabricated in order to reduce the weight. According to Bekoz and Oktay (2013), steel foam can have porosity up to 70 % and having low density with high compressive stress.

2.3.2 Titanium Foam

Titanium is a strong metal with low density metal and metallic white in colour. Titanium can be alloyed with many alloying element. There are many advantageous of titanium alloys such as high tensile strength, high crack resistance and ability to withstand moderately high temperature without creep. The application of titanium is highly used in aircraft, armour plating and space craft. Since titanium has superb properties, many researchers are interested in fabricating titanium foam. For example, Shbeh and Goodall (2016) have suggested to fabricate open pores titanium foams via metal injection molding with space holder. The space holder that they used is potassium chloride with two different shapes in order to observe which shape has better flowability. It was found that spherical shape has better flowability compared to cube. Furthermore, Torres *et al.* (2014) have developed porous titanium for biomedical application by comparing the technique of loose sintering and space-holder technique.

2.3.3 Aluminium Foam

Aluminium is light, has one third the density of steel and has high heat and electricity conductivity like copper. Besides, it has excellent corrosion resistance in most environment (Stanev *et al.*, 2016). Due to many advantages of aluminium foam it has been considered as good metal foam in term of properties. Aluminium foam has widely used in application such as railway, automobile and aerospace industry (Huo *et al.*, 2012). Besides that, Salvetr *et al*. (2016) have mentioned due to high stiffness in spite of low density, recyclability, high energy absorber and vibration suppression, aluminium foam is a good metal foam to be used in application of noise and vibration absorption. In addition, Luong *et al*. (2013) have suggested that the performance of aluminium foam is high in terms of energy absorption compared to polymeric material while considering the greater weight per volume unit.

2.3.4 Magnesium Foam

Magnesium is a silver white metal and can be found in earth crust and sea water as magnesium salt. However, magnesium cannot be found in pure form because it bonds with another element. Hence, to obtain pure magnesium several processes need to be done. Magnesium has great advantage compare to other metals because it is 35% lighter than aluminium and 75% lighter than steel. Besides that, it also has good mechanical and physical properties.

Magnesium and its alloy have an important role among structural materials. Magnesium is used mainly in the construction of vehicles and the aerospace industry, where it is used mainly in mechanically less-strained components. These special applications include, for example steering wheels, dashboards, seats and gear boxes. In future, magnesium is also proposed to be a material able to store hydrogen. Magnesium metal has a lot of advantages, such as specific strength, stiffness, high modulus elastic, good defense noise and excellent vibration reducing performance. They exceed other materials, especially with their low density (1.74 g/cm^3) compared to aluminium (2.7 g/cm^3). Magnesium also is the lightest metal in industrial application. This makes magnesium lighter than aluminium. Hence for light-weight construction, Mg foams have even greater potential than Al foams.

Moreover, magnesium foam has better properties in terms of melting point, stability at high temperature, and harder aging (Yilong *et al.*, 2016). Similarly Jaroslav and Vojtěch (2014) have stated that that the properties of magnesium which is biodegradability and biocompatibility and with sufficient mechanical strength makes it suitable in application of load bearing orthopaedic implants. Moreover, magnesium foam has better vibration resistance, more convenient installation, disassembly and other excellent properties compared with ceramic foam.

2.4 Composite Metal Foam

Porous magnesium is emerging as an interesting type of material because of lightweight materials and porous scaffold substitutes. Introduction of pores into magnesium often reduce its strength. Porous pure magnesium with 35% porosity was reported to have a peak compressive strength of 17 MPa which is lower than the yield strength of sand cast magnesium. To improve the strength of porous magnesium, it can be done by a reinforcing phase to produce porous magnesium composite (Zou and Li, 2016). 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.

2.4.1 Metal foam reinforced with silicon carbide (SiC)

Silicon carbide (SiC) has been widely used as reinforcement in magnesium foam. Shen *et al.* (2016) have fabricated magnesium foam by stir casting technology followed by hot extrusion. They found that SiC cluster which was caused by the push effect in the as-cast composite and disappeared after hot extrusion process. Xia *et al.* (2015) has reported that amount of SiC influenced the strain rate, cell size, relative density and energy absorption. The cell size has a significant effect on energy absorption properties. According to Ghasali *et al.* (2017), SiC can be used as reinforcement to improve the mechanical properties of base metal. Most of reinforcement material can be used to enhance mechanical properties however, SiC has their own contribution to the mechanical and physical properties of magnesium composite foam.

2.4.2 Metal foam reinforced with carbon nanotubes (CNT)

Metal-matrix composite can be fabricated as light weight and high strength material because of their low density and high values of aspect ratio and mechanical strength. However, carbon nanotubes are minorly used as reinforcing material because of their tendency to form clusters, poor dispersion ability and poor wettability of carbon by molten metal due to a large difference of surface tension. Moreover, the limitation of CNT is they tend to widespread which will cause formation of interfacial reaction product in molten metals. There are many processing method that had been done to overcome the problem such as powder metallurgy, molecular level mixing, and casting but the result is limited. The process can be successful if carbon nanotubes are dispersed well in the metal matrix and formed strong interfacial bonding. The retention of structural integrity of CNT are the main challenges in order to develop metal matrix nanocomposite for industrial application. Recently a manufacturing of metal matrix composite reinforced with CNT using sandwich method which structural layer of multiwall carbon nanotubes were used (Isaza Merino *et al.*, 2016).

2.4.2 Metal foam reinforced with alumina (Al_2O_3)

Metal foam also can be reinforced with alumina (Al_2O_3). Metal foam with alumina reinforcement showed high thermal and mechanical stability (Wang *et al,* 2016). Cay *et al.* (2013) have conducted an experiment using alumina powder with size 0.5 μm as the reinforcing phase with amount of 0.05 and 1 wt.%. They found that the density of Mg-0.1% Al_2O_3 is higher than that of Mg-0.05%Al_2O_3. Besides that, Kouzeli and Dunand, (2003) have mentioned that alumina particle does not coarsen during sintering and it maintain their shape and size after sintering process. As the result, the compressive stress of magnesium reinforced alumina foam was high compared to magnesium foam. Moreover, Licitra *et al.* (2015) have used hollow sphere alumina particles, with average diameter and wall thickness of 3000 ± 220 μm and 107 ± 7 μm, respectively. The alumina enhanced the properties of metal foam by increasing the properties of compressive and strength. It also contributed to the brittleness of the metal foam as it increases the strength.

2.5 Fabrication Method of Magnesium and Magnesium Composite Foam

Magnesium and magnesium composite foam can be fabricated by several methods such as melt forming agent, topological-ordered architecture, investment casting, melt gas injection and powder metallurgy depending on the desired properties. Each of these methods has the advantageous and disadvantageous.

2.5.1 Melt Foaming Agent

Melt foaming agent is a method which injecting gas at certain pressure into the liquid to fabricate metallic foam. The gas bubble will in metallic molten and it will rise to the surface quickly due buoyancy forces in the low dense liquid. However, the rise can be controlled by slowing down the bubble from rise to the surface by increase the viscosity of the molten metal. Besides using injecting gas, blowing agent which decomposes under the influence of heat and release gas can be adding in this method. The forming process will propel as the gas has been release. Figure 2.2 shows the process of melt forming agent. Yang *et al.* (2010) have using this method to fabricate magnesium foam by using Ca particle as thickening agent and calcium carbonate ($CaCO_3$) as blowing agent. Firstly, a defined quantity of pure magnesium (~1 Kg) was melted in crucible at fixed temperature. To increase the viscosity of molten magnesium, Ca was introduced. Then blowing agent $CaCO_3$ powder (2 wt.%) were added at proper foaming temperature. While in fabrication, the stirring and holding foaming time were set as 30 s. To control the pores structure of final product the temperature was adjusted in the range of 680-750 ˚C and as the result that they obtained, the porosity of magnesium foam is ∼53.0% to ∼72.0% and pores size around ~ 2.0mm. Wang *et al.*

(2017) have fabricated closed-cell AZ31 magnesium alloy foam by melt foaming using Ca and $CaCO_3$ as thickening and blowing agents, respectively.

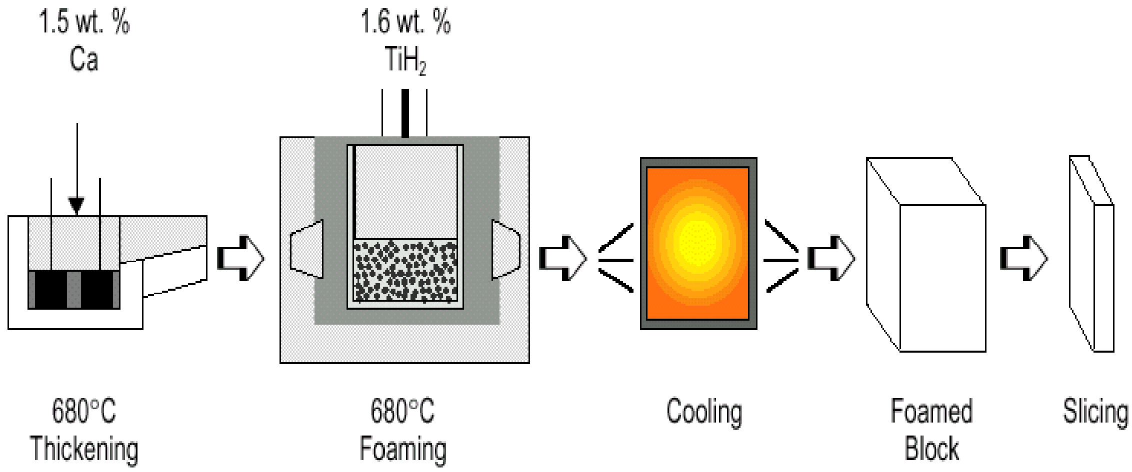

Figure 2.2: Fabrication metal foam by melt foaming agent (Banhart, 2001)

2.5.2 Topological-Ordered Architecture

Staiger *et al.* (2010) have introduced a method to prepare open-cell porous magnesium with topological-ordered architecture. The process consists of several steps such as (i) creating a 3D model with a desired architecture using computer-aided design (CAD); (ii) 3D printing of a positive polymeric template of the CAD model; (iii) infiltration of the polymeric template with NaCl paste; (iv) burn-out of polymeric materials and sintering of NaCl; (v) pressure-assisted infiltration of the negative NaCl template with liquid Mg; and (vi) removal of the NaCl template.

2.5.3 Investment Casting

This investment casting method or also known as infiltration method is one of the process of fabricating the magnesium metal foam. Space holder material NaCl normally was used as a precursor due to its low cost and easy to remove during leaching process. The NaCl placed inside the mould as the preform. Then, molten metal was poured into the mould and infiltration process take place. During infiltration, molten metal infiltrate inside the preform by gravitational force. An inert gas was supplied throughout the system. The specimen was immerged in distilled water to leach out NaCl. Figure 2.3 shows the of schematic diagram of investment casting.

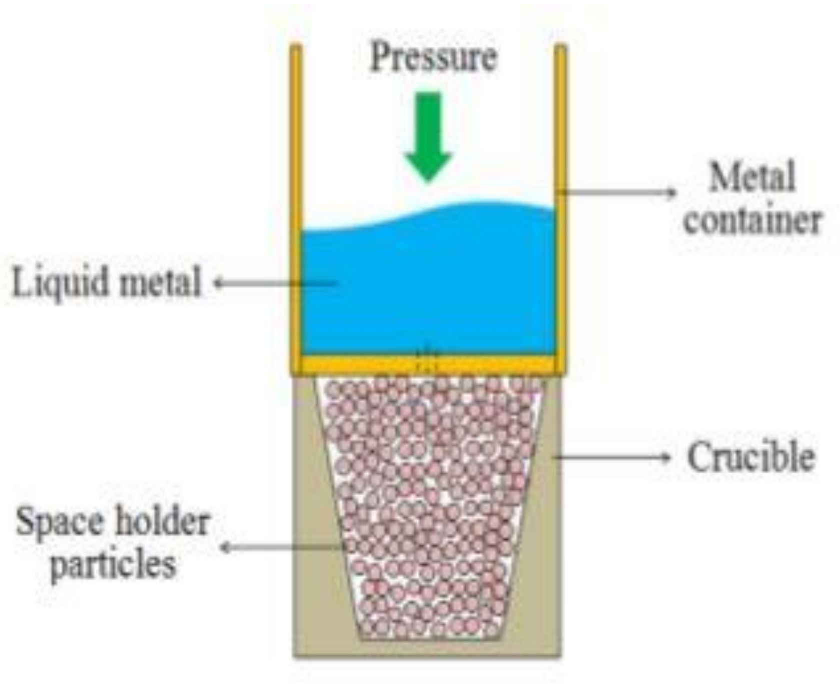

Figure 2.3: Schematic diagram of investment casting (Báez *et al*, 2015).

2.5.4 Melt Gas Injection

Melt gas injection method is much easier and not required complex process to fabricate metal foam. The pores in the structure formed by rotating impellor because the viscosity of molten metal need to be high. During the rotational of impellor, the gas was introduced under the surface of the molten metal. The molten metal with trapped bubble is cooling immediately in order to prevent the bubble gas escape. This method has high effectiveness however the size of bubble is hard to control and it will produce irregular shape of pores. Figure 2.4 shows the process of melt gas injection.

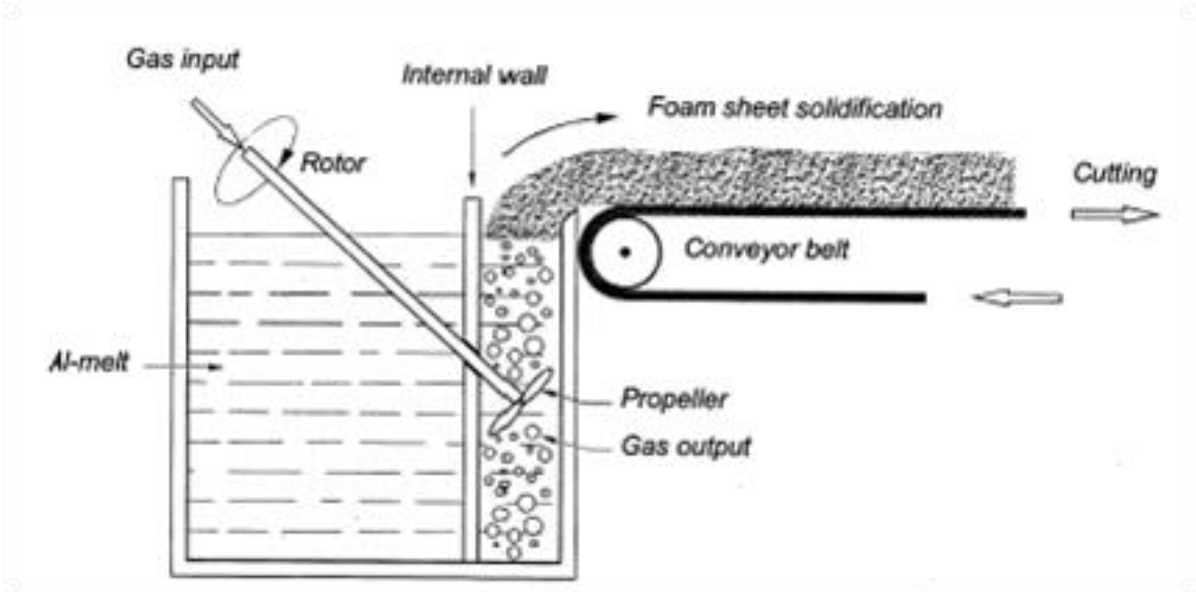

Figure 2.4: Schematic of melt gas injection in fabrication of metal foam (Banhart, 2001)

2.5.5 Powder Metallurgy (PM)

A powder metallurgy method for fabricating metal foams was invented at the Fraunhofer Institute for Applied Materials Research in Bremen, Germany (Hausner, 1995). A metallic foam is fabricated by consolidation of a metal powder with a particulate foaming agent (typically TiH_2) followed by heating near the melting point when the foaming agent releases hydrogen, expanding the material. The produced foams have closed cells with cell size of 1–5 mm (Sadighikia *et al.*, 2015). This method allows for low cost and direct net-shaped fabrication of foamed parts with a relatively homogeneous pore structure. Using the powder metallurgical production method, it is now possible to obtain metallic foams of various metals and alloys with complex geometry. Figure 2.5 shows the process of powder metallurgy to form metal foam.

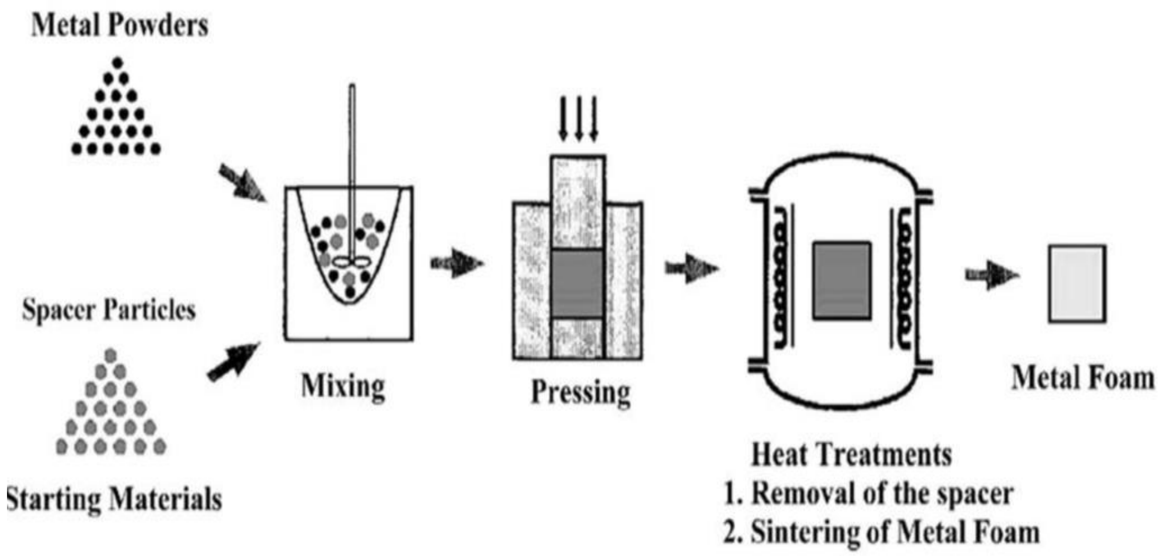

Figure 2.5: Schematic diagram of powder metallurgy to fabricate metal foam (Li *et al.*, 2006)

PM route for the production of open-cell metal foam is based on co-pressing of a metal powder with a leachable powder (space holder) followed by removal of the space holder to leave a porous metallic network. The space holder material must be easy to decompose and do not form a reaction with the material that had been used. There are many types of space holder used such NaCl (Aghion and Perez, 2014), carbamide (Hao *et al*. 2009), ammonium bicarbonate (Čapek and Vojtěch, 2014) and PMMA (Bi *et al.*, 2015). The space holder directly determines the structure parameters as the porosity percentage and the pore shape, the liquid phase processing technique employed for production, the spacer removing process, the sintering technology, size and shape of obtained parts, and finally their price. Carbamide (urea, $(NH_2)_2CO$) was widely used because it low in cost, high availability, easy to process and easy-leachable substance. During preparation of green body by compaction, carbamide is normally used because it easy to remove by leaching and sintering. During sintering process carbamide will be decomposed to carbon and nitrogen (Bafti and Habibolahzadeh, 2010). The removable process of carbamide can be done by two step which is leaching and sintering. It can be leached in solution of NaOH and sintering using sintering dissolution process (SDP).

Wen *et al.* (2004) in their experimental produced magnesium foam using powder metallurgy method which apply the space holder particle to create pores as the space holder removed. Beside that Yilong *et al.*, (2016) have fabricated magnesium foam using powder metallurgy with space holder technique. The magnesium powder use has high purity with purity ≥ 99.5%, particle size 50-75 μm and carbamide particle with purity ≥ 99.5%, particle size 1.1~1.2 mm as their raw material. Carbamide as raw material can hold spaces of pores at low temperature and decomposed completely at temperature 400˚C as the carbamide will not react with magnesium powder. Furthermore, high strength stainless steel foams were also produced with powder metallurgy approaches that may represent an interest when high mechanical strength and corrosion resistance are required. Besides, steel hollow spheres with high strength have been prepared and characterized (Kennedy, 2012).

2.6 Sintering Dissolution Process

Sintering dissolution process has been widely used as the sintering method for magnesium and magnesium composite foam. Compared to other method of sintering, sintering dissolution process can produce higher and better properties in term of strength and mechanical properties. Sintering dissolution process consist of four steps; mixing, compaction, dissolution and sintering process.

2.6.1 Mixing Process

Mixing process was done to ensure homogenous distribution of space holder and magnesium powder and obtained uniform pore distribution. Therefore, magnesium powder and carbamide must be thoroughly mixed before used to prevent segregation and to maximize homogeneity. Liquid binder such as hydroxypropyl methyl cellulose (HPMC), polyvinyl alcohol (PVA) and ethanol was added during mixing process to ensure space holder coated with metal powder and obtained sticky surface for adhesion of magnesium particles (Bekoz and Oktay, 2014). Based on research done by Ghasali *et al.* (2017), the carbamide particle and Mg powder were manually mixed until full homogeneity was achieved, in order to avoid segregation of dissimilar powder and particles, a small amount of paraffin powder or ethanol (2 wt.%) was sprinkle during the mixing process. In addition, Yilong *et al.* (2016) also have added a little amount of ethanol as dispersing agent so homogeneous blend of materials can be obtained. Cay *et al*, (2013) have conducted the mixing process using planetary ball mill to blend the mixture of magnesium, alumina and carbamide. Ghasali *et al.* (2017) used magnesium and boron carbide powder as the starting material and the powder was mixed using high energy ball mill without any protective atmosphere. The time taken to complete the mixing process is 10 min and the mixture was dried on hot plate at 70 ˚C with stirring magnet.

2.6.2 Compaction Process

Powder compaction can be done using uniaxial pressing, the standard compaction technique. It must be ensured that carbamide is completely embedded in the magnesium matrix. Compaction of magnesium and carbamide particles mixture powder is carried out to obtain green body into required shape and easy handling for further process. The specific strength of green body is low and required sintering to achieve the strength. Fluidity of powder is a main factor in packing the powder into die. The powder not distributed uniformly if the fluidity is low. Therefore, to obtain low density and homogeneous compaction density, powder properties must be controlled (Yanai *et al.*, 1998). Compression pressure is also an important parameter to be considered before compacting the samples. There is no good bonding between the matrix and reinforcement if the compression pressure is not enough and the samples will collapse if the compression pressure is too high. In addition, if the compression pressure is too high, it will destroy the structure of space holder. Yilong *et al.*, (2016) have reported that the powder of the mixture was uniaxially pressed at 200 MPa for 1 min to transmit the pressure sufficiently and avoid the bridging effect which the pores were connected each other. However, Báez *et al*, (2015) have stated that the mixture of magnesium and space holder was uniaxially compacted in a die at a given pressure of 300 MPa and kept for 2 min before it removed from the die. Stanev *et al.*, (2016) have demonstrated that mixed substances are compressed under various uniaxially pressure in range of 200-350

MPa. It is reported that, pressure above 350 MPa will result in crashing the carbamide particles and affect the shape of the carbamide and size of the pores.

2.6.3 Dissolution of Carbamide Particle

Dissolution process is the process of removal the space holder. The carbamide space holder was successfully removed by dissolution in water and NaOH solution. Wang *et al.* (2014) have reported that carbamide space holder can be easily and quickly dissolve in water. Water also can easily penetrate into carbamide particle and make them dissolve accelerative. Moreover, Bekoz and Oktay, (2014) also use water as leaching technique. This is because leaching in water have better dissolution rate and free toxicity. Recently, Jin *et al*. (2007) have reported that NaOH aqueous solution can dissolve carbamide directly and quickly. The solvent system is inexpensive and less toxic. Too long or too short dissolution time should be avoided in order to prevent the increasing of oxidation and increase the residual impurities in the final product. According to Hao *et al*. (2009) it is important to control the time of dissolve because it will affect the result of sintering quality. To achieve the porosity of 40-80% porosity, the most suitable immersing time should be between 0.5-5 hours in NaOH solution.

2.6.4 Sintering Process

Sintering process is important as it increases the strength of green body and bonding of magnesium particles (Hao *et al*, 2009). Compare to many sintering process, sintering dissolution process produced better strength and properties. Sintering is carried out in tube furnace using inert atmosphere at suitable sintering temperature, below the melting point of magnesium. Generally sintering atmosphere used are endothermic gas, exothermic gas, dissociated ammonia, hydrogen and nitrogen. Typically, sintering temperature are varying within 70-90% of the melting point of metal. Sintering is responsible for producing physical and mechanical properties in the powder metallurgy bond among the powder particles and it is called as solid states process. Mechanical performance of the bond is affected by the bonding of the particles. The strength between the bond and the particles is depends on the complex mechanism of diffusion, recrystallization grain growth and pores shrinkage (Kalpakjian *et al,* 2001).The sintering was done to improve the strength, electrical conductivity and ductility of metal foams. During sintering, the structure and porosity will obtain and it is depending on the temperature, time and processing details. Based on work done by Yilong *et al.* (2016) carbamide start to decompose at temperature of 160˚C and it will stop decomposed at temperature of 400˚C. Hao *et al.* (2009) have mentioned that the most compatible sintering temperature is in the range of 610-630˚C for 2.5 hour. As the result, sintering process increased strength, ductility and electrical conductivity. Besides that, sintering also contribute to removal of liquid from powder and prevent oxidation layer to be form on the surface of the metal foam.

2.7 Properties of Magnesium and Magnesium Composite Foam

To determine the properties of magnesium and magnesium composite foam, some test have been conducted to identify the strength, porosity, density, compression strength and energy absorption. Besides that, the morphology of magnesium and magnesium composite foam was observed to determine the type of pores, pores size, pore shape and pore distribution that will influence the mechanical properties of magnesium and magnesium composite foam (Čapek and Vojtěch, 2014).

2.7.1 Morphology of Magnesium and Magnesium Composite Foam

The morphology of magnesium composite foam is important because the mechanical strength is influenced by type of pore, pore size, pore shape and pore distribution (Jaroslav and Vojtěch, 2014). From the microstructure, the homogeneity of pores distribution can be determined and the density and porosity value of the structure can be evaluated. Figure 2.6 shows the morphology structure of magnesium composite under optical microscope. The pores exist in foam are varied in shape such as spherical or ellipse.

Cay *et al.* (2013) have found that the pores exist is magnesium composite foam was in irregular shape, the thickness between pores was on the order of ten microns and the pore size increase as the porosity increase. Meanwhhile, Xia *et al.* (2015) have used ceramic microsphere as the reinforcement for fabrication of magnesium foam composite. They found that magnesium composite foam with 20% ceramic microsphere under foaming temperature of 680 ˚C showed that pores structure are homogeneous and the pores are in spherical and separated. Besides that, there no burning, coking or reunion of ceramic microsphere is observed during the entire process. It is clear that ceramic microsphere in cell wall well distributed and each of them well oriented by maintaining the original morphology.

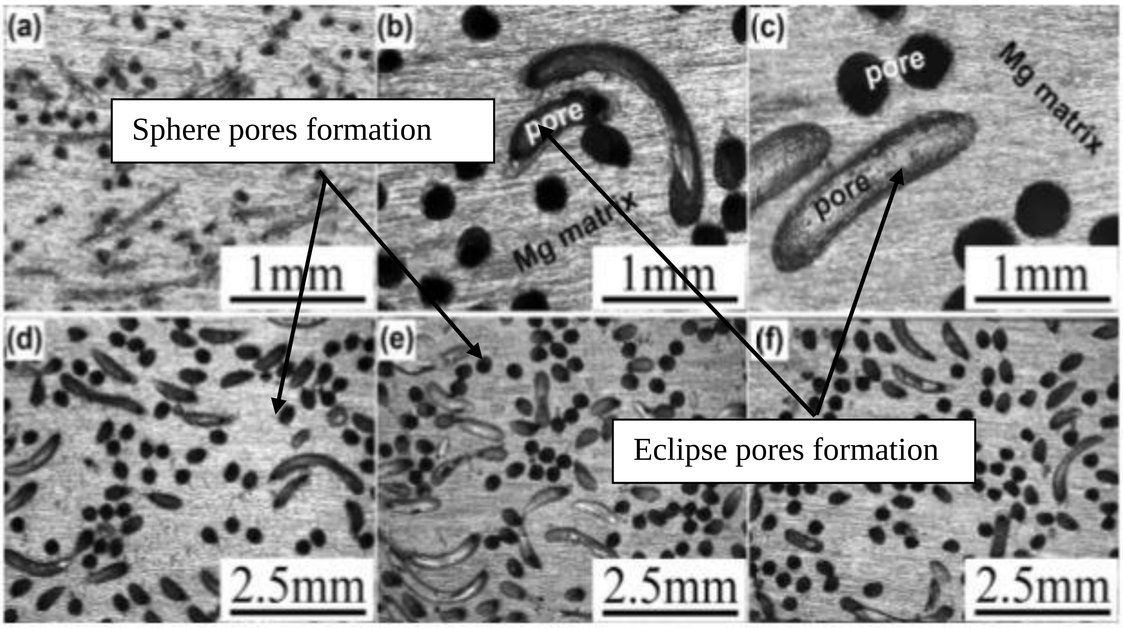

Figure 2.6: Structure of magnesium composite under microscope (Li *et al.* 2016)

2.7.2 Density and Porosity

Density and porosity contribute important properties in application of metal foam. By increasing the porosity in the foam, it can increase the absorption energy of the material. Bulk density of the material can be defined as mass per unit volume. The porosity and density was measured based on Archimedes principle. This was done by measuring the void space inside the material by the fractional volume of void over the total volume.

It is important to measure density and porosity as the compression properties and energy absorption depends on the porosity. The amount of porosity can be controlled by controlling the amount of space holder as 50 wt.% of space holder can provide the amount of porosity in the range of 60-75 %. Higher amount of porosity normally can be used in application of filter. The distribution of porosity is important factor that contribute to the strength of mechanical properties. During compression, the