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## Processing and Characterization of Porous Mg Alloy for Biomedical Applications

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### ABSTRACT

Recently, there has been an increasing demand for biodegradable implants material. Magnesium (Mg) alloy with their superior properties appears to be promising material for the biomedical applications. This work reports the preparation of porous Mg alloy materials through powder metallurgy technique by using ammonium bicarbonate ( $\text{NH}_4\text{HCO}_3$ ) as space holder material and hexane as solvent. Microstructure and mechanical properties of different composition of  $\text{NH}_4\text{HCO}_3$  were evaluated. The obtained porous Mg alloy with porosity ranging from 42% - 64% and pore size 200-500  $\mu\text{m}$  indicates a porous structure with interconnected open pores. An increase in the porosities will weaken the strength of porous Mg alloy, however the properties remain comparable to natural bone properties among the others porous material. Hence, it is suggested that porous Mg alloy has the potential to serve as degradable implants.

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## INTRODUCTION

In recent years, particular attention has been paid to porous materials especially in bone tissue engineering applications as they provide stable biological fixation and enhance bone ingrowth through porous network (Wu *et al.* 2012). An ideal porous implant is expected to have sufficient strength in order to sustain stress and physiological loading during the implantation period, biocompatibility and osteoconductivity to allow integration between implant and bone without causing any harmful effect to the body, biodegradable and bioresorbable preferably at a controlled rate, eventually being replaced by new bone tissue (Bose *et al.* 2012; Zhuang *et al.* 2008). Previous investigation was carried out on numerous porous implant materials including hydroxyapatite and natural polymer, however these materials limited in mechanical properties and inappropriate in load bearing applications (Wen *et al.* 2001; Zhuang *et al.* 2008; Wang *et al.* 2009). Porous metal such as stainless steel, titanium and cobalt based alloys have good mechanical properties, however limitation in the release of metal ion will lead to adverse reaction into the human body (Staiger *et al.* 2006; Zhang *et al.* 2013). Moreover, mismatch between Young's modulus of metallic materials and surrounding bone tissue, resulting in stress shielding effect. Hence, it is essential to develop new porous material with both structure and properties resemble the natural bone.

Mg attractive to serve as implant applications due to its favorable properties such as biodegradability and biocompatibility, its mechanical strength comparable to natural bone and light weight (Čapek & Vojtěch 2013). Common techniques used to manufacture porous metal materials are injection of an inert gas into a melt, directional solidification metal-gas eutectic (GASAR process) and powder metallurgy techniques (Ryan *et al.* 2006). Powder metallurgy (PM) is the most preferable techniques which use space holder filler to generate porous objects. The properties of porous material can be altered by controlling the size, shape and quantity of the space holder material during fabrication. Nonetheless, the mechanical properties of the porous material are not depending entirely on these selections, but also require a sufficient sintering temperature in order to enhance bonding between powder particles. (Hao *et al.* 2009) found that the optimal sintering temperature in a range of 610°C to 630°C.

In this study, the porous Mg alloy samples are prepared by using powder metallurgy technique. The microstructure and mechanical properties of the samples with different porosities are investigated.

## MATERIALS AND METHOD

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### Preparation of porous Mg alloy:

Commercially available AZ91 Mg alloy powders (particle size  $\leq 25\mu\text{m}$ ) with spherical shape are used as starting material. Ammonium bicarbonate ( $\text{NH}_4\text{HCO}_3$ ) as space holders were sieved manually, to get particle size of  $\leq 212\mu\text{m}$  in order to meet requirement for biomedical application with chemical properties of it to be completely removed at low temperature ( $< 200^\circ\text{C}$ ) to avoid the reaction with host powder (Wen *et al.* 2001).

The AZ91 Mg alloy powders are manually mixed with different ranges of  $\text{NH}_4\text{HCO}_3$  from 30-50vol% for 30 min. Liquid hexane are added at a volume fraction of 30% in order to avoid the segregation of the powders before it is uniaxially pressed at a pressure of 325 MPa into green compacts (D 12mm X H 10mm). The samples were subsequently heat treated at  $180^\circ\text{C}$  for 3h in air to burn out the space holder and sintered at  $600^\circ\text{C}$ ,  $620^\circ\text{C}$  and  $640^\circ\text{C}$  for 4h under argon atmosphere.

### Microstructure analysis:

The bulk density and porosity of the porous bodies are measured using Archimedes method. Total porosity of the porous bodies is measured according to the equation below (Zhuang *et al.* 2008):

$$\Pi = (1 - \rho / \rho_s) \times 100 \quad (1)$$

where  $\rho$  and  $\rho_s$  are the density of porous bodies and its corresponding theoretical density, respectively. Pore structure and pore size distribution of the porous bodies are characterized using Scanning electron microscopy (SEM-TM 3000-Hitachi) and Image J software. The phase constituents of the porous bodies are observed using X-ray diffraction (XRD).

### Mechanical properties analysis:

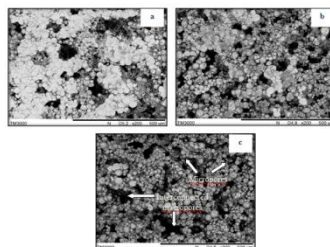
Compression tests are carrying out using Shimadzu Universal Testing Machine according to the ASTM standard (D695-02a). The cylindrical samples are subjected to compression crosshead speed of 1mm/min, to a maximum reduction in sample height of 80%. The elastic modulus is calculated from the stress strain curve obtained.

## RESULT AND DISCUSSION

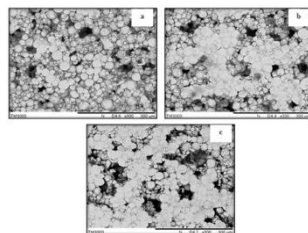
### Microstructure of porous magnesium alloy:

The SEM images of the porous Mg alloy with different measured porosities are shown in Fig.1. According to Eq. (1), the porosities of the three types of porous samples are about 42%, 54% and 62% respectively. Clearly shown, the space holder content controls formation of porosities of final sample. Visual characterization of the porous samples shows two types of pores which are open interconnected macropores and isolated micropores with the size ranging from  $200\text{-}500\mu\text{m}$  and  $30\text{-}60\mu\text{m}$  respectively. Macropores formed from decomposition of space holder, while micropores are resulting from incomplete compaction and the volume shrinkage during the sintering process of Mg alloy powders. Proper combination of macro and micropores is desirable for the ingrowth of new bone tissue (Wu *et al.* 2012). In addition, good interconnectivity of pore is essential in stimulating bone regeneration and allow the flow transport of body fluid (Nouri *et al.* 2007). As observed in the Fig.1 (b) and (c), an increase in the porosities leads to insufficient diffusion between Mg particles as the structures will weaken the strength of porous Mg alloy. Incomplete diffusion between Mg alloy particle occur due to poor heat transport in porous material (Randall M. German 1996). This result implies that porous Mg alloy with porosities of 42% considered as the desired porosities in biomedical applications.

Fig.2 shows the SEM micrograph of porous Mg alloy sintered at different temperature. Observed, inter diffusion between Mg alloy particles increase equally with respect to sintering temperature. The appropriate sintering temperature is  $640^\circ\text{C}$  because the diffusion effects result in an obvious increasing of the mechanical properties. (Hao *et al.* 2009) indicates that too lower or too high sintering temperature would influences in strength of the porous Mg alloy as insufficient bonding between Mg alloy particles arise from low sintering temperature and require prolonged time to ensure bonding between Mg alloy particles. Indeed, higher sintering temperature leads to non-uniform distribution of cell size and shape distortion, due to partial melting of the Mg particles.



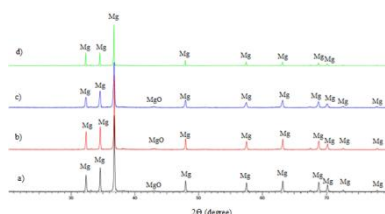
**Fig. 1:** SEM micrograph of the porous mg alloy specimens, sintered at  $640^\circ\text{C}$ , with different porosity level  
a) 42%, b) 54% and c) 62%.



**Fig. 2:** SEM micrograph of the 42% porosity mg alloy specimens, sintered at different temperature: a) 600°C, b) 620°C and c) 640°C.

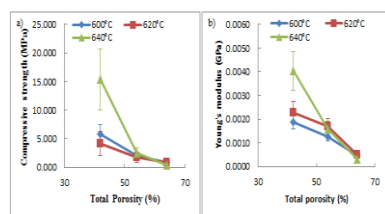
#### *X-ray diffraction (XRD) analysis:*

Fig. 3 shows the X-ray diffraction patterns of Mg alloy powder and the porous Mg alloy with 42% porosity sintered at different temperature withno elements of  $\text{NH}_4\text{HCO}_3$  areas detected remaining after sintering that yields a well matched of Mg alloy powders. This observation support the result attained from SEM analysis (Fig. 2). Despite detection of the Mg peaks in all of the samples, minor phase of MgO was noticed in the patterns due to oxidation during the sample preparation and sintering process (Capek & Vojtěch 2014), in conjunction with, (Bobby Kannan & Singh Raman 2008) claimed that MgO is biologically a non-toxic oxide.



**Fig. 3:** XRD patterns of the porous Mg alloy sintered at the temperature of a) 600°C, b) 620°C, c) 640°C and d) Mg alloy powder.

#### *Mechanical properties:*



**Fig. 4:** a) Compressive strength, b) Young's modulus of the porous Mg alloy sintered at different temperature.

Fig. 4 demonstrates the changes in compressive strength and Young's modulus values of the porous Mg alloy as a function of porosity at different sintering temperature. The compressive strength and Young's modulus decrease generally with an increasing in porosities at all sintering temperatures. (Xi-qin *et al.* 2006) noted that the porosities, especially the open pores will influence the actual load bearing area and thus evident in the decrease of mechanical properties of the porous Mg alloy sample. Higher sintering temperatures contribute to higher compressive strength and Young's modulus and thus, confirm that 640°C is the desired sintering temperature. Although investigation in the present study shows the mechanical properties of porous Mg alloy is lower than human cortical bone (160 – 240 MPa) (Eddy *et al.* 2012) and not suitable to be applied in the load bearing applications, however the present mechanical properties is fairly comparable to those of cancellous bone (1.5 – 9.3 MPa) (Eddy *et al.* 2012). Hence it is suggest that porous Mg alloy is suitable to be applied in non-load bearing applications or in combinations with other fixation devices such as plates, screws and pins, as suggested by (Hutmacher *et al.* 2007)

A summary of the mechanical properties of natural bone tissue and others porous material are investigated in Table 1. The table exhibited the mechanical properties of porous Mg alloy are comparable to natural bone which also exceed than non-metallic porous material. Therefore, it is recommended that porous Mg alloy is one of the promising porous materials for biomedical applications.

**Conclusion:**

Porous Mg alloy samples were successfully fabricated through powder metallurgy technique under different sintering temperature. Samples formed in different porosities were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and compression test to investigate the influence of the porosities in the microstructure and mechanical properties. Based on the results, the desired sintering temperature to fabricate porous Mg alloy is 640°C. The result indicates that  $\text{NH}_4\text{HCO}_3$  plays a role in improving the mechanical properties. In particular, a porous Mg alloy with 42% of porosity, a compressive strength of 15 MPa and a Young's modulus of 0.05 GPa shows a comparable properties similar to natural bone. Therefore, it is recommended that porous Mg alloy is one of the promising porous materials for biomedical applications.

**Table 1:** Summary of mechanical properties of various natural bone and porous materials.

Porous material	Porosity (%)	Pore size ( $\mu\text{m}$ )	Compressive strength (MPa)	Modulus (GPa)	Reference
Natural bone	-	-	2 - 180	0.01 - 23	(Zhuang <i>et al.</i> 2008; Eddy <i>et al.</i> 2012)
Porous Mg alloy	42 - 62		1 - 15	0.001 - 0.05	This study
Porous Mg	36 - 55	200 - 400	15 - 31	0.05 - 1.2	(Zhuang <i>et al.</i> 2008)
Porous Mg	52 - 70	~1250	4 - 14	-	(Hao <i>et al.</i> 2009)
Porous Mg	29 - 331	250 - 500	20 - 70	-	(Capek & Vojtěch 2014)
Porous Mg	50	200 - 500	3	-	(Wen <i>et al.</i> 2001)
Porous Mg	35 - 55	100 - 400	12 - 17	-	(Wen <i>et al.</i> 2004)
Porous HA	50 - 70	200 - 400	1 - 17	0.12	(Zhuang <i>et al.</i> 2008)
Porous composite Bioglass	77 - 80	~100	~0.42	0.14 - 0.26	(Zhuang <i>et al.</i> 2008)

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