Fabricating and Testing of Porous Magnesium Through Powder Metallurgy Technique using TWSH (Titanium Wire Space Holder) for Biodegradable Bone Scaffold Material

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Abstract

Magnesium (Mg) and its alloys are a potential biodegradable bone scaffold materials as their biocompatibility and mechanical properties fit to natural bone. Several studies have shown that Mg alloys as biomaterials have higher mechanical properties similar to cancellous bone once composed and produced in a specific production route. Fabricating porous Mg for bone scaffold material aims to reduce the rigidity and strength of the material as well as adjusting the porous density to the original nature of bone. This leads to the formation of interconnected porosity, with physical and mechanical properties similar to cancellous bone. Therefore, this study aims to describe the production and characterization of porous Mg material for potential application as bone scaffold through powder metallurgy technique with pieces of Titanium (Ti) Wire Space Holder. Mg containing Ti pieces were compacted and sintered before immersed in hydro fluoride acid solution to form a porous Mg structure. Density and porosity, micro Vickers hardness, micro structure test, and Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) were performed to prove the existing porous structure inside Mg metal. The result showed that the inter-connected porous Mg samples had potential application as cancellous bone implant.

Keywords: p*orous Magnesium, cancellous bone, bone scaffold, space holder*

I. INTRODUCTION

Several people have recently been affected by bone defect regeneration and other organic tissue problems due to fractured or broken bones caused by accidents, hard hitting, or aging. To handle such problems, bone tissue implantation must be performed to restore function. Bone implantation is often carried out using biomedical material, commonly known as biomaterial.

Previous studies have shown that biomaterial interacts with human tissue and body fluids to treat, improve, replace, and regenerate anatomical elements of the human body or implants. The application of *biodegradable* biomaterial is aimed at eliminating the need to remove the material implanted due to its non-toxicity, biocompatible, and naturally degradable properties [1].

Porous metal has been reported to have low weight with adjustable density (Lefevbre et all, 2008). The well-combined metal and pores make the material suitable for structural and functional application (Chiras et all, 2002). The use of porous metal is very supportive for metal implants of cancellous bone structure, as it stimulates the growth of bone tissues. In addition, the material implanted in the bone acts as a scaffold or temporary structure, which can strengthen it from corrosion.

To make use of its natural degradable property, magnesium (Mg) is used as a porous metal material due to its natural degradable property in the human body [2]. Mg is a safe bone implant material as it has biodegradability, non-toxic, and supporting physical and mechanical functions properties, which are similar to the nature of bone [3].

Several studies have stated that Mg can be combined with Titanium (Ti) due to its lightness as well as chemical and mechanical nature [4]. Porous Mg is often produced from Mg powder combined with Ti (wire), as a space holder [5].

In this study, porous Mg was produced through a compacting-*sintering* method using a *squeeze casting* device. Macro-structure analysis and corrosive testing were then performed to identify corrosive acceleration. The production of porous Mg was through Ti wire corrosion process.

Density-porosity testing, micro Vickers hardness, micro-structure analysis, and Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) testing were performed to compare the properties of the product to those of porous *cancellous bone,* as a biomedical bone implant material.

II. MATERIAL AND METHOD

Fabricating porous Mg was carried out using powder combined with Ti wire (space holder) by applying compacting-*sintering* method. In addition, Ti corrosion was performed using hydrofluoride (HF) acid solution, followed by physical and mechanical assessment.

A. Device and Material

Material used was the powder of Mg AZ31 with sizes 100 µm, 200 µm, and 250 µm, Ti wire 32 GA (AWG) 200 µm, compacting-*sintering* device*,* and HF acid 1 M. Compacting-*sintering* device consisted of a hydraulic press machine with a capacity of 20 tons, *heater* coil machine with a temperature capacity of 800°C, a *unit control heater* TC4M, *dies-punch,* and argon gas, as shown in Figure 1 [6].

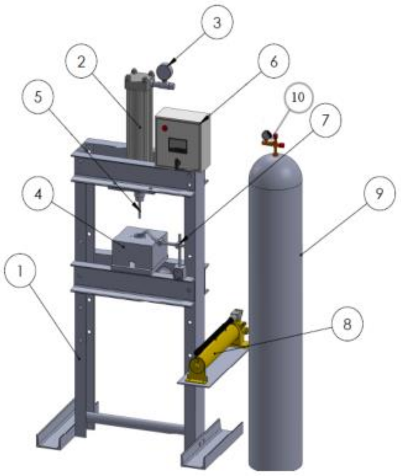
B. Method of Study

The method of study applied was based on the parameters and variables, as shown in Table 1.

Table 1. Study variables

No.	Powder Mg size	Powder Ti size	Comp. Mg:Ti	Press. (Mpa)	T (^{0}C)
1	100 µm	100 µm	3 to 1	250	400
2	200 µm	200 µm			
3	250 µm	250 µm			

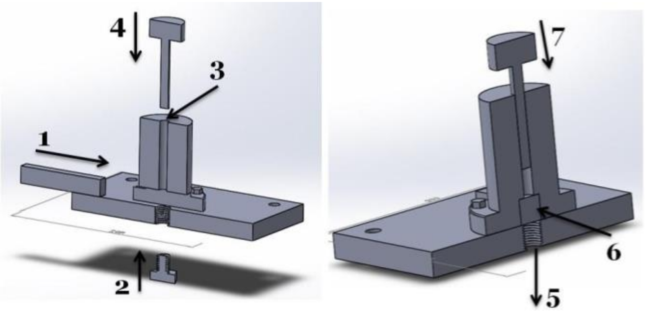
First, the assembly of the squeeze *casting* device was carried out, as shown in Figure 2. Subsequently, the powder of Mg with the sizes of 100 µm, 200 µm, and 250 µm was mixed with the powder of Ti wire of 200 µm, with a weight fraction of 3:1.



1 Hydraulic frame
2 Hydraulic tube
3 Press indicators
4 Heating unit
5 Punch
6 Heater controllers
7 Argon gas
8 Hydraulic pumps
9 Argon tube
10 Gas indicators

Figure 1. Squeeze casting technique [6]

The mixed powder was then put into *dies* and compacted at 250 MPa for 2 minutes. The sample was removed from *dies* and put again into dies with sintering at 400°C for 5 minutes, and finally the sample was removed.



1 Slide ejector; 2 Locking Bolt; 3 Inserting PM; 4 Hydraulic Pump; 5 Unlocking Bolt; 6 Ejector; 7 Pressure out

Figure 2. Procedure of Compacting-*Sintering*

III. RESULTS AND DISCUSSION

The results were presented in the form of micro-structure testing, corrosion testing, density-porosity testing, macro hardness testing, and SEM-EDX.

A. Analysis of Macro-Structure

Analysis of macro-structure was carried out to identify the spreading of Ti and the binding powder Mg at 100 µm, 200 µm, and 250 µm (Figure 3).

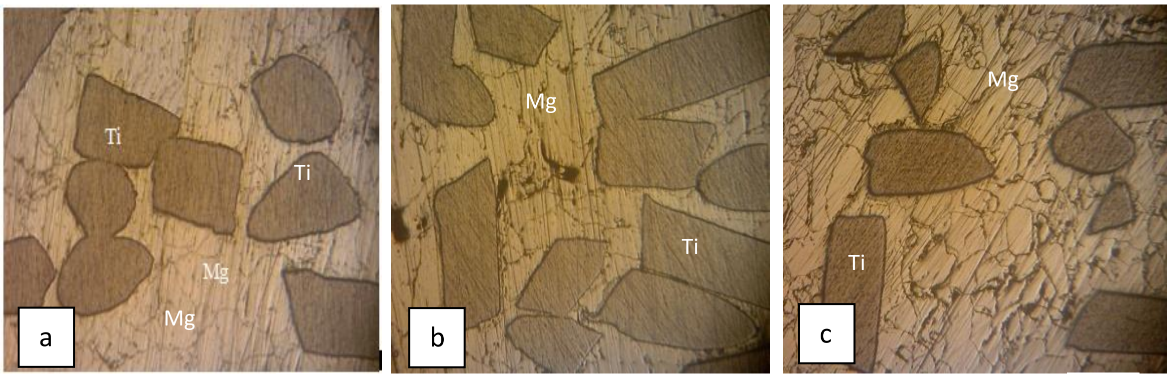


Figure 3. Macro-structure of Mg-Ti composite of (a) 100 µm, (b) 200 µm, and (c) 250 µm powder size (bar is 100 µm)

B. *Corrosive Testing*

Corrosive testing was performed to identify mass-decreasing acceleration, corrosive acceleration, and the formation of porous Mg with mass-decreasing estimation (CPR) *corrosion rate,* as shown in Figure 4. Mass specimen (gram) after corrosion of Ti, and total mass specimen at various soaking times were recorded.

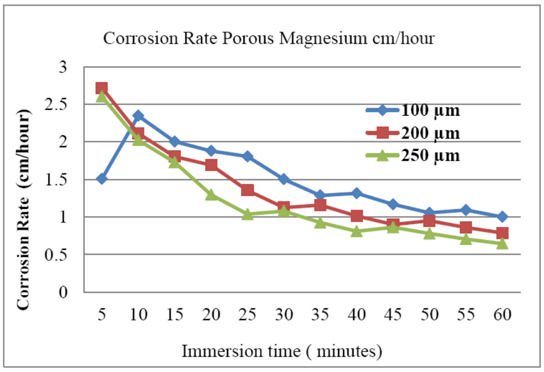
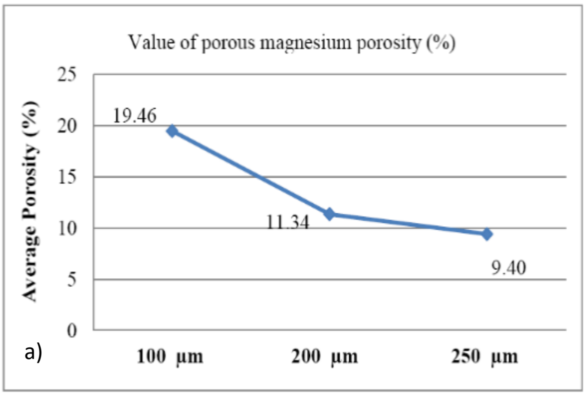


Figure 4. Mass-decreasing Ti at (a). P-Mg 100 µm, (b). P-Mg 200 µm, and (c). P-Mg 250 µm

C. *Density-Porosity Testing*

Density-porosity testing was performed to identify the density and porosity of porous Mg. The result using ASTM C373-88 standard was based on Archimedes' law. The result of density-porosity testing is shown in Figure 5.



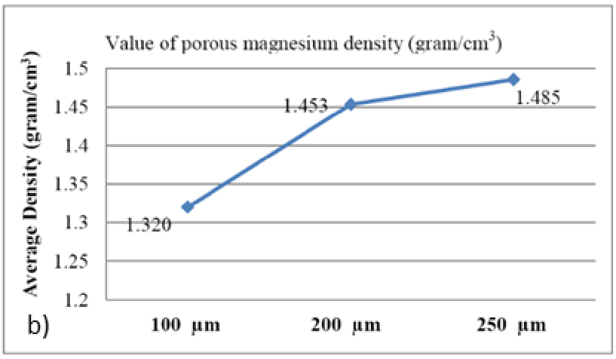


Figure 5 Graphic of density (a) and porosity (b) over Mg powder size

D. *Micro Vickers Hardness Testing*

Hardness testing was carried out to identify the value of hardness with the JIS B 7725:2010 testing standard. The procedure was performed with a weight of 0.1 N pressure for 12 seconds and 6 times testing with different position. The result of hardness testing is shown in Figure 6.

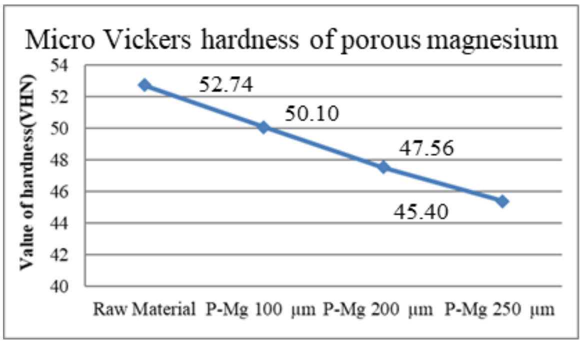


Figure 6. Micro hardness value of Mg-Ti composite with 100 µm, 200 µm, and 250 µm Mg powder size

E. *Analysis of Micro-structure*

Micro-structure analysis aimed to identify the structure and metallography of the casting Mg-Ti composite product. The micro-structure of Mg-Ti composite before the degradation process of Ti space holder is shown in Figure 7.

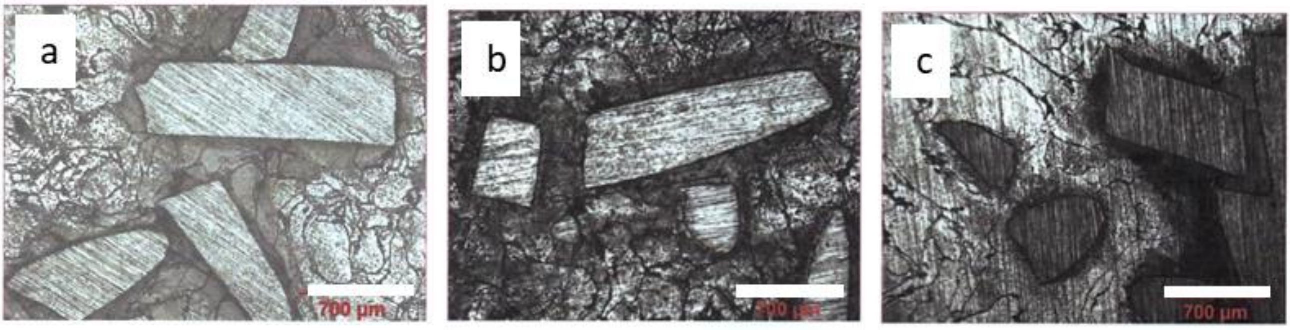


Figure 7. Structure of Micro-Specimen (a). P-Mg 100 µm, P-Mg 200 µm and (c). P-Mg 250 µm

F. SEM-EDX

SEM-EDX testing is purposed to identify micro-structure and the properties of structure composites of Mg-Ti using sample P-Mg 200 µm after corrosion process is shown on Figures 8 to 10.

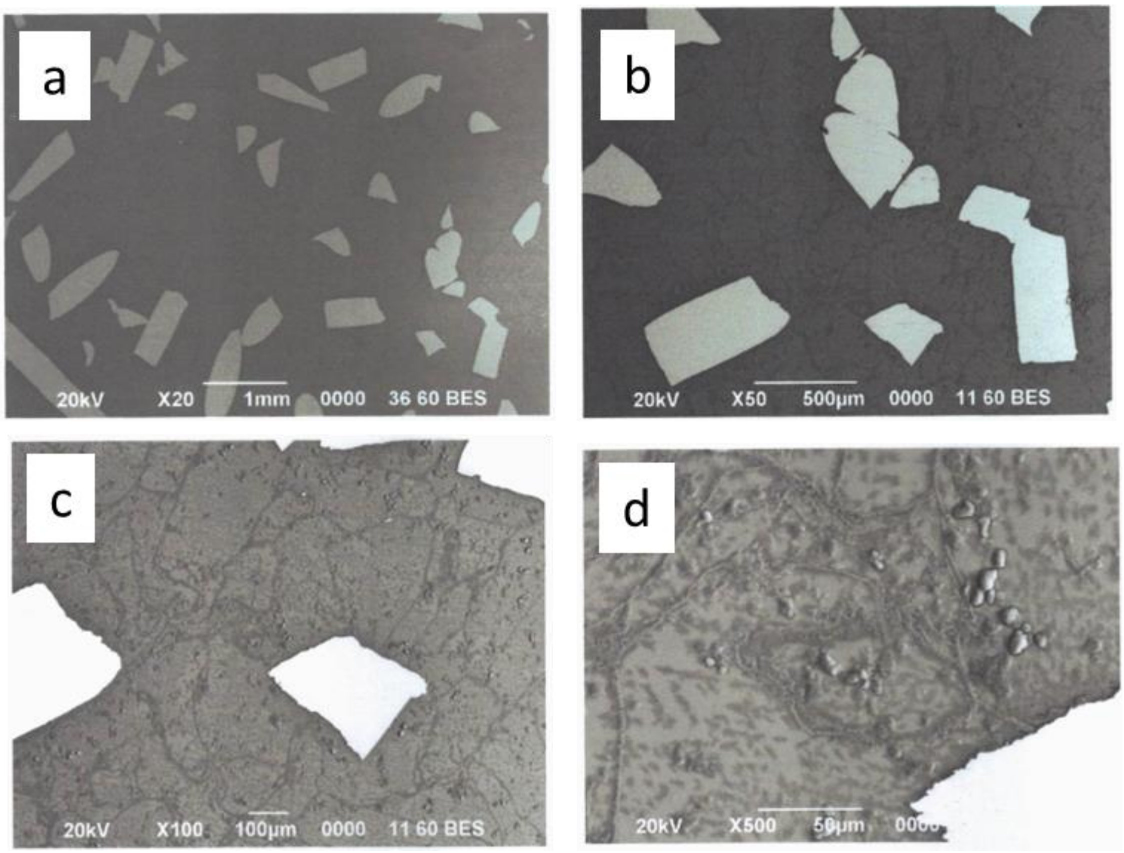


Figure 8. Microscope photo of electron P-Mg 200 µm enlarging (a) 20x, (b). 50 x, (c) 100x, and (d) 500x

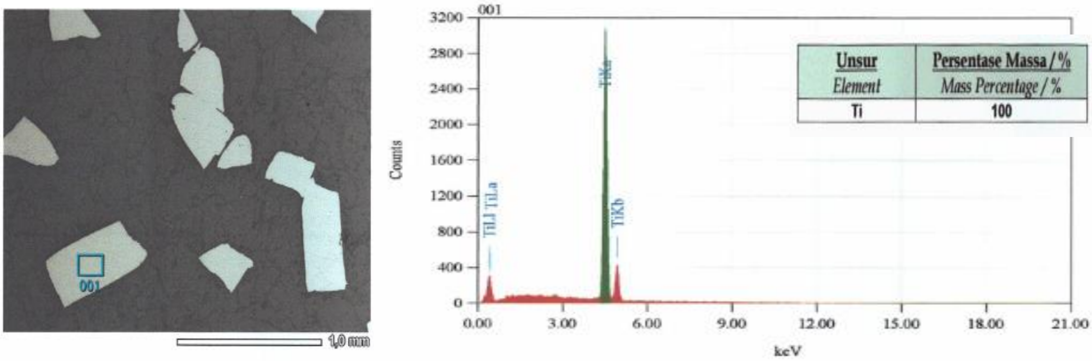
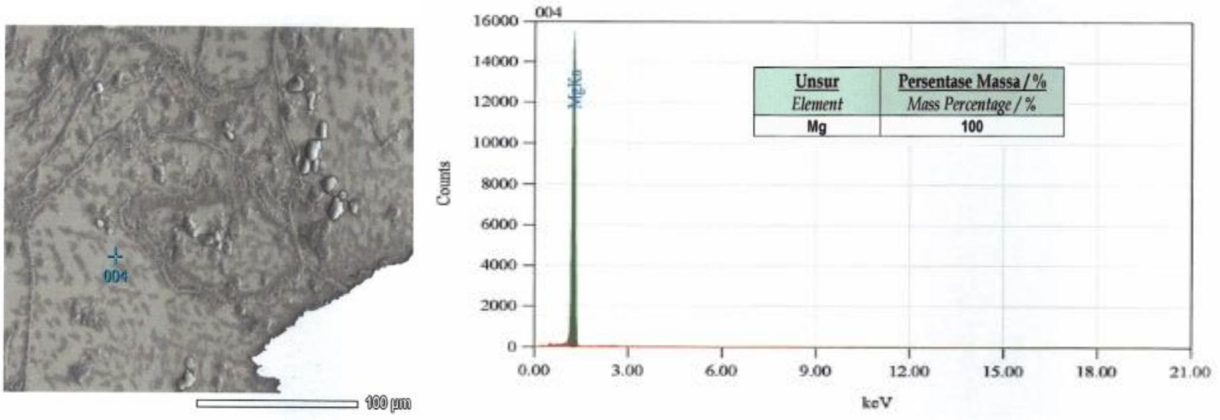


Figure 9. Qualitative analysis of P-Mg 200 µm, bar is 1.0 mm



Unsur Element	Persentase Massa / % Mass Percentage / %
Mg	100

Figure 10. Qualitative analysis of P-Mg 200 µm, bar is 100 µm

G. DISCUSSION

The result of fabricating and testing porous Mg from macro-structure analysis showed that the spreading of Ti wire settled as sediment in the middle and bottom part of the specimen, as it was caused by the higher density of Ti compared to Mg. Furthermore, corrosion testing showed that a mass-decreasing specimen occurred due to corrosion of Ti using fluids of HF fluids. Specimen of P-Mg 250 µm indicated that the specimen had the highest corrosion acceleration rate. This was 0.08 gram/hour, and its corrosion acceleration rate was 1.001 cm/hour. The result of density-porosity testing showed that specimen P-Mg 250 µm had a value of density of 1.453 g/cm^3 and a value of porosity of 11.34 %. However, the value was less than cancellous bone porosity, which was 15-30 %. This suggested that the bigger the size of Mg powder combined with Ti (wire) powder, the lower the density, and the higher the porosity.

The result of micro hardness testing showed that the value of hardness of raw material 52.74 VHN, P-Mg 100 µm was 50.10 VHN, P-Mg 200 µm was 47.56 VHN, and P-Mg 250 µm was 45.40 VHN. Those values were much higher than the value of cancellous bone [3,6]. The result of micro-structure testing shows that the binding of Mg powder was very satisfactory through the compacting-sintering process, yet the remains found in Ti specimen showed that Ti corrosion only occurred at the surface of the specimen. Based on the result of SEM-EDX testing, the metallography specimen was in good condition when compared to others [5,7].

EDX result showed a high concentration of Ti because HF acid liquid could not penetrate inside the specimen. Therefore, the corrosion process of Ti wire space holder was not the same as in the surface area. This was due to Ti powder combined with Mg powder did not spread well, making it difficult to allow Ti corrosion in its inner part of the specimen, as connectivity among Ti powder did not occur.

IV. CONCLUSIONS

In conclusion, the corrosion of Ti during testing indicated that specimen P-Mg 250 µm had a high corrosive rate, which was 0.07 gram/hour, and its corrosion acceleration rate was 0.7916 mmpy. Density-porosity testing showed that a specimen with a powder size of 250 µm had density of 1.453 g/cm^3 and porosity of 11.34 %, which was close to *cancellous bone* of 15-30%. Further hardness testing showed that Mg-Ti composite still had a higher hardness number when compared to natural bone (about 10.20 VHN). SEM-EDX results indicated potential production of Mg-Ti composite for porous degradable bone scaffold using Mg powder metallurgy.

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