

## Effect of Space Holders on Fabrication of Porous Titanium Alloy-Hydroxyapatite Composite through Powder Injection Molding

(Kesan Pemegang Ruang terhadap Pembentukan Komposit Aloji Titanium-Hidroksiapatit Berbusa melalui Pengacuan Suntikan Serbuk)

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### ABSTRACT

*Powder injection molding (PIM) is able to produce porous titanium alloy/hydroxyapatite composite through the space holder technique. Thermal debinding and sintering processes were the main challenges due to different properties of metal and ceramic in producing such composite. This study focused on the effect of different space holders on the physical and mechanical properties of debound and sintered porous titanium alloy/hydroxyapatite composite. The feedstock is containing of 80 wt. % of titanium alloy/hydroxyapatite with 20 wt. % of space holders such as sodium chloride (NaCl) and polymethylmethacrylate (PMMA), respectively. The binders were then removed from the injected samples by two stages of debinding; solvent and thermal debinding. The sintering was performed at three different temperatures 1100°C, 1200°C and 1300°C at a heating rate of 10°C/min and holding time of 5 h. It was found that the samples containing PMMA space holder was fractured after sintering. While, the samples containing NaCl space holder successfully formed pores and not fractured. At sintering temperature of 1300°C, the density, compressive strength and porosity volume percentages for the sintered sample containing NaCl space holder were 3.05 g/cm<sup>3</sup>, 91.7 MPa, and 11.9 vol%, respectively.*

*Keywords: Hydroxyapatite; metal foam; powder injection molding; space holder; titanium alloy*

### ABSTRAK

*Pengacuan suntikan serbuk (PIM) boleh menghasilkan komposit aloji titanium/hidroksiapatit berbusa dengan menggunakan teknik pemegang ruang. Proses bagi penyahikatan dan persinteran merupakan cabaran utama disebabkan sifat bahan logam dan seramik yang berbeza dalam penghasilan komposit tersebut. Fokus kajian ini adalah kesan berbeza pemegang ruang terhadap sifat fizikal dan mekanik penyahikatan serta persinteran komposit aloji titanium-hidroksiapatit berbusa. Bahan suapan mengandungi 80 % bt. aloji titanium/hidroksiapatit dengan 20 % bt. pemegang ruang seperti sodium klorida (NaCl) dan polimetilmetakrilat (PMMA). Bahan pengikat kemudian disingkirkan daripada sampel yang disuntik melalui dua peringkat penyahikatan; larutan dan penyahikatan terma. Persinteran dijalankan pada tiga suhu yang berbeza 1100°C, 1200°C dan 1300°C pada kadar pemanasan sebanyak 10°C/min dan masa pegangan selama 5 jam. Didapati sampel yang mengandungi pemegang ruang PMMA hancur selepas persinteran. Manakala, sampel yang mengandungi pemegang ruang NaCl berjaya menghasilkan liang-liang berbusa dan tidak hancur. Pada suhu persinteran 1300°C, ketumpatan, kekuatan mampatan dan peratusan isi padu keliangan sebanyak 3.05 g/cm<sup>3</sup>, 91.7 MPa, dan 11.9 vol%.*

*Kata kunci: Aloji Titanium; hidroksiapatit; logam berbusa; pemegang ruang; pengacuan suntikan serbuk*

### INTRODUCTION

The use of titanium alloys, cobalt-chromium alloys, and stainless steel began in the 1960s (Best et al. 2008). Titanium and its alloys are widely used for bone implants due to their strong mechanical properties and high chemical resistance. Ti-6Al-4V has a higher Young's modulus compared to human bones, which leads to stress shielding (Nomura et al. 2010). It also has poor biocompatibility and unable to promote the growth of natural tissue (Thian et al. 2001). Hydroxyapatite (HA) is a ceramic material, which has poor mechanical properties and limited for high load applications. However, it has a chemical structure that is similar to human bone and able to stimulate tissue growth (Chu et al. 2006). The Ti-

6Al-4V and HA composites are able to form a biomaterial with good biological and mechanical properties as well as good corrosion resistance (Thian et al. 2002). Powder Metallurgy (PM) or Powder Injection Molding (PIM) has been used in the fabrication of metal composites for biomedical implants application (Deing et al. 2014; Jurczyk 2012). In biomedical implants, the concept of preparing porous structure is developed due to enhance the ingrowth of tissue into the pores. Therefore in PM, there are various techniques that are able to fabricate porous composite such as gas entrapment (Murray & Dunand 2003), conventional pressing and sintering (Oh et al. 2003) and space holder (Bram et al. 2002; Manonukul et al. 2010; Raza et al. 2014; Wen et al. 2001).

Sodium chloride (NaCl) and polymethylmethacrylate (PMMA) were chosen as the space holders in this experiment. NaCl is widely available in the market which is low cost, low toxicity, soluble in water where it can be easily eliminated through the solvent debinding process (Patnaik 2003). In addition, NaCl provides good adhesion and cell spread during the *in vivo* process of porous NiTi (Kohl et al. 2008). Torres et al. (2012) found that the important factors in controlling the dissolution rate of porosity and Young's modulus were salt content, immersion time and water temperature. The other space holder; PMMA, was chosen due to its lightweight and good impact strength. Nishiyabu et al. (2005) initially demonstrated the potential of PMMA as space holder in producing a macropores structure with PM. Indeed, it was found that PMMA is suitable as a space holder where the acceptable level of oxygen and carbon content based on the ASTM standards was obtained for porous Titanium Alloy after sintered at 600°C (Engin et al. 2011).

Recent research has shown that there are challenges in combining two or more powders having different properties such as size, materials and thermal coefficient. Ti-6Al-4V and HA composites have been studied for several years due to their capabilities in biomedical implantation (Arifin et al. 2015; Thian et al. 2002). However, combining these two materials is complicated due to different thermal expansion coefficient that results to residual stress which will form crack on the sintered part (Pattanayak et al. 2011; Thian et al. 2002). In addition, since porous structure of Ti-6Al-4V/HA composite is needed, the cell size and its distribution had to be controlled to ensure such structure is similar to human bone. The main objective was to analyse the effects of debinding and sintering parameters on Ti-6Al-4V/HA composite containing NaCl and PMMA space holders using PIM. A preliminary study of a porous structure for human bone was expected in this study.

#### MATERIALS AND METHODS

The gas atomized Ti-6Al-4V and HA were manufactured by TLS Technik GmbH, Germany and Sigma, Aldrich, respectively. NaCl and PMMA were chosen as the space holders. NaCl proved to be a better choice as a temporary space holder because it can be removed easily through dissolution in water and non-toxic, which is suitable for bio-implant application (Jha et al. 2013). Table 1 shows the size and density of the raw materials used in this study. A

multi-component binder system consists of polyethylene glycol (PEG), poly-methyl methacrylate (PMMA) and stearic acid (SA) was used. PEG binder is an environmentally friendly material and can be dissolved in water easily. PMMA acted as the backbone binder due it provides enough strength for handling purpose and can be removed from the injected part in a short time (Omar & Davies 2001). Meanwhile, stearic acid worked as a surfactant for powder wetting (Ibrahim et al. 2009).

The morphological analysis was carried out for Ti-6Al-4V, HA and space holders by using FESEM Machine Model Zeiss Merlin Compact. The XRD analysis was performed using a diffractometer with a radiation of over a 2-theta range at 10-90 degrees using a Bruker D8 machine. Figure 1 shows the FESEM images of powders and space holders. It was observed that the particle shape for Ti-6Al-4V, HA, PMMA and NaCl is spherical, irregular, spherical and rectangular, respectively.

Differential scanning calorimetry (DSC) analysis was conducted using DSC 1 Mettler Toledo to measure the melting temperature of the binder components prior preparing the feedstock as according to ASTM D3418 (standard test method for transition temperatures and enthalpies of fusion and crystallization of polymers). The STA 449 F3 NETZSC thermal gravimetric analyser (TGA) was used to measure the decomposition temperature of the binder components so that the temperatures for mixing and debinding can be designed based on ASTM E1131 (standard test method for compositional analysis by thermogravimetric). A heating rate of 10°C/min was used for both tests from the ambient temperature to 700°C in nitrogen condition.

A dry ball milling machine was used to mix 90 wt. % Ti-6Al-4V and 10 wt. % HA at a speed of 100 rpm for 2 h, as reported by Arifin et al. (2015). 80 wt. % Ti-6Al-4V/HA was then mixed with 20 wt. % of space holders using a Brabender Plastograph® EC plus machine at room temperature at a speed of 40 rpm for 2 h to maintain the space holder structure where it helps to form pores during sintering. The mixture of 60 vol.% Ti-6Al-4V/HA-space holder was then mixed with 40 vol.% of a PEG-based binders system consists of 70 vol.% PEG, 25 vol.% PMMA and 5 vol.% SA using the brabender machine at 160°C and at the speed of 40 rpm for 2 h. The prepared feedstock was molded into a tensile bar by using a table-top injection moulding machine (model DSM Xplore). The molding parameters were in accordance with previous research done by Raza et al. (2014), as shown in Table 2.

TABLE 1. Characterization of raw materials

Raw Material	Type	Measured Particle Size ( $\mu\text{m}$ )	Density ( $\text{g}/\text{cm}^3$ )
Ti-6Al-4V	Gas Atomized powder	19.61	4.43
Hydroxyapatite (HA)	Powder	5.341	3.132
NaCl	Crystalline	381.396	2.165
PMMA	Crystalline	160	1.19

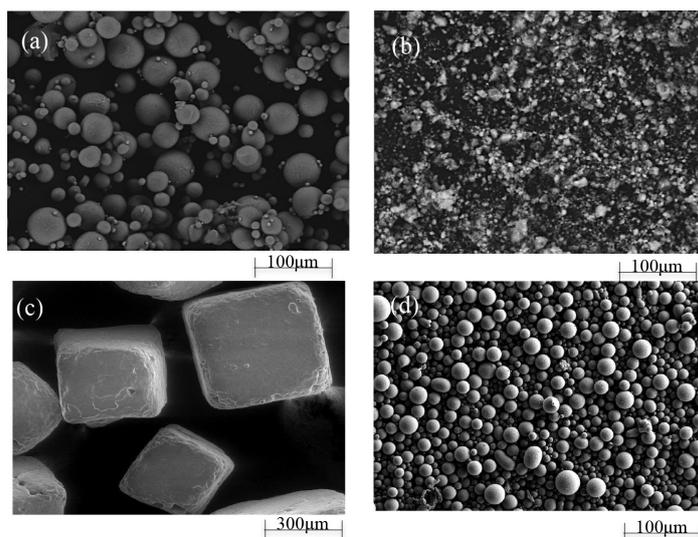


FIGURE 1. FESEM micrographs of (a) Titanium Alloy and (b) HA (c) NaCl (d) PMMA

TABLE 2. Injection molding parameters

Injection parameters	Value
Injection pressure	8 bar
Compression pressure	8-10 bar
Holding pressure	8-10 bar
Melt temperature	160±5°C
Mold temperature	55±5°C
Injection time	8 s

A two-stage debinding process starting with solvent debinding followed by thermal debinding was carried out. Solvent debinding was carried out in a distilled water bath at 60°C for 6 h to extract PEG. In thermal debinding, PMMA was removed from the solvent extracted samples by heating the samples at 500°C at a heating rate of 3°C/min in an argon atmosphere with a holding time of 1 h, using a split debinding furnace (VTC-500 4TSF). The debound samples were then subjected to sintering processing a tube furnace model HTF-15/200-60 at three different

temperatures; 1100°C, 1200°C and 1300°C at a heating rate of 10°C/min with a holding time of 5 h. The density, compressive strength and morphologies of the sintered samples were observed. The density test was performed using the Archimedes principle according to MPIF 42 (determination of density of coated or sintered powder metallurgy products). The compression tests were carried out on the Universal Testing Machine Model Z100 based on MPIF 61 (determination of the compressive yield strength of powder metallurgy materials).

## RESULTS AND DISCUSSION

### X-RAY DIFFRACTION (XRD) OF POWDERS

Based on the X-Ray diffractogram shown in Figure 2(c), it can be observed that the HA peaks show low intensity where the crystallisation of Ti-6Al-4V-6. Powder is dominant when mixing with 10 wt. % HA. In Figure 2(e), it can be observed that peaks appeared on the Ti-6Al-4V/

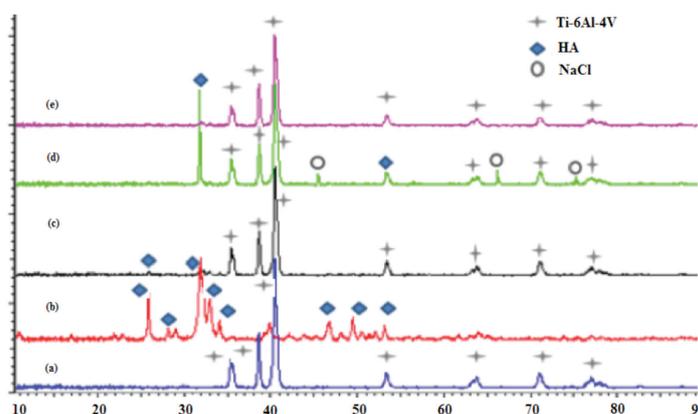


FIGURE 2. XRD result (a) Ti-6Al-4V (b) HA (c) Ti-6Al-4V /HA (d) Ti-6Al-4V/HA- NaCl (e) Ti-6Al-4V/HA- PMMA

HA-PMMA is similar pattern to Ti-6Al-4V/HA composite peaks. Meanwhile, Figure 2(d) shows that the sample consist of Ti-6Al-4V/HA-NaCl composed of three peaks; the Ti-6Al-4V, HA and NaCl peaks.

MORPHOLOGY OF GREEN SAMPLE

Figure 3 shows the morphology images of green samples containing (a) PMMA and (b) NaCl space holders. It was observed that the surface of green sample containing PMMA space holder looked denser and well dispersed compared to that of NaCl space holder. This is due to not all NaCl melted which resulted to poor wetting on the surface of powder particles.

THREE-POINT BENDING TEST OF GREEN SAMPLE

The comparison between PMMA and NaCl space holders on the flexural strength, modulus and elongation is presented in Figure 4. Samples containing PMMA space holder showed better performance compared to that of NaCl space holder. This is because PMMA was presented in a larger quantity and acted as the backbone binder compared to NaCl.

Therefore, green samples containing PMMA have higher resistance towards deformation and breakage.

SOLVENT DEBINDING OF GREEN SAMPLE

Figures 5 and 6 shows the SEM micrographs for both samples containing different space holders where samples with NaCl space holder formed more pores compared to those with PMMA space holder. This is due to the removal of PEG and NaCl during the solvent debinding, which resulted to significant formation of pores. After 30 min of removing NaCl, as shown by Figure 5(a), large holes were observed. Such formation significantly appeared after 150 min due to more PEG and NaCl components were removed, as shown in Figure 5(c). Based on Figures 6, it was observed that initially, the sample looked dense and wet because PMMA space holder was presented in a large quantity. Manonukul et al. (2010) reported when more PMMA is added, the wettability with less viscosity of feedstock is higher. After 150 min, the surface of sample formed micropores and seemed to be less dense. In addition, the pores formed in the samples containing

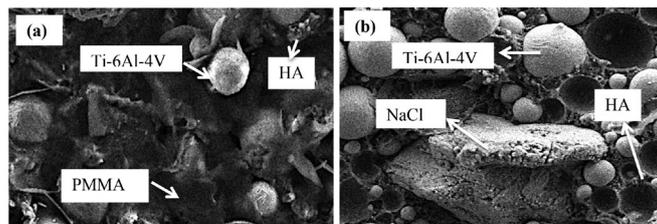


FIGURE 3. SEM micrographs for green sample consisting (a) PMMA and (b) NaCl space holder

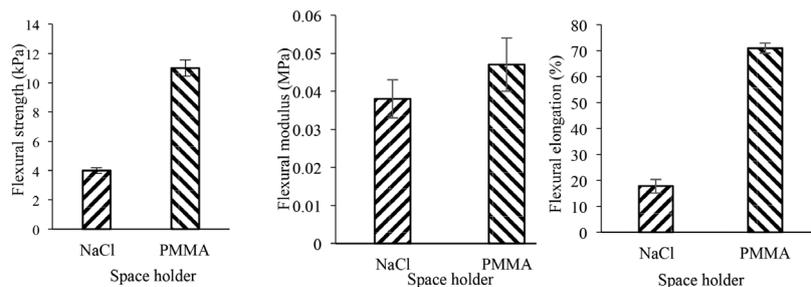


FIGURE 4. Comparison of (a) flexural strength (b) flexural modulus and (c) flexural elongation for green part contain different types of space hold

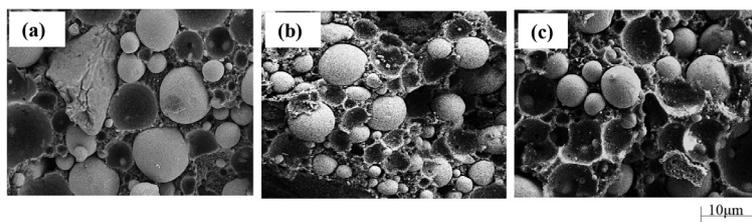


FIGURE 5. SEM micrographs for sample containing NaCl space holder after (a) 30 min (b) 150 min and (c) 360 min solvent extraction

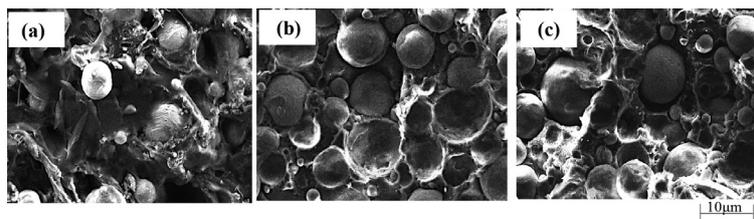


FIGURE 6. SEM micrographs for sample containing PMMA space holder after (a) 30 min (b) 150 min and (c) 360 min solvent extraction

PMMA space holder are smaller compared to those containing NaCl space holder.

#### THERMAL DEBINDING AND SINTERING ANALYSIS

Samples containing NaCl space holder was successfully debound, however, was observed on the debound samples containing PMMA space holder. Swelling is a known defect that commonly occurred during thermal debinding process (Enneti et al. 2012), due to improper decomposition of binders. Large amount of PMMA resulted to severe swelling. In addition, the heating rate of 3°C/min may have been too high for the samples containing PMMA space holder. Such swelling was reported when a high heating rate was used to decompose 50% PMMA with extra dwell time (Manonukul et al. 2010). Therefore, a lower heating rate is necessary to refrain the formation of such defect. Figure 7 shows that the sample containing PMMA space holder fractured at minimum sintering temperature of 1100°C. Pachauri and Hamiuddin (2016) reported that the quality of debound part affects the quality of the sintered part. Such influence was proven based on the debound PMMA space holder samples that have defects. Samples containing NaCl space holder were successfully sintered at three different temperatures; 1100°C, 1200°C and 1300°C and remained in good condition.



FIGURE 7. Physical deformation after sintering temperature at 1100°C (a) Ti-6Al-4V/HA with NaCl (b) Ti-6Al-4V/HA with PMMA

Morphology analysis was conducted on Ti-6Al-4V/HA samples containing NaCl space and presented in Figure 8 where the samples were sintered at different temperatures; (a) 1100°C, (b) 1200°C and (c) 1300°C. It was observed that more necking occurred at 1300°C and relatively larger than the sample sintered at 1100°C. Thian et al. (2002) reported Ti-6Al-4V/HA sintered at 1100°C showed the pores are interconnected. In addition, sufficient necking regions between particles were formed while maintaining open pore structure. Such structure was formed due to the decomposition of the binder system (Weil et al. 2006).

Based on Table 3, the volume percentage of pores formed was inversely proportional to the increasing

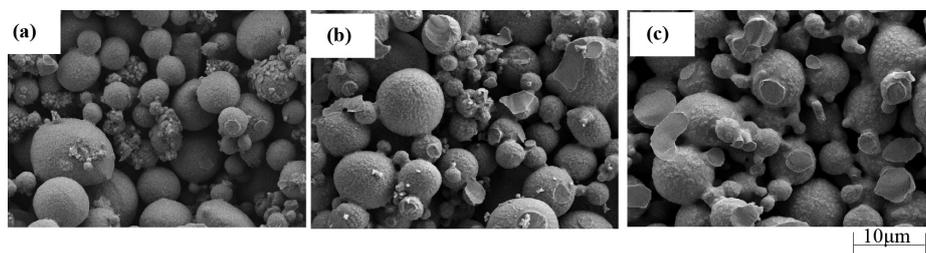


FIGURE 8. SEM images for sample sintered at (a) 1100°C, (b) 1200°C and (c) 1300°C

TABLE 3. Volume percentage of pores at three different sintering temperatures Ti-6Al-4V/HA-NaCl

Sintering temperature (°C)	Porosity volume percentage (%)
1100	20.8 ± 0.3
1200	18.3 ± 0.1
1300	11.9 ± 0.3

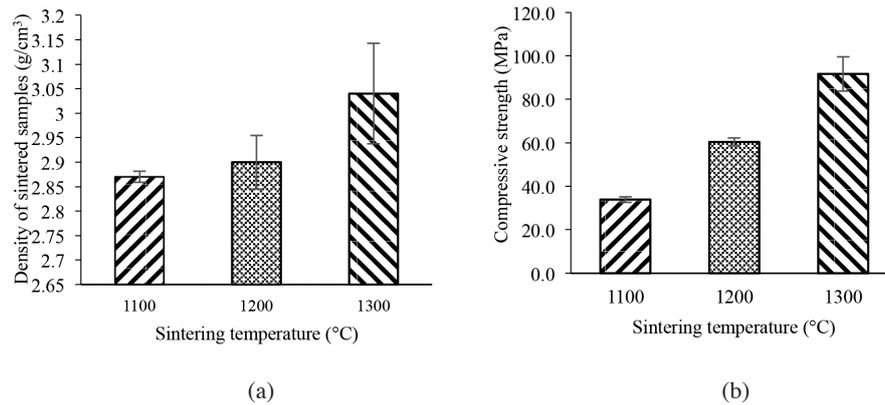


FIGURE 9. (a) Density and (b) Compressive strength of Ti-6Al-4V/HA-NaCl samples sintered at 1100°C, 1200°C and 1300°C

sintering temperature, as referred to the previous research by Ahmad et al. (2010). The formation of pores was the least when the highest sintering temperature of 1300°C was applied. This is due to the enlargement of necking when the sintering temperature increased. Such enlargement covers the pores that formed between the particles. According to Raza et al. (2015), pores were reduced and the enlargement of necking was found to be aligned with the increasing holding time of 4 to 5 h, when titanium alloy/HA composite was sintered at 1300°C. Arifin (2015) found that necking was clearly observed at 1300°C compared to 1100°C and 1200°C. Figure 9(a) shows the density of sintered samples at different sintering temperatures. As expected, the highest density was given by the sample sintered at 1300°C due to more necking was formed and enlarged (Ji et al. 2001). Therefore, the formation of necking eventually increased the density of sample and reducing the pore size and its volume. Meanwhile, the compressive strength of the sintered samples increased as the sintering temperature increased, as shown by Figure 9(b). Such finding is similar as reported by Arawi et al. (2012). The density and strength of titanium alloy particles were directly proportional with the sintering temperature (German 1996); therefore higher compressive strength is needed to break the sample.

#### CONCLUSION

Poor thermal debinding process will produce sintered samples with poor mechanical properties. Such hypothesis was proven by the samples containing PMMA space holder where appropriate heating rate and holding time for binder removal are needed. However, porous Ti-6Al-4V/HA composite containing NaCl space holder was successfully fabricated and the porosity for such composite decreased from 20.8 to 11.9 vol% when sintered from 1100°C to 1300°C. 20.8 to 11.9 vol% porous Ti-6Al-4V/HA-NaCl composite with density and compressive strength of 3.05 g/cm<sup>3</sup> and 91.7 MPa was obtained when sintered at 1300°C.

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