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Early Investigation in Porous Aluminum Development via Powder Metallurgy Technique

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Abstract:

Porous aluminum (Al) has gained significant attention owing to its peculiar properties including lightweight, high strength to weight ratio as well as excellent impact energy absorption. In spite of these advantages, early investigation prior to satisfactory porous Al development is rarely reported in open literature. Therefore, the current study was initiated to perform an early investigation on porous Al development via powder metallurgy technique. In this study, 30 wt. % of polymethylmetacrylate (PMMA) was introduced as a temporary space holder to create a desirable porous structure. The effects of milling time on the morphology and densities of elemental powder mixture and porous Al were evaluated systematically. Prolong milling time to 12 hr resulted in smaller elemental powder particle size with improved powder mixture agglomeration along with a broad distribution of powder mixture particle size. In line with this, a reduction in peaks intensity was demonstrated by x-ray diffraction (XRD) analysis when the milling time was increased from 4 hr to 12 hr, confirming powder particle size reduction. Apart from these, a decreased in green densities from 1.742 g/cm3 to 1.736 g/cm3 were recorded with increasing milling time from 3 hr to 9 hr in which a slight increased to 1.743 g/cm3 was recorded with after 12 hr milling. On the other hand, maximum milling time of 12 hr reduced the total porosity of porous Al from 31.75% to 30.45% whereas its density was found to increase from 1.465 g/cm3 to 1.471 g/cm3, respectively. Therefore, it can be emphasized that milling time of 12 hr was sufficient to design a desirable porous structure with satisfactory results of green and sintered densities as well as porosity level.

Keywords: *Milling Time, Porous Al, Powder Metallurgy, PMMA*

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I. INTRODUCTION

The fact that a reduction of density in a porous material reduces its overall weight in general, and the impact energy absorption of porous metals in particular, was acknowledged not a long time ago [1]-[4]. The extensive plateau region in the stress strain curve of porous metals is the main contributor for excellent characteristic of energy absorption capacity of porous metals thus meticulous control of porous metals processing is essential.

It has also been discovered that lightweight porous metals have potentiality in minimizing fuel consumption and hazardous emissions to the environment when applying in automotive applications [1]-[6]. In addition to these, porous metals offer other practical advantages, such as high strength to weight ratio, high specific surface area, high thermal conductivity as well excellent design flexibility and tailor-ability [1]-[8]. Apart from automotive applications, the attributes of porous metals also allow themselves to other areas including construction, aerospace and marine. In this context, the basic traits of porous metals namely relative density, cell structure, wall thickness, strut

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integrity, and cell morphology homogeneity determine their appropriate applications [1]-[8]. As yet, the processing routes of porous metals can be categorized into four main techniques known as electrodeposition, vapour deposition, liquid state processing and solid state processing [1]-[6]. The latter technique delivers comparatively lower level of distortion and residual stress generation in comparison to other processing techniques available [1]-[3]. Moreover, sufficiency of reproducibility of porous metals owing to the well control of processing parameters as well as absence of undesirable chemical reactions at the metal and reinforcement interface can also be accomplished by solid state processing [1]-[4]. In the recent study of Medina Ramirez, Vintila and Drew (2019), aluminum (Al) alloy foam with 18% to 80% porosity level via dolomite space holder decomposition was successfully fabricated by powder metallurgy technique based on solid state route. In another study conducted by Papantoniou, Markopoulos and Manolakos (2018), the average macro-porosity between 63% to 65% of Al foam was accomplished with the aid of saccharose crystals space holder through powder metallurgy technique. Moreover, copper (Cu) foam with different pore sizes and porosities were obtained using acrawax space holder was documented in a study of Sharma, Modi and Kumar (2018). These show that powder metallurgy technique based on solid state process is effective in developing porous metals with controlled properties. This technique comprises of mixing, compaction and sintering stages in which each stage plays an important role to ensure desirable final products. Moreover, in this technique, the porous structure is commonly designed by utilizing the use of space holder material or porous structure creator such as polymethymetacrylate (PMMA), sodium chloride (NaCl), carbamide (urea), calcium carbonate ($CaCO_3$), titanium hydride (TiH_2) and saccharose (sugar) [1]-[7].

The volume and morphology (size and shape) of these space holder material can be manipulated in creating different geometrical structure of porous metals. It is important to note that proper identification of space holder material to be combined with the base metal is fundamental to ensure unwanted physical and chemical reaction between these two materials. Thus, space holder material with reliable chemical stability is preferable in obtaining resultant porous metals with excellent quality traits. On the other hand, the configuration of porous structure in terms of open-celled structure or closed-cell structure often depending on the content of the space holder material added. Sharma, Gupta, Modi and Prasad (2013) reported that the transformation of closed-cell structure to interconnected or open-celled structure could be realized with the addition of space holder material of at least 50%. Similar finding was also revealed in the studies of Dabrowski, Swieszkowski, Godlinski, and Kurzydlowski (2010) as well as Bhattarai et al. (2008) in which open-celled structure with interconnected passage could be created with increasing the space holder content of minimum 60%. The total porosity to be designed in the resultant porous metals however, rely on its usage and intended applications. Despite of the advantages and uniqueness of porous metals, early investigations especially involving mixing of metallic powders during early stage processing prior to porous Al development with good quality properties are rarely reported in open literature. This is especially true for designing porous metals with closed-cell structure. Therefore, current study was initiated to produce closed-cell porous Al with medium porosity of 30 wt. % via powder metallurgy technique which focusing on the effects of milling time and sintering temperature on the density, porosity as well as compressive properties of the resultant porous Al.

II. EXPERIMENTAL

A. Raw Materials Selection

Aluminum (Al), magnesium (Mg) and tin (Sn) powders of 99.9% purity with an average particle size of 45 μm were introduced as the base metal. Mg and Sn powders at fixed content of 1.5 wt. % and 2.5 wt. % were added as sintering additives to promote liquid phase sintering of Al. On the other hand, polymethylmetacrylate (PMMA) particles of 99.9% purity with an average particle size of 150 μm was incorporated as a space holder or porous agent at fixed content of 30 wt. % prior to porous structure development whereas safe oil of low sulfur content (CLE) was added as a liquid binder to aid

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homogenization of powder mixture during milling. These starting materials were purchased from NovaScientific Resources (M) Sdn Bhd except for PMMA particle and CLE safe oil which were bought from Sigma Aldrich (M) Sdn Bhd and JX Nippon Oil and Energy, Japan. In this study, Al powder particles were examined to have spherical particle shape with some irregularity was detected while Mg and Sn powder particles were observed to have irregular particle shape as illustrated in Fig. 1 (a-c). Moreover, PMMA space holder particle were identified to be completely spherical in shape as seen in Fig. 1 (d).

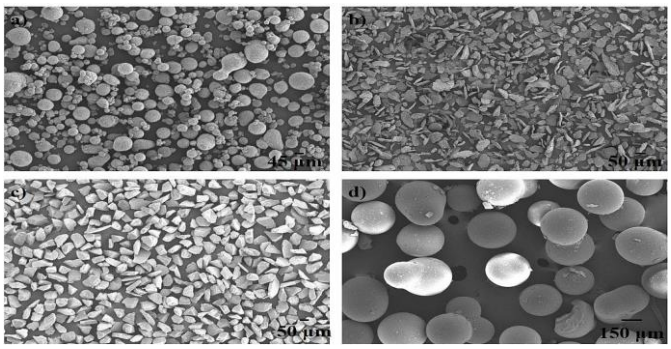

Fig. 1. Microstructure of (a-c) Al, Mg and Sn powder particles and d) PMMA space holder particle

B. Porous Al Preparation

Solid state processing based on powder metallurgy technique was implemented to design porous Al as graphically illustrated in Fig. 2. In this technique three milling processes were introduced to initially mix the base metal powders of Al, Mg and Sn at different milling time of 12 hr by table ball milling. Ball milling medium of zirconia was applied with balls to powder ratio of 1 to 7 and the resultant base metal powders mixture was known as elemental powder mixture. Then, second milling of PMMA space holder particle with liquid binder was performed for 1 h followed by final milling of elemental powder mixture and PMMA particle and liquid binder mixture for 2 hr in which known as final powder mixture. Compaction of final powder mixture at 250 MPa pressure proceeded to prepare final powder mixture compact of 10 mm diameter. Finally, sintering process took place at fixed sintering temperature and sintering time of 580 °C and 2 hr prior to porous Al development. Argon (Ag) gas was supplied throughout the sintering process to minimize contamination of porous Al body. The resultant porous Al was then washed using acetone to remove impurities before characterizations.

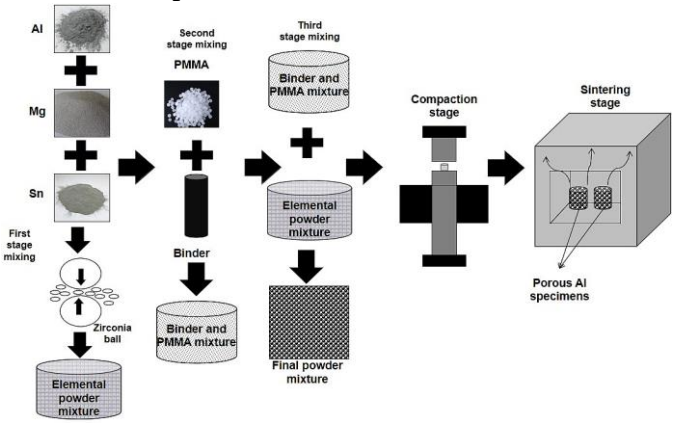

Fig. 2. Graphical illustration of Porous Al development via powder metallurgy technique

III. RESULTS AND DISCUSSION

A. Morphology of Elemental Powder Mixture After milling For Different Hour

The morphology of elemental powder mixture after milling process are shown in Fig. 3 (a-d). Theoretically, extreme plastic deformation of powder particles during milling resulted in notable morphological changes in terms of powder particle size and shape [9]-[12]. In this milling process, two important phenomenons often took place namely as fracturing and cold welding prior to achieve a steady state condition [13]-[15]. It is observable that early milling of 3 hr resulted in larger and coarser powder mixture particles size of around 49 µm which were bigger than the size of starting powder particles as seen in Fig. 3 (a). This can be attributed to the predominant welding mechanism as well as the fact that soft nature of starting Al matrix increased the tendency of these particles to weld together thus forming larger particles. Moreover,agglomeration of powder mixture particles in a few areas were also apparent at this stage hence contributed to bigger size of powder mixture particles. After 6 hr of milling, the powder mixture particles became smaller and finer with an average powder mixture particle size of 32 µm. Flattening of some powder mixture particles was also noticeable due to the dominant mechanism of fracturing over welding. However, clustering of powder mixture particles

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was still evident at this point as depicted in Fig. 3 (b). Furthermore, increasing milling time up to 9 hr increased the powder mixture particle size from an average of 32 µm to 38 µm as the welding mechanism and agglomeration of powder mixture particles were higher than fracturing. This implies that at this level, longer milling time accelerated the processing temperature that led to the occurrence of welding phenomenon and even the impact energy was inadequate to break down the powder mixture particles size [16]-[19]. As milling time advanced to 12 hr, combination of spherical and flattening of powder mixture particles with smaller and uniform in size of around 22 µm were established, suggesting work hardening had activated the fracturing mechanism. In addition, clustering of powder mixture particles was also greatly improved at this stage. In the current study, a broad particle size distribution of powder mixture was also observed regardless of different milling time due to the reduction in size of larger particles which occurred simultaneously with the growth of smaller particles through agglomeration mechanism [14]-[19]. The main difference however relied on the reduction of the average of powder mixture particle size with increasing milling time from 3 hr to 12 hr. Nevertheless, it is important to note that continue milling time was not advisable as this commonly induced contamination along with the formation of undesirable phases [12]-[14].

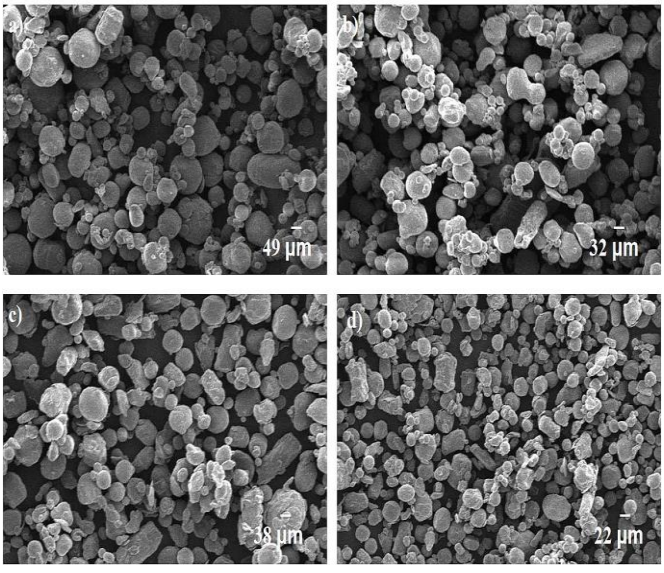

Fig. 3.The effects of milling time on the morphology of elemental powder mixture after milling for a) 3 hr, b) 6 hr), c) 9 hr and d) 12 hr

B. X-Ray diffraction (XRD) Analysis of Elemental Powder Mixture

XRD patterns of elemental powder at different milling time are illustrated in Fig. 4 (a-c). The main elements of the XRD peaks were identified to be Al and Sn powders, characterized by the (111), (200), (220) and (311). Moreover, no additional peaks was recorded, suggesting absence of additional phases or impurity phases formation during milling. In this context, nonexistent of impurity phases indicates a clean interface as well as unwanted reaction between each of metallic powder particles during milling [12]-[16]. On the other hand, a gradual decreased in peaks intensity with increasing milling time was noticed, confirming particle size reduction of elemental powder mixture as previously evidenced in Fig. 3 (a-c). In addition, such decreased was also related to the decreased of the crystallite size, as well as the increased in the lattice strain [15]-[17]. This can be explained by the fact that as milling progressed, powder mixture particles were plastically deformed due to the impact between balls and powders hence resulted in grain refinement and lattice strain enhancement [16]-[18]. Finally, non-existence of Mg peak intensity in the diffraction patterns might be due to the low content of Mg powder added during processing.

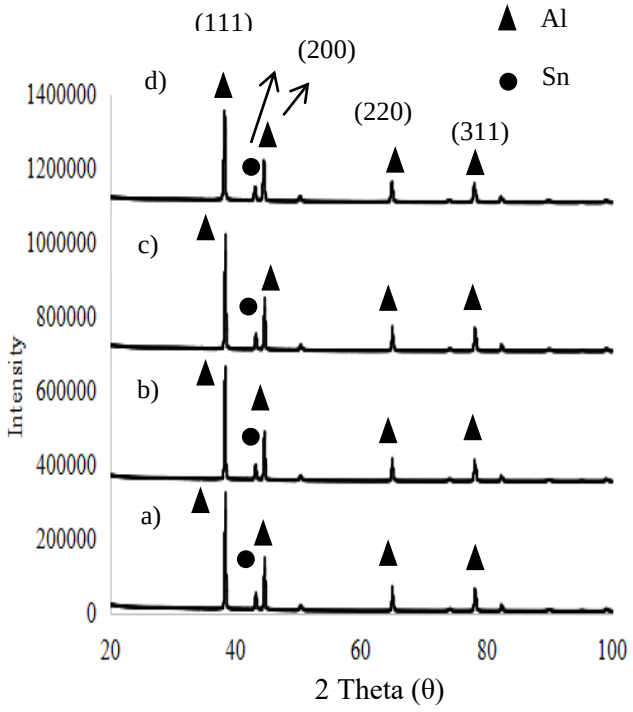

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Fig. 4.XRD peaks of Elemental Powder Mixture at Different Milling Time of a) 3 hr, b), 6hr, c) 9 hr and d) 12 hr

C. Green Densities of Elemental Powder Mixture

Owing to the deformation hardening mechanism during milling with increasing milling time, the ability of the milled powders to be consolidated was also reduced thus decreasing the densities of compacted milled powders [15]-[19]. Such phenomenon was also discovered in the current study where the densities of the compacted milled powders decreased from 1.742 g/cm^3 to 1.736 g/cm^3 with increasing milling time from 3 hr to 9 hr while prolonged milling time up to 12 hr slightly increased the green density to 1.743 g/cm^3. Although it has been reported that broad distribution of powder mixture particle size as accomplished in the current study often resulted in higher density of powder compact, a reduction in green densities of powder compact was identified with increasing milling time up to 9 hr as a result of agglomeration, visible flattening of the milled powders in some areas as well as extreme plastic deformation during processing as confirmed in Fig. 3 (-c) and Fig. 5. In other words, cold welding that occurred during early stage of milling often resulted in large flake-shaped particles as evidenced in Fig. 3 (a) thus degraded the green density of milled powders. However as milling proceeded, particularly at maximum milling time of 12, a slight increased in green density of powder compact was noticed probably due to the formation of smaller powder mixture particles as well as minimization of powder mixture agglomeration in which later assisted in the powder mixture flow capability during compaction [9]. This is actually related to the dominant process of fracturing of larger powder mixture particles during the later stage of milling due to relatively effective movement of particles and a lower tendency to form bridge thus better packing properties [14]-[19].

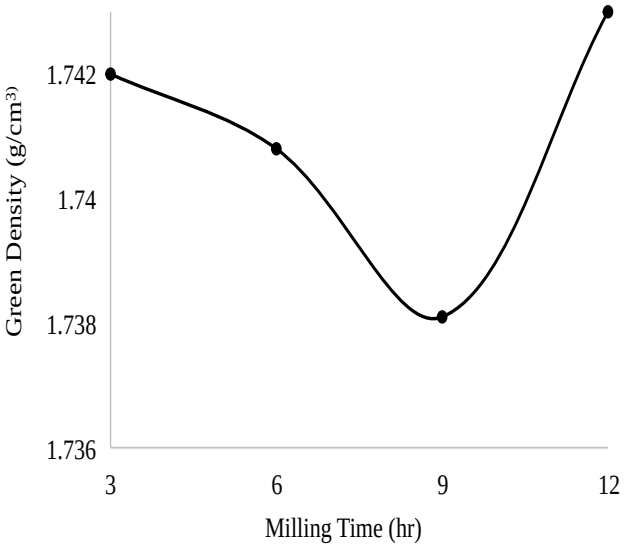

Fig. 5.Green density variation as a function of milling time

D. Morphological Evaluation of Porous Al

Fig. 6 (a-d) displays the images of morphological evaluation of porous Al at different milling time of 3 hr, 6 hr, 9 hr and 12 hr. It can be observed that the macropores with size of about 150 μm acquired via the decomposition of PMMA space holder were closed and sealed throughout all the specimens. Noted that the formation of these closed-cell macropores were consistent as the content of PMMA space holder was fixed at 30 wt. %. These closed-cell macropores were also found to replicate the original morphology of PMMA space holder in terms of size and shape and separated by a dense cell wall of about 45 μm thickness. At an early stage of 3 hr milling, clear micropores formation, cracking and roughness of cell walls were observed as a consequence of predominant process of cold welding that resulted in agglomeration of powder mixture particles hence increased the powder mixture particles size as demonstrated in Fig. 3(a). As the milling time was enhanced from 3 hr to 12 hr, the presence of micropores at the cell walls were greatly decreased as evidenced in Fig. 6(c-d), respectively. This suggests that longer milling time encouraged densifying of the micropores at the cell walls with smoother surface as a result of high surface energy activated by the collision between balls and powders during milling [10]-[12]. Another possible reason could be that, during liquid phase sintering of porous Al, the interdiffusion in the liquid phase was maximized owing to the uniform

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distribution of powder mixture particles along with its reduction in sizes thus contributed to the enhanced solubility between the milled powders and liquid phase throughout sintering [10]-[13].

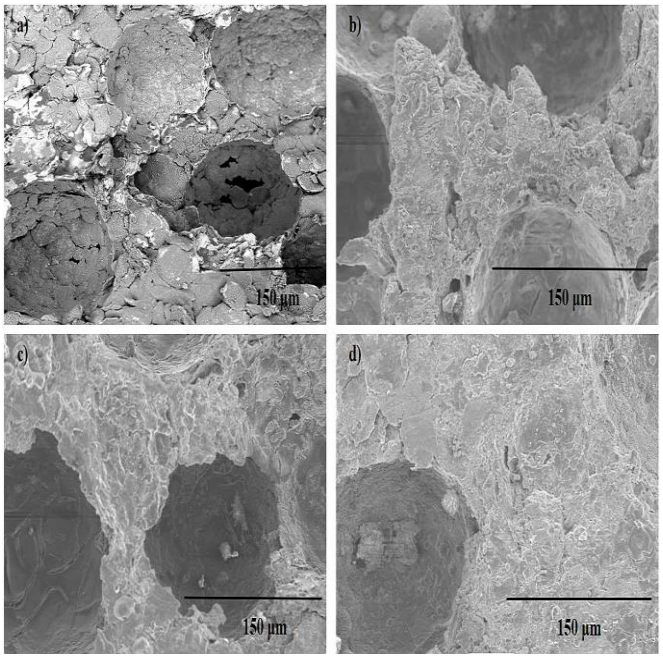

Fig. 6.Morphology of Porous Al at Different Milling Time of a) 3 hr, b), 6hr, c) 9 hr and d) 12 hr

E. Density and Porosity of Porous Al

The effects of different time of 3 hr, 6 hr, 9 hr and 12 hr on the density and porosity of porous Al are shown in Fig. 7 (a-b). It can be seen that the density increased from 1.465 g/cm^3 to 1.469 g/cm^3 while the porosity decreased from 31.75% to 30.67% with increasing milling time from 3 hr to 12 hr, respectively. As milling extended, grain refinement and particles size reduction took place simultaneously due to enhanced surface energy that led to densification of porous Al [10]-[16]. Moreover, micropores minimization at the cell walls with smoother surface associated with prolonged milling was also play a vital role in such increasing and decreasing pattern of densities and porosities of porous Al as confirmed from Fig. 6(a-d). On the other hand, dispersion and agglomeration improvement of powder mixture particles as accomplished in the current study with increasing milling time was also identified to assist densification of porous Al hence reduced the overall porosity. Apart from this, Samal, Parihar and Chaira (2013) mentioned that densification of sintered metal compacts could also be related to the increased in surface area of the milled powders as a result of finer particles, thereby increasing particle to particle contact especially with prolonged milling. It is noteworthy that this could resulted in improved of sintering activity hence decreased the total porosity as realized in the current study [18]-[19].

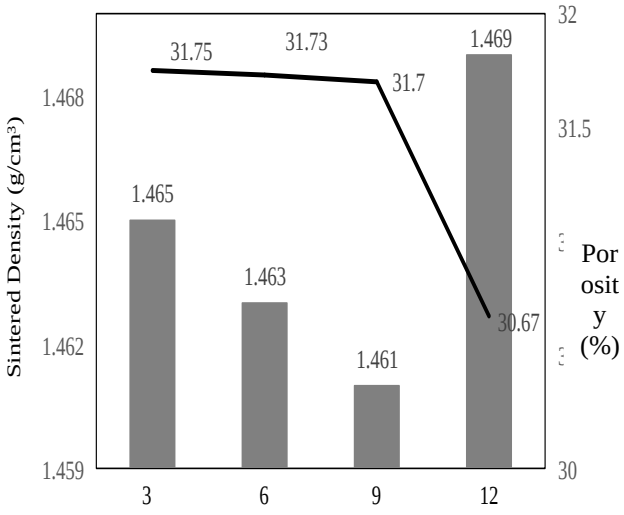

Fig. 7.The effe ing Time on the Density and Porosity of Porous Al

IV. CONCLUSION

Porous Al at fixed content of 30 wt. % PMMA was successfully fabricated by powder metallurgy technique. In this process, the effects of ball milling time on the morphology and density of elemental powder mixture was initially investigated followed by its effects on the microstructure, density and porosity of porous Al prior to design porous body with desirable quality traits. The results obtained for elemental powder mixture investigation showed that increasing milling time from 3 hr to 12 hr decreased the average particle size along with improved dispersion of elemental powder mixture owing to dominant process of fracturing over welding. On the other hand, macropores with closed-cell structure that mimic the original morphology of PMMA space holder in terms of size and shape were obtained after sintering. In this context, smoother cell walls of porous Al with obvious grain refinement and microporosity reduction were observed when milling was enhanced from 3 hr to 12 hr, respectively. These findings were then affected the density and porosity of porous Al in which the

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density increased from 1.465 g/cm^3 to 1.471 g/cm^3 whereas the porosity decreased from 31.75% to 30.45% with prolonged milling duration. Therefore, it can be concluded that extended milling to 12 hr resulted in satisfactory morphology, density and porosity of porous Al.

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