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The Effect of Milling Time on Properties of Magnesium-Based Composite Fabricated via Powder Metallurgy

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ABSTRACT. The aim of this study was to investigate the effect of milling time on the phase formation, microstructure, densification, microhardness and compressive strength of Mg-Zn/HA composite that possess the ability to be used as biodegradable bone fixation device. The mixture of Mg-6 wt.% Zn/8 wt.% HA composite was mechanically alloyed (MA) in a planetary ball mill. Milling time of the composite powders were varied from 2-8 hours. The effect of milling time on the phase formation and morphology of the composite was investigated by X-ray diffraction (XRD) measurement and optical microscope (OM). The density, microhardness and compressive behavior of the sintered composites were also determined by the principle of Archimedes, Vickers microhardness test and compression test, respectively. The results suggested that a homogenous supersaturated solid solution of Mg-Zn/HA was obtained after 2 hours of milling time and the structures undergo structural refinement after being milled for 8 hours. Significant enhancement of hardness and compressive strength were observed in the composite as the milling time was prolonged to 8 hours. The hardness and compressive strength of Mg-Zn/HA being milled for 2 hours were as low as 47.18 HV and 122.49 MPa, respectively, while the composite achieved the maximum hardness and compressive strength as high as 70.47 HV and 209.39 MPa after mposite achieved the best mechanical properties. being milled for 8 hours. The optimum milling time to fabricate the composite was 8 hours, since the co

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1. INTRODUCTION

Magnesium and its alloys have recently attracted much attention as the new generation of biodegradable metallic bone fixation devices, owing to its mechanical compatibility which mimic the mechanical properties of the natural bone and its capability to be degraded and subsequently disappeared after the healing process is completed [1]. In term of mechanical compatibility, the Young's modulus of Mg (about 45 GPa) is much closer to that of natural bones (5-23 GPa for compact bone) which is the most desirable to avoid the stress shielding problem resultant of the mismatch in elastic moduli. In addition, Mg possess good biocompatibility to be used as bone implant materials because it is an essential element to the human body and largely stored in bone tissues [2]. However, a critical problem faced by Mg and it alloys that restricting the commercialization is the rapid degradation rate in physiological environment. In this work, the magnesium-based composite was fabricated through the route of powder metallurgy, which employed the mechanical alloying as the powder processing technique of the powders composite. Mechanical alloying (MA) is a powder metallurgy processing technique for producing composite metal powders which it involves the repeated cold welding, fracturing and re-welding of the powders particles, in a high energy ball mill [3]. Processing the powders composite using mechanical alloying technique gives the advantages of producing powders with controlled fine microstructure due to the repeated numerous impact between the ball and the powders, in addition with reduction of defects that normally generated from casting process such as porosity and inclusions [4]. Mechanical alloying parameters such as milling time, milling speed, and ball-to-powder ratio (BPR) are among the most dominant process variables that significantly alter the properties of resultant composite. The present study is focused on the effect of milling time to the phase analysis, microstructure, physical and mechanical properties of resultant composite.

2. MATERIALS AND METHODS

A mixture of elemental Mg, Zn and HA was weighed accordingly to weight fraction. The Mg-Zn/HA composite was composed of Mg-Zn/8 wt.% HA (weight of Mg:Zn is 94:6). The powders were then subjected to milling process with the speed of 220 rpm and milling time was varied to 2, 4, 6 and 8 hours using high energy planetary mill (Fritsch Pulverisette P-5). Argon gas was purged into the stainless steel jar to prevent oxidation or any possible contamination of the powders. The ball-to-powder ratio of 8.75:1 was kept constant for all milling process using stainless steel balls. The composite powders were then consolidated into 10 mm pellets using stainless steel mold under the compaction pressure of 400 MPa for 2 min. The pellets were sintered at 300 °C (soaking time: 1 hour) under controlled argon flow. The sintered pellets were then subjected into phase analysis using X-ray diffraction (XRD) and morphological study. Density of the sintered pellets was measured according to Archimedes principle, and the result was correlated with the theoretical density and was calculated as Eqs. 1-3.

Microhardness of the sample was evaluated using Vickers microhardness with indentation load of 300 gf and 10 s of dwell time, while compression test was carried out using universal testing machine (UTM) Instron 5982.

$$\text{Theoretical Density} = \rho_{Mg}V_{Mg} + \rho_{Zn}V_{Zn} + \rho_{HA}V_{HA} \quad (1)$$

$$V_{Mg} + V_{Zn} + V_{HA} = 1 \quad (2)$$

$$\text{Relative density (\%TD)} = \frac{\text{Sintered Density}}{\text{Theoretical Density}} \times 100\% \quad (3)$$

3. RESULTS AND DISCUSSION

Fig. 1 shows the XRD patterns of the Mg-Zn/HA composite being milled for various milling times (2, 4, 6 and 8 hours) after being sintered for 1 hour under argon flow. As the milling was prolonged to 6 and 8 hours, the peaks were slightly shifted to the left and broadened, contributed by grain refinement as the milling induced more energy to reduce the size of the particles. 2 hours milling time was observed to sufficiently form the homogeneous Mg-Zn solid solution, by the elimination of any Zn peaks in the pattern. By prolonging the milling up to 8 hours, there was no observable phase changes in the composite, indicating only α-Mg solid solution was detected in the patterns. The peaks indicating the presence of HA were not so obvious in the patterns, since the HA was having low intensity in XRD, due to the amorphous behavior of the ceramic particles.

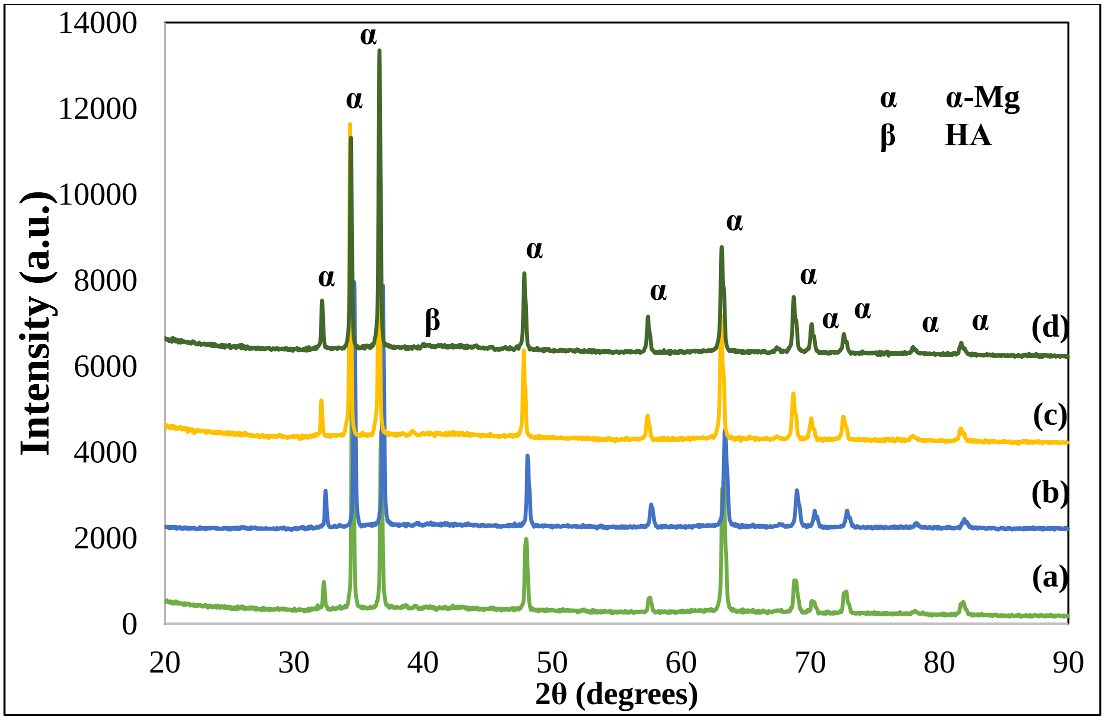

Fig. 1 XRD patterns of Mg-Zn/HA being milled for (a) 2 hours, (b) 4 hours, (c) 6 hours and (d) 8 hours

Micrographs of the Mg-Zn/HA composite that was milled under various milling time from 2 to 8 hours was depicted in Fig. 2. Mg-Zn/HA composite that was milled for various milling time showed significant changes in the grain size formation and lamellae-like formation, as the composite milled for 8 hours experienced the most grain refinement and some numerous pores in the micrograph, as compared to 2 hours milled composite. Formation of the lamellae of Mg-Zn solid solution was more pronounced in the composite that was milled for 8 hours, due to much repeated fracturing and cold welding processes during the milling, which tend to form more homogeneous solid solution between Mg, Zn and HA particles. Extending the milling time up to 8 hours induced more impact between the milled particles, thus assist the refinement of microstructure along with the size of the pores.

The reduction in powder size at longer milling time increased the contact area between grains, leading to enhance the densification, sinter ability and its properties afterward. However, the microstructure of the composite was observed to have lots of small-sized pores after been milled for 8 hours, possibly due to elongated size of the ductile powders after being milled for such long milling time and experienced lots of impact. Elongated size of the composite powders caused the powders to be hard to compact, thus resulted in small and even distribution of pores.

Fig. 3 shows the plot of microhardness and relative density (%TD) of Mg-Zn/HA composite being milled for various milling time. As the milling time prolonged, the microhardness as well as the densification was dramatically increased, except for the composite milled for 8 hours, which less densified than that of 6 hours composite. Details on the values of sintered density, theoretical density and microhardness values were tabulated in Table 1. Decrement in densification of the particular composite is mainly contributed by the presence of small pores inside the microstructure, as portrayed in Fig. 2 (d). At 8 hours milling time, the powders were subjected to excessive impact which caused the powders to be extremely refined in microstructure, but also caused the elongated highly deformed powders to experience low compressibility in compaction. Even though the densification was slightly reduced, the microhardness exhibited higher value than those composites fabricated at shorter milling time (2, 4 and 6 hours). Higher microhardness value achieved by this particular composite was contributed by the strain hardening of powder, which longer milling time was accompanied by higher quantity of impact to the powder, and subsequently assist in higher solubility of Zn in the Mg lattice.

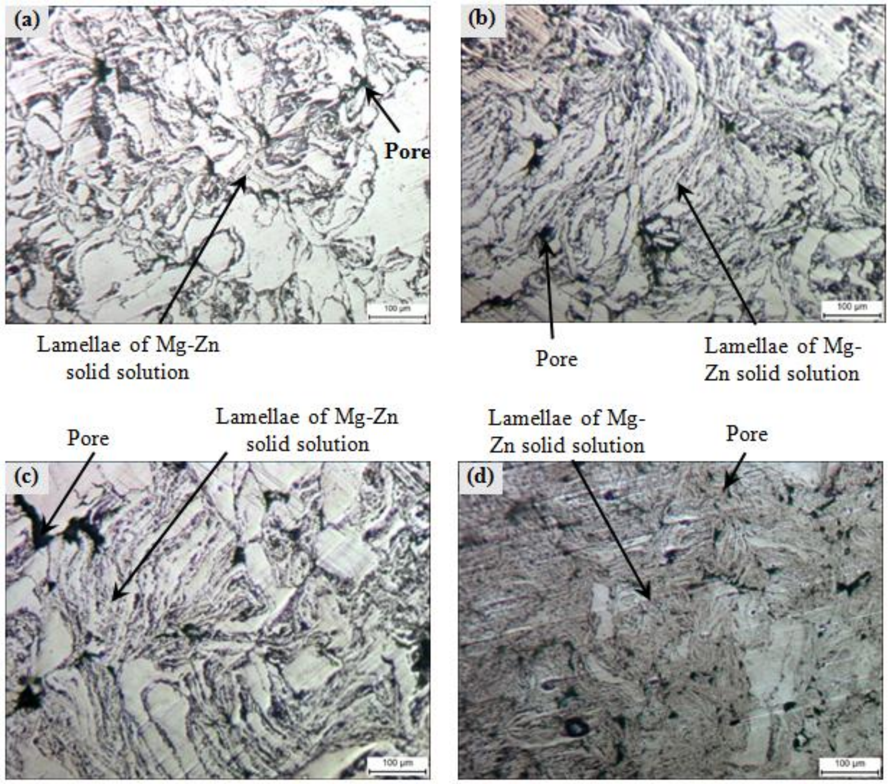

Fig. 2 Micrographs of Mg-Zn/HA composite fabricated by planetary mill for (a) 2 hours, (b) 4 hours, (c) 6 hours and (d) 8 hours

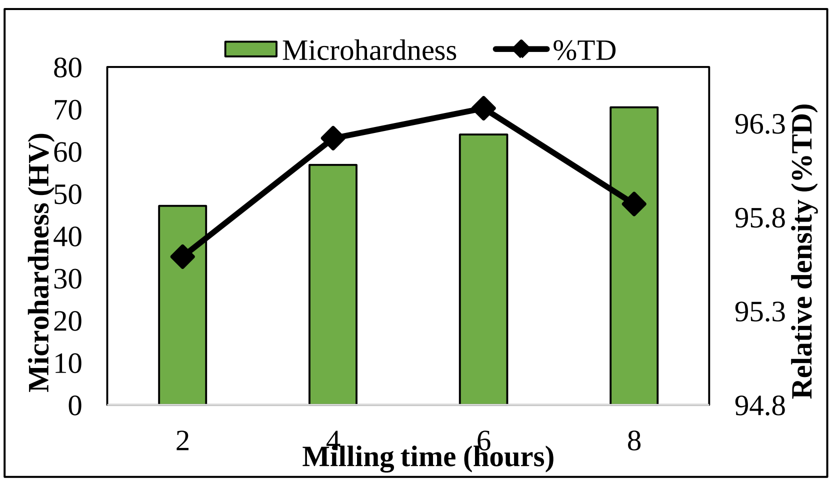

Fig. 3 Plot of microhardness and densification of Mg-Zn/HA composite being milled under various milling time

Table 1 Data tabulation on density and microhardness of the composite

Milling time (hours)	Sintered density (g/cm^3)	Theoretical density (g/cm^3)	Relative density (%TD)	Microhardness (HV)
2	1.7987	1.8863	95.59	47.18
4	1.8106	1.8863	96.22	56.83
6	1.8136	1.8863	96.38	64.07
8	1.8040	1.8863	95.87	70.47

Fig. 4 shows the ultimate compressive strength of Mg-Zn/HA composite that was milled under various milling time. The compressive strength was noticeably increased as the milling time was prolonged to 8 hours. This increment was mainly contributed by the refinement of the particle size. Generally, the strength of composite is highly dependent on the effectiveness of stress transfer between the matrix and the reinforcement [5]. Finer particle size attributed by longer milling time increased the surface contact area between the particles, thus increased the efficiency of stress transfer between the matrix of Mg-Zn alloy and the reinforcement of HA particles.

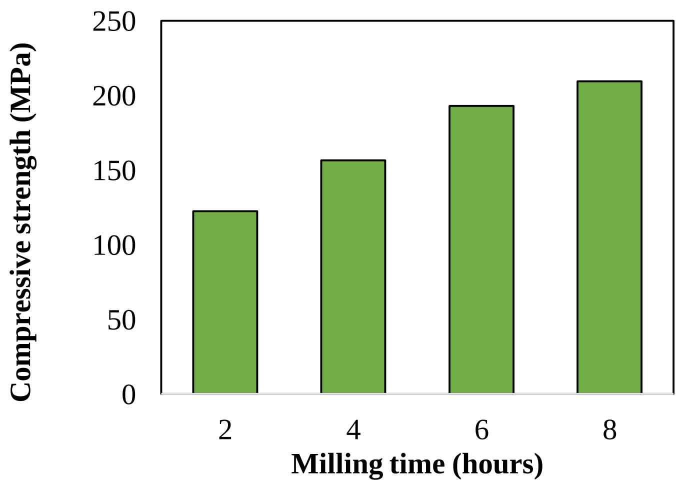

Fig. 4 The ultimate compressive strength of Mg-Zn/HA composite milled under various milling time

4. SUMMARY

In this work, Mg-Zn/HA composite was prepared through powder metallurgy route, with the mechanical alloying (MA) as the powder processing technique. With variation in milling time, XRD diffraction pattern shows only α-Mg solid solution was formed in the composite that was mechanically milled at 2 hours and prolonged milling time. Composite prepared at 8 hours milling time shows the best microhardness (70.47 HV) and compressive strength (209.39 MPa), due to refinement of the powders at the particular milling time, but exhibited a considerable slightly lower value in densification (95.87%). Extending the milling time of the composite might result in further enhancement in the microhardness and compressive strength as the microstructure experienced the extent in grain refinement. However, reaching to some extent, the mechanical properties might stay constant as the milling time is prolonged, indicating the maximum refinement can be achieved for the particular composite.

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