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Microstructure and microhardness of carbonate apatite particle-reinforced Mg composite consolidated by warm compaction for biodegradable implant application

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Abstract
Magnesium-based composites with carbonate apatite reinforcement are attractive biodegradable implant materials. In this study, we observed the effect of carbonate apatite content (5, 10, and 15% wt.) and milling time (3, 5, and 7 h) on the microstructure and microhardness of magnesium-carbonate apatite composites fabricated by powder metallurgy. The consolidation process involved warm compaction without sintering. Characterization was achieved through density testing, x-ray diffraction (XRD), optical microscopy, SEM-energy dispersive x-ray spectroscopy (EDS), and microhardness testing. The powder milling time affects the distribution of apatite carbonate; adding carbonate apatite can increase the hardness of magnesium-based composites. In the XRD spectrum, we identify the dominant magnesium peak but not the magnesium oxide peak. Carbonate apatite powder is distributed at the grain boundaries. The hardness range is 40.26–44.82 Hv or increase by 8.21%–20.23% compared to the hardness of consolidated pure magnesium. The relative density is around 95.92%–98.71%, whereas the relative density of pure magnesium is 99.58%. The obtained optimal conditions for fabricating magnesium composites are the following: content of 10 wt% carbonate apatite (milled for 5 h) with a hardness of 43.58 Hv.

1. Introduction
Efforts to develop biodegradable materials for biomedical applications have been very intense recently, due to their significant benefits for patients as well as doctors. Orthopedic biodegradable implants are especially coveted because they do not require post-implantation surgery [1, 2]. The combination of strength and controlled degradation rate during the healing process and safety for the human body are the most important requirements of implant materials. Almost 70% of implant materials are made of metals [3]. In particular, magnesium (Mg) and Iron (Fe) exhibit many uses in orthopedics and cardiovascular applications [1, 4]. Between them, Mg is considered superior to other metals [1, 2, 6, 7]; it is the lightest metal (ρ 1.74–2.0 g cm$^{-3}$), and it demonstrates the following: a modulus of elasticity similar to cortical and cancellous bones (41–45 GPa); a strength-to-weight ratio greater than those of polymers; and a high limit of toxicity in the body, 700 mg/day [3, 5], compared to 10–20 mg/day of Fe. Mg also functions as an enzyme activator and protein regulator.

Nevertheless, Mg demonstrates several disadvantages, including a high degradation rate of 8 mm year$^{-1}$ and low strength and ductility (yield strength $= 30$ MPa, ultimate tensile strength $= 100$ MPa, $\varepsilon = 7\%$). Moreover, there have been cases where hydrogen gas bubbles appeared under the skin of the patient after a period of time [6]. Mg belongs to the elements group with a mild toxic level, same as Ca, Li, Al, Zr. Mg poisoning
can cause symptoms such as nausea, muscle paralysis, hypotension, cardiac arrest, kidney failure, and disturbed breathing patterns [8]. Therefore, certain improvements are required for Mg to be developed as a biodegradable implant material.

In addition to the new Mg alloy form, as with the rare earth element [9], another potential development method is through the formation of a composite structure [5]. The development of new structures from Mg composite structures is considered more flexible, especially in terms of component design, use of reinforcing materials, and adjustment of biocompatibility [7, 10]. Preparing magnesium composites through powder metallurgy (PM) is considered more appropriate because liquid magnesium is very active, and some reinforcement materials will exhibit low wettability and be reactive when in contact with it. In addition, in the PM process, using a higher volume fraction (up to 50%) of the reinforcing particles and adjusting homogeneity is possible [10].

Calcium phosphate-based particles, such as hydroxyapatite (HA) and β-Tricalcium phosphate, are more desirable for orthopedic applications because of their biocompatibility, bioresorption, and bioactivity [2, 7, 11, 12], which are especially useful in bone graft applications. In the present study, Mg composites are reinforced with carbonate apatite (CA) powder. CA is a type of calcium phosphate bioceramic. Besides being biodegradable, CA exhibits bone-like elements, promotes bone formation (osteocompatibility), and is more easily absorbed than HA [13–15]. The type of biological CA that is most commonly found is type B, in which some phosphate (PO\(_4\)) groups are substituted by carbonate (CO\(_3\)) [7, 16–20]. Previous studies found CA to be absorbed and replaced with new bone tissue without fibrotic tissue [14, 21]. This is different from HA which forms fibrotic tissue.

Consolidation of Mg composites is typically conducted at a relatively low temperature (~400 °C) [18–22] and without conventional sintering because consolidation is considered energy efficient and fast, and the formation of MgO and grain growth are avoided or at least minimized. In addition, the milling conducted to mix the powder demonstrates an effect on the dispersion of the reinforcing particles in the matrix [23].

In the initial stage of this study, the effect of CA addition and milling time are observed in magnesium composites fabricated with PM processes through mechanical milling and warm compaction. The microstructure and mechanical properties of the Mg-CA composite are investigated, including microhardness testing.

2. Materials and methods

Commercial magnesium powder (purity: ≥99.9%, size: 60–300 μm, Merck KGaA, Austria) and local CA powder with a size of ±74 μm were used as initial materials. Mg-xCA mixing was varied based on the CA reinforcement content: x = 5, 10, 15% wt., and for each content, 9 ml Mg-xCA powder were milled using a planetary ball mill PM 400-Retsch at the rotational speed of 200 rpm. The mixing time was 3, 5, and 7 h. For controlling the results, pure magnesium powder (without milling) was used as control. First, the consolidation process was conducted by inserting Mg-CA powder in a cylindrical hollow mold (ϕ: 10.5 mm) and pressing it with a manual hydraulic press at a pressure of 250 MPa in cold conditions. Further, the mold was heated in a muffle furnace up to 350 °C (heating rate of 10 °C/min), held for 10 min. Subsequently, the Mg composite powder was consolidated again by warm compaction at a temperature and pressure of 330 °C and 350 MPa, respectively.

Composite characterization was conducted by density testing, x-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM) coupled with energy dispersive x-ray spectroscopy (EDS), and micro HV hardness testing. Composite density was measured using the Archimedes principle with the DahoMeter tool (DH-300X). XRD analysis was conducted using a Rigaku (MiniFlex 600) diffractometer with CuK\(_α\) radiation and a scan rate of 10 degrees/min. The microstructure of the composite was observed using an Olympus optical microscope (BX41M-LED) and a Hirox digital Microscope (KH8700). SEM-EDS analysis was implemented on an Inspect F50. Microhardness testing was conducted with a compressive force HV1 (9,807 N). Measurements were made in five measurement points on each sample.

Besides to knowing the phases formed in Mg-xCA, the xrd test data is also used to determine the crystal size (D) and the lattice strain (\(ε\)) which refers to the equation referred to in [23].

\[
D = \frac{0.9 \lambda}{B \cos \theta}
\]

\[
ε = \frac{B}{4 \tan \theta}
\]

where: θ = angle (in radians), \(λ = 1.54059^\circ\) A (Cu-Ni radiation) and B = FWHM (full bandwidth at half maximum)
The samples for microstructure and microhardness testing were prepared by cutting the samples with a Minitom-Struers Diamond Cutter with a wheel size of 127 mm × 0.4 mm. Samples were grinded with a SiC paper up to 1200-SiC grit paper and polished with α alumina slurry (0.3 μm) using the Struers Grinding & Polishing Machine. The samples were then etched in a solution of 1.5-g picric acid, 25-ml ethanol, 5-ml acetic acid, and 10-ml distilled water to obtain OM and SEM images.

3. Results

3.1. Density and microstructure of Mg-xCA composite samples

In figure 1, the Mg-xCA composites could be made with the PM method through pressure applied at a temperature of 330 °C (molds temperature). The same process was reported by Gu [17] for making hydroxyapatite (HA)-reinforced Mg composites, but, in the present work, consolidation was not followed by extrusion or any other forming process.

The density test results for the consolidated pure Mg and all the Mg-xCA composite samples with various CA contents and milling times can be seen in table 1.

The XRD results of the Mg-xCA sample for each process are shown in figure 2. Pure Mg XRD results are used as a comparison. Referring to the XRD test results, the effect of changes in milling time and Mg-xCA composition on crystallinity changes (crystal size and lattice strain) have been calculated with the Scherer equation [23] and can be seen in table 2 and figure 3.

Figure 4 shows the shape of the shift in peak Mg in the composition of Mg-xCA which causes a reduction in crystal size and an increase in lattice strain. SEM photos of the shape changes of the Mg-xCA grain (for x = 10 and 15% wt) that were prepared with the milling time (3, 5 and 7 h) and followed by a warm compacting are shown in figure 5.

Figure 6 shows the microstructure of the composite Mg-xCA with a composition of x = 5, 10, and 15% wt. with milling times of 3, 5, and 7 h. The pure Mg microstructure is used for comparison.

The shape and size of the Mg-xCA powder after 3, 5, and 7 h of milling can also be seen in figure 7, which is intended to determine the effect of the milling time on the shape and size of the Mg−xCA powder.

To further observe the structure and composition of Mg−xCA, figure 8 shows SEM images of Mg−xCA (x = 5, 10, 15% wt., each milled for 3 h).
Table 3 shows a summary of the EDS results at one grain-boundary point of the consolidated Mg-xCA, whereas table 4 shows the results at one surface point of the Mg-xCA powder.

Figure 9 shows the SEM-EDS results on Mg-5CA (milled for 3 h), specifically to identify the appearance of small black spots.

3.2. Mechanical properties

The microhardness (HV) of the fabricated composites is shown in table 5 and figure 10.
Figure 11 shows an indentation image of the microhardness testing of Mg-5CA and Mg-15CA (each milled for 7 h) aimed at recognizing differences in the measured hardness.

4. Discussion

4.1. Density and microstructure of Mg-xCA composite samples

Densification decreases when CA is present in magnesium composites (table 1). In this study, the relative density range of all composites is 95%–99%. The lowest densification is found in Mg-15CA composites. The tendency of decreasing densification due to the presence of reinforcement was also reported by del Campo [22] in their Mg-HA composite fabrication study.

The XRD results (figures 2(a)–(d)) show that the dominant peak in all Mg-xCA composite samples is the Mg peak at 2\(\Theta\) = 32.1, 34.3, 36.5, 47.7, 57.3, 63, 68.5, 69.9, 72, 77.7, and 81.5°. There are no other dominant peaks associated with other phases, such as MgO. In figure 2(a), there are no peaks at 2\(\Theta\) = \(\pm\) 43, 62, and 75° that would indicate the presence of MgO. The XRD patterns of the Mg-CA composites correspond to that of Mg-HA observed by Gu [25] with no other peaks, such as peaks for MgO. When compared with the consolidated pure Mg, the pattern of the Mg peak is almost the same as those of the fabricated Mg-xCA composites. Adding CA only changes the intensity of the Mg peak, especially at 2\(\Theta\) = 32.1, 34.3, and 36.5°.

A similar phenomenon is also seen in the consolidated Mg-5CA, Mg-10CA, and Mg-15CA samples (each milled for 3, 5, and 7 h), as shown in figures 2(b)–(d). The milling time does not affect the phases formed in the Mg-xCA composites. No phases are recorded, other than the Mg peak. This means that the CA content (5, 10, and 15% wt.) and the milling time (3, 5, and 7 h) effects on the consolidation of Mg-xCA with warm compaction do not lead to the formation of other phases, such as the MgO phase.

Milling time affects the change in crystallinity. It tends to reduce the crystal size and enlarge the lattice strain. The results of the Mg crystallinity calculation (table 2 and figure 3) show a pattern of crystal size relation to the milling time of Mg-xCA composition. On the Mg-5CA, the milling time of 3 h increases the crystal size. The decrease occurred at 5 and 7 h. On Mg-10CA, a decrease in crystal size occurs after 3 h of milling, then it rises at.

| Milling Time (h) | D (nm) | E(%) |
|------------------|--------|------|
|                  | Mg-5CA | Mg-10CA | Mg-15CA | Mg-5CA | Mg-10CA | Mg-15CA |
| 3                | 41.78  | 27.86  | 33.37   | 0.2631 | 0.3967   | 0.3315   |
| 5                | 24.95  | 40.03  | 27.54   | 0.4437 | 0.2754   | 0.3993   |
| 7                | 25.39  | 29.65  | 42.35   | 0.4343 | 0.3735   | 0.2612   |

* Mg-xCA powder is then compacted by a warm compaction (WC) process. Mg-pure is used as a comparison: \(D = 33.81\) nm and \(\varepsilon = 0.3259\%\).

Figure 3. Relationship graph of milling time during preparation for: (a) crystal size and (b) lattice strain on warmed compacted Mg-xCA.

![Graph](image-url)
5 h and decreases again at 7 h. Whereas for Mg-15 CA, the decrease in crystal size occurs at milling times of 3 and 5 h, but it rises at 7 h compared to pure Mg without milling.

The pattern of lattice strain change is inversely proportional to the pattern of crystal size change. As crystal size decreases, the lattice strain rises, according to equation (2) and 3 [23]. The longer milling time tends to increase lattice strain due to lattice distortion.

The process of milling with a planetary ball mill has 2 important mechanisms namely the cold welding mechanism between different particles and the re-destruction mechanism of cold welding particles as a longer milling time [26]. Referring to the milling mechanism, the first 3 h of the milling process on Mg-5CA begins with the occurrence of cold welding due to collisions between particles with a ball mill, a longer time (5 and 7 h) crushing mechanism will damage the existing cold welding layer, so that it occurs decrease in crystal size. An increase in% wt. CA in Mg composites tends to cause the particle destruction mechanism to occur earlier than the mechanism of inter-particle cold welding. At Mg-10 CA, the decrease in crystal size was initiated at milling time of 3 h, whereas at Mg-15CA the decrease occurred at 3 h and 5 h.

Figure 4. Effect of milling time on peak Mg(2θ = 36.7°) at: (a) Mg-5CA; (b) Mg-10CA and (c) Mg-15CA each with its center aligned peak. As a comparison Mg pure without milling is used.
Reduction in crystal size is indicated when the Mg peak shifts more to the left, the line broadening (FWHM) is wider and the intensity or sharpness of the Mg peak decreases compared to the Mg peak on pure Mg without milling (figure 4). This peak shift pattern has also been reported in Al/Al2O3 synthesis by PM [26].

There is a tendency for an increase in the average size of matrix particles over the length of milling time. It is related to the cold welding mechanism of matrix particle followed by work hardening, as previous studies have reported on the formation of Al/Al2O3 [2]. In this research, the cold welding mechanism of matrix particle (Mg) occurs at 5 h of milling (for Mg-10CA) and 7 h of milling (for Mg-15CA). Increased in grain size and also increased hardness are shown respectively in figures 5 and 10.

Referring to the optical microscopic images (figure 6), the grain boundaries become clearer (dark color) with increasing CA content, when compared to the pure Mg grain boundaries. In the preparation of Mg-xCA powder with the milling process, it appears that CA particles are spread out on the grain boundary. The grain size of Mg-xCA looks relatively similar to the particle size of Mg powder, which ranges from 60 to 300 μm. The effect of increasing the milling time on the microstructure is shown by the presence of a very small number of Mg-xCA grains destroyed by the ball mill blow to the Mg-xCA powder when the milling was longer. No significant differences were seen in the microstructural shape and size after 5 and 7 h of milling. Therefore, a 5-h milling time could be chosen to achieve greater efficiency in the use of energy and equipment.

Figure 7 shows that a longer milling process (3, 5, and 7 h) and a higher CA content (5, 10, and 15% wt.) cause deformation and disintegration of the Mg-CA composite powder. Significant shape changes are observed in the Mg-10CA and Mg-15CA composites powders (milled for 5 and 7 h). This is attributed to strong collisions between the mill ball and the powder and friction between the CA and magnesium particles, making the composite powders deformed and destroyed. This phenomenon was also reported by Alizera in Mg-Nb and Mg-Ta composites powder [10] and by Annur in a Mg-Ca composite powder [27].

Figure 8 shows that the CA particles are distributed at the grain boundaries of Mg (light gray). Table 3 (EDS results) shows that the oxygen content in the consolidated pure Mg grain boundary is quite high and tends to increase further with the CA reinforcement (5, 10, 15% wt.) to Mg-xCA. The presence of oxygen is believed to originate from the oxygen trapped between the Mg grains during the consolidation process and from the oxygen in the carbonate apatite-forming elements. The trapped oxygen causes the appearance of minor porosity at the grain boundaries, which may result in reduced densification of the Mg-CA composites. The addition of CA in the Mg-xCA composite also causes an increase in C, P, and Ca as content forming elements of carbonate apatite.
but, on the contrary, a decrease in the Mg content. The same phenomenon is also observed in the EDS results from the surface area of the Mg-xCA powder before it is consolidated (table 4).

In figure 9, a small black spot is found on the SEM Mg-5CA image (milled for 3 h). Although other phases, such as MgO, are not detected on the XRD spectra, the EDS results show high Mg and O contents, an initial indication for the appearance of the MgO phase. The MgO phase is believed to appear during the warm compaction process because they are not found in the SEM-EDS results of the Mg-5CA powder.

4.2. Mechanical properties
Table 5 and figure 10 show that the average hardness of the Mg-xCA composites varies in the range of 40.26–44.82 HV, whereas the hardness of pure Mg is only 37.28 HV. Alternatively, hardness may be increased by 8.21%–20.23% compared to pure Mg. The Mg-15CA composite (milled for 7 h) shows the greatest hardness of 44.82 HV, whereas the Mg-5CA composite (milled for 7 h) has the lowest hardness of 40.26 HV. Although the Mg-10CA composite (milled for 5 h) shows the high hardness of 43.58 HV, this is slightly below that of the Mg-15CA composite (milled for 7 h).

Figure 10 shows that milling for 5 h provides the Mg-xCA powder with the optimal hardness, especially at 10% wt. CA content. The hardness of Mg-15CA (milled for 3 and 5 h) decreases, presumably due to the lower relative density of Mg-15CA and the presence of trapped oxygen, which can weaken the bond strength between Mg and CA.
The presence of CA increases the average microhardness of Mg-xCA. CA addition to the Mg matrix restrains local dislocation movements during the indentation process, which in turn increases hardness. The same phenomenon has been reported in Mg-HA by Del Campo \[22\] and Gu \[25\], but the hardness value obtained by Del Campo was higher, around 530–610 MPa \[22\] or equivalent to 54–62 Hv \[28, 29\], although the procedure followed in their experiments were different that the ones employed here. In particular, Del Campo’s consolidation process involved a combination of compaction and extrusion. The obtained grain size was smaller, and the reinforcement distribution was readjusted. In addition, Del Campo used uniaxial isostatic pressure \[22\] equipment for the compaction. Based on the present results and those of the previous studies, the factors determining the mechanical properties and the uniformity of the composite’s hardness are as follows:

![Figure 7. SEM images of powders of Mg-5CA, Mg-10CA, and Mg-15CA (each milled for 3, 5, and 7 h) and pure Mg powder (without milling).](image-url)
grain size, porosity (investigated through the compaction method), and the distribution and content of reinforcement [5, 7, 22, 25, 30, 31].

Figure 11 shows the results of microhardness testing conducted on different grain sizes. The grain size of Mg-5CA is greater than that of Mg-15CA. In this case, the measuring portion of Mg-15CA is the part of the

**Figure 8.** SEM images (resolution of 96 dpi, magnification of 500×) of consolidated: (a) Pure Mg (without milling), (b) Mg-5CA, (c) Mg-10CA, (d) Mg-15CA (each milled for 3 h). Light gray at the grain boundaries shows CA particles.

**Table 3.** EDS results at one grain-boundary point of consolidated Mg-5CA, Mg-10CA, and Mg-15CA (each milled for 3 h) and consolidated pure Mg (without milling).

| Element | Wt % |
|---------|------|
| Pure Mg | Mg-5CA, 3 hr | Mg-10CA, 3 hr | Mg-15CA, 3 hr |
| CK      | 02.27 | 04.19 | 05.77 | 05.27 |
| OK      | 17.42 | 22.60 | 30.61 | 31.25 |
| MgK     | 65.59 | 57.75 | 25.47 | 09.96 |
| PK      | 06.36 | 07.32 | 15.52 | 20.29 |
| CaK     | 08.35 | 08.15 | 22.63 | 33.23 |

**Table 4.** EDS results at one surface point of the Mg-5CA, Mg-10CA, and Mg-15CA powders (each milled for 3 h) and pure Mg powder (without milling).

| Element | Wt % |
|---------|------|
| Pure Mg | Mg-5CA, 3 hr | Mg-10CA, 3 hr | Mg-15CA, 3 hr |
| CK      | —     | 03.73 | 03.07 | 04.06 |
| OK      | 04.49 | 30.14 | 22.84 | 25.69 |
| MgK     | 05.31 | 57.34 | 54.48 | 52.32 |
| PK      | —     | 04.04 | 07.97 | 08.47 |
| CaK     | —     | 04.75 | 11.63 | 09.47 |
grain that had been destroyed after milling for 7 h; therefore, the indentation trace on Mg-5CA is greater than the Mg-15CA trace. Therefore, Mg-15CA hardness (after 7 h of grinding) can reach 45.4 Hv due to the increased resistance to dislocation movements. For further development, combining the process of warm compaction with other forming processes such as extrusion, rolling, and even superplastic deformation with equal channel angular pressing (ECAP) \cite{29, 30, 32-34} could be the option to increase the mechanical properties.

Table 5. Average microhardness (HV) of the consolidated Mg-CA composites (Mg-5CA, Mg-10CA, and Mg-15CA) prepared by milling for 3, 5, and 7 h and of the consolidated pure Mg (without milling).

| Composition | Microhardness (HV) | Average hardness (HV) | \(
\frac{HV_{\text{average}} - HV_{\text{pure Mg}}}{HV_{\text{pure Mg}}} \) |
|-------------|---------------------|-----------------------|------------------------------------------------|
|             | Milling time (h)    | 3  | 5  | 7  | 3  | 5  | 7  | 3  | 5  | 7  |
| Mg-5CA      |                    |   | 40.34 | 41.80 | 40.26 | 8.21 | 12.12 | 7.99 |
| Mg-10CA     |                    |   | 42.34 | 43.58 | 42.04 | 13.57 | 16.90 | 12.77 |
| Mg-15CA     |                    |   | 41.52 | 41.22 | 44.82 | 11.37 | 10.57 | 20.23 |

Note: Mean HV for consolidated pure Mg (without milling) = 37.28 Hv.

Figure 9. SEM-EDS images (resolution of 96 dpi, magnification of 500 × ) on small black spots of consolidated Mg-5CA (milled for 3 h).
5. Conclusion

In this research, powder-based magnesium-CA (Mg-xCA) composites were developed using local CA ingredients with a consolidation process through warm compaction. In preparing Mg-xCA powder, the milling time affected the distribution of CA in the composite; however, increasing the milling time too much may destroy the powder. The milling time can be adjusted to obtain equitable distribution and minimize agglomeration. Adding CA reinforcement to Mg composites can increase hardness but decrease relative density. The warm compacting process can minimize the formation of MgO, as seen in the XRD results, so it may be considered for the initial consolidation process. Milling time (up to 7 h) and wt. CA affect the crystal size and lattice strain of Mg on the Mg-xCA composition in a specific pattern. We found that the optimal conditions for fabricating Mg-xCA composites are as follows: 10% wt. CA, 5 h milling time, and hardness achieved at 43.58 Hv. This means that the increase in hardness of Mg-10CA (after milled for 5 h) is 16.9% compared to the hardness of consolidated pure Mg; therefore, this parameter can be a reference for the initial fabrication of Mg-xCA for biodegradable implant applications. The use of smaller powder sizes and combining warm compacting with other forming processes can be an option for the further improvement of the mechanical properties of Mg-xCA.
composites. Besides that, investigating the degradation rate and the biocompatibility of Mg-xCA should be part of the focus of further research to complement its characteristics as a biodegradable implant material.

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