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The Influence of Sintering Temperature of Powder Metallurgy Mg-3wt.% Zn/6wt.% β-TCP: The Hardness and Corrosion Behavior

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Abstract. Powder Metallurgy (PM) is an advanced manufacturing technique that enables the fabrication of materials with unique microstructures and properties. Magnesium (Mg) alloys, particularly magnesium–zinc (Mg-Zn) alloys, are gaining popularity in biomedical applications due to their lightweight nature, good strength, and compatibility with biological systems. However, their mechanical and corrosion properties still need improvement, which can be achieved through alloying and reinforcement. This study investigates the effect of different sintering temperatures on the hardness and corrosion behavior of Mg-3wt.% Zn/6wt.% β-Tricalcium Phosphate (β-TCP) composites. Mg-3wt.% Zn was mixed with 6wt.% β-TCP powder using a roll mill at 120 rpm for 60 minutes. The mixture was then compacted at a pressure of 200 MPa. Sintering was conducted in an argon-controlled atmosphere at temperatures of 450°C, 500°C, and 550°C. The microstructure of the sintered samples was examined using an optical microscope, and Vickers microhardness testing was used to measure hardness. The corrosion behavior was evaluated using a potentiostat. Results showed that sintering at 450°C produced the highest hardness and good corrosion resistance.

Introduction

The magnesium (Mg)-based alloy has garnered significant attention as a bone implant compared to other traditional metal implants, like Co-based alloys, stainless steel, and titanium. Mg-based alloys have good biocompatibility, biodegradability, and low-stress shielding effect, and are also the lightest structural alloys [1-3]. However, the clinical application of Mg-based implants faces significant challenges, primarily related to their rapid corrosion rate in physiological environments [4]. This accelerated degradation can lead to premature loss of mechanical integrity and the production of hydrogen gas, which may cause tissue damage and impair healing processes.

Zinc (Zn) has emerged as one of the most promising alloying elements for biomedical magnesium applications due to its beneficial effects on both mechanical properties and biocompatibility [5]. The presence of 3 wt.%Zn in the Mg matrix leads to the formation of intermetallic phases, which contribute to the strengthening of the alloy.

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β-tricalcium phosphate (β-TCP) is a bioactive ceramic that is used for bone repair. It possesses unique characteristics that promote the growth of new bone tissue, distinguishing it from other materials used for bone grafts. The resorbable characteristic of this material closely simulates the solubility of bone materials, allowing it to integrate with new bone development [6]. Incorporating β-TCP particles in the Mg-Zn is well-acknowledged to impart valuable bioactivity properties, as β-TCP possesses excellent biocompatibility and bioactivity because of its close resemblance to the chemical and structural properties of human bone [7]. The mechanism of β-TCP with the formation of an apatite layer can improve the bioactivity performance of Mg-Zn alloy after immersion in a simulated body fluid solution.

Sintering temperature is one of the most important processing parameters in powder metallurgy, heavily affecting the microstructure, mechanical properties, and corrosion behavior of magnesium-based composites. Generally, higher sintering temperatures improve densification and particle bonding, resulting in better mechanical properties [8]. However, excessive temperatures can cause negative effects such as element segregation, especially the loss of volatile elements like zinc, coarsening of reinforcement particles, and the formation of brittle intermetallic networks [7,8]. The porosity, distribution of second phases, and interfacial characteristics developed during sintering directly influence corrosion rates and the uniformity of degradation in environments similar to physiological conditions. Therefore, optimizing the sintering temperature is essential to balance mechanical performance and corrosion resistance.

Methodology

Mg-3wt.% Zn/6wt.% β-TCP composites were prepared by powder metallurgy technique. Raw materials of Mg powder (from Sigma Aldrich) and 3wt.% of Zn powder (from Hmbg) were mixed with 6wt.% of β-TCP powders (from Sigma Aldrich). The raw materials were mixed for 60 minutes using a roll mill machine at 120 rpm. Then the mixed powders were uniaxial compacted using a hand press machine with a pressure of 150 MPa. The green compact samples were then sintered in a tube furnace at sintering temperatures of 450°C, 500°C, and 550°C, respectively, for 2 hours inside an argon atmosphere. The sintered samples were polished according to the standard preparation of the metallographic samples. For microstructural analysis, all samples were observed under an optical microscope, and the hardness values were measured using the Vickers hardness test.

NOVA software and AUTOLAB PGSTAT 204 were both implemented for the linear polarization test. A three-electrode cell system was used in the electrochemical measurements, consisting of the sintered sample as the working electrode (WE), a platinum rod as the counter electrode (CE), and a saturated calomel electrode (SCE) as the reference electrode as shown in Fig.1. To ensure a consistent exposed area, the specimen's surface was covered with electroplating tape, leaving only a 1 cm^2 region exposed. Phosphate buffer saline (PBS) solution was used as the electrolyte. The polarization scan was performed with a scan potential range from -250 mV to +250 mV at a scanning rate of 1 mVs^{-1}.The corrosion rate of Mg-3wt.% Zn/6wt.% β-TCP composites were measured using equation (1).

$$Corrosion\ Rate\ (mm/year) = 0.1288 \times I_{corr} \times \frac{EW}{\rho} \quad (1)$$

Where, EW is equivalent to the corroding species and ρ is the density in g/cm^3. The I_{corr} is the corrosion current density in unit of $mAcm^{-2}$.

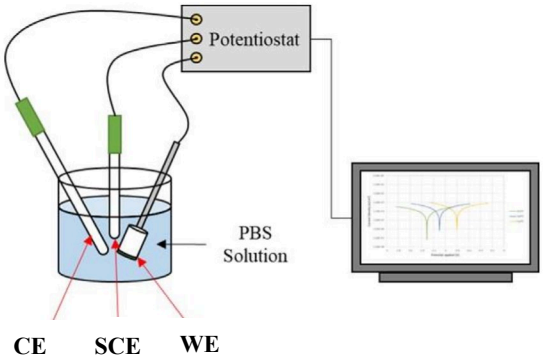


Fig. 1: The three-electrode cell setup for polarization test.
***CE**: Counter Electrode, **SCE**: Saturated Calomel Electrode, **WE**: Working Electrode*

Result and Discussion

Fig. 2 shows the hardness value after sintering at three sintering temperatures 450°C, 500 °C, and 550°C, respectively. It can be seen that the trend of hardness value decreases after sintering at 500°C and then slightly increases at a sintering temperature of 550°C. From Fig. 2, sample sintered at 450°C represents the highest value of hardness, which is 36.33 HV. Meanwhile, the lowest hardness value is 24.43 HV at a sintering temperature of 500°C, and the value of hardness slightly increases to 30.05 HV at a sintering temperature of 550°C. The highest value of hardness is due to strong interparticle bonding. The β-TCP disperses through the Mg-3wt.% Zn matrix and enhances their hardness value [10]. However, at 500°C of sintering temperature, the grain coarsening of the sample gives the lowest values of hardness.

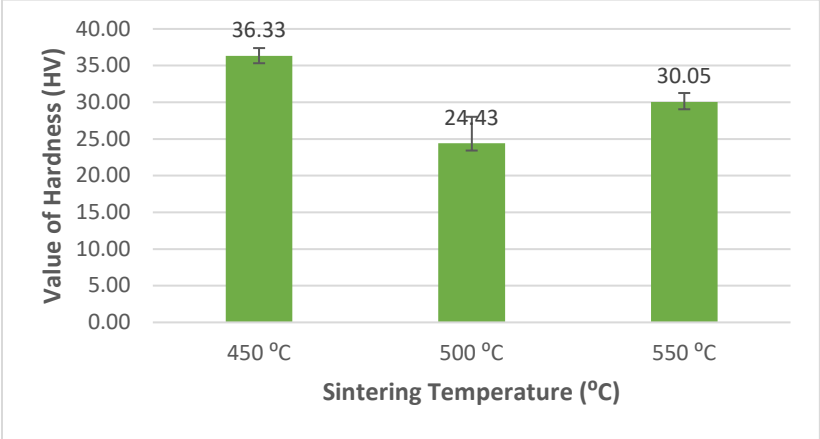


Fig.2: The value of Hardness with various sintering temperatures

The optical microstructures of Mg-3wt.% Zn/6wt.% β-TCP composites after sintering at 450°C, 500°C, and 550°C are shown in Fig. 3. The microstructure of the sample sintered at 450°C [Fig. 3a] exhibits relatively fine, well-defined grains and small pores present in low to moderate amounts. At the sintering temperature of 500°C [Fig. 3b], the grain size increases compared to the sample sintered at 450°C. Meanwhile, at 550°C [Fig. 3c], the microstructure displays coarse grains with pores located at and along the grain boundaries. The formation of pores decreases with increasing sintering temperatures.

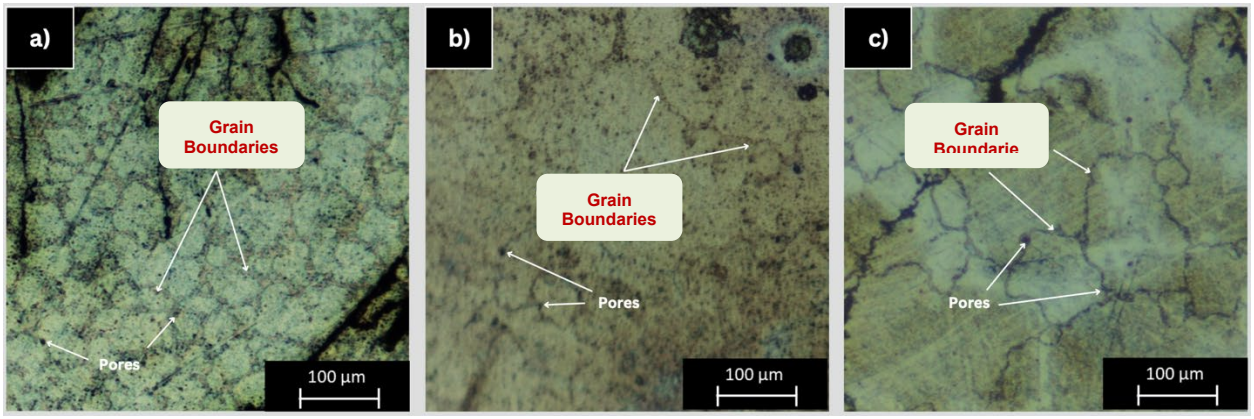


Fig.3 Optical microstructures of sample after sintered at different sintering temperatures (a) 450°C, (b) 500°C, (c) 550°C

The results of the linear polarization test performed on specimens immersed in phosphate-buffered saline (PBS) solution are presented in Table 1. The corrosion potential (E_{corr}) and corrosion current (I_{corr}) were determined by extrapolating the polarization curve depicted in Fig. 4, while the corrosion rate was extracted from the NOVA software. However, equation (1) could also be used to obtain the corrosion rate of the Mg-3wt.% Zn/6wt.% β-TCP. E_{corr} serves as an indicator of the likelihood of corrosion during the test, while I_{corr} quantifies the severity of corrosion experienced by the specimens [11].

The E_{corr} value of the sample sintered at 450°C was -1.3104 V. Meanwhile, samples sintered at 500°C and 550°C show a shift to more positive E_{corr} values, measuring -1.0942 V and -0.8034 V, respectively. The change in corrosion potential (E_{corr}) toward more positive values gives an increasing corrosion rate. Thus, the sample sintered at 450°C exhibited the most favorable corrosion performance, with the lowest corrosion rate. These findings indicated that the highest corrosion rate occurred at higher sintering temperatures, which was attributed to grain coarsening and higher porosity. Yan et al. [12] highlighted that coarse grains play a significant role in causing non-uniform corrosion and increasing localized corrosion, which results in accelerated material degradation.

Table 1: Result of linear polarization of 450°C, 500°C, and 550°C samples

Sintering Temperature (°C)	E_{corr} (V)	I_{corr} (A/cm^2)	Corrosion Rate (mm/year)
450	-1.3104	6.29×10^{-5}	0.7306
500	-1.0942	1.32×10^{-4}	1.5353
550	-0.8034	1.51×10^{-4}	1.7519

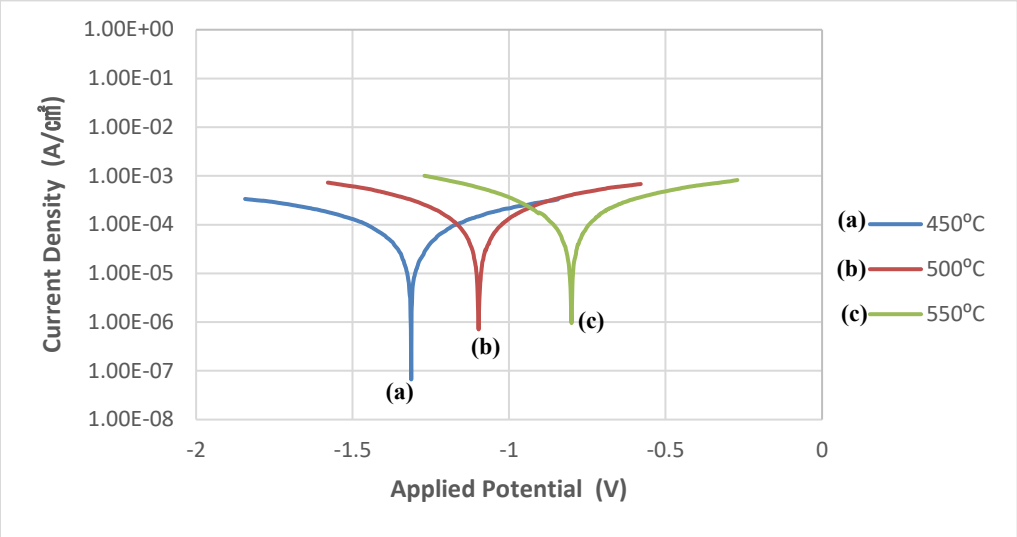


Fig.4: Tafel plot of different samples, (a) 450°C, (b) 500°C, and (c) 550°C

Fig. 5 shows the optical microstructure after corrosion testing for samples sintered at three different temperatures. It can be seen that sample sintered at 450°C (Fig.5 (a)) demonstrates minimal pitting and localized corrosion, as fine grains provide a more uniform protective layer. Meanwhile, samples sintered at 500°C have more pitting, and localized corrosion occurs due to larger grain boundaries acting as corrosion initiation sites (Fig.5 (b)). More pitting and localized corrosion occur as coarse grains create weak spots in the protective layer (Fig.5 (b)).

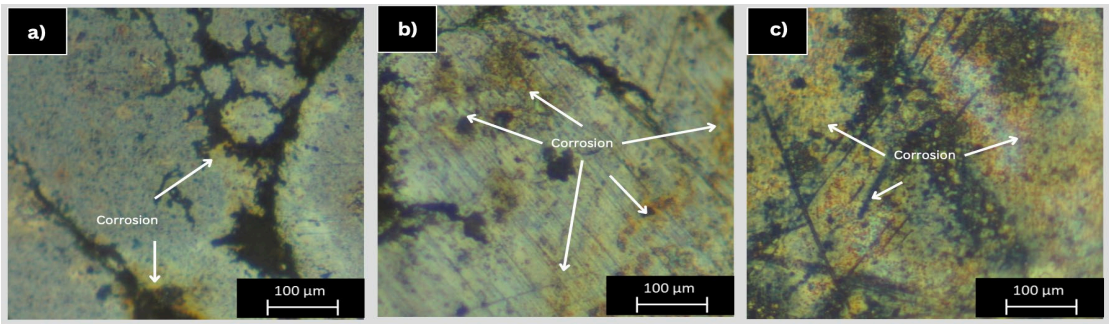


Fig.5: Optical microstructures after corrosion testing for samples sintered at different sintering temperatures, (a) 450°C, (b) 500°C, (c) 550°C

Conclusion

In conclusion, optimizing sintering temperature is essential to improve the hardness and corrosion resistance of Mg-3wt.% Zn/6wt.% β-TCP composites, making them more viable for biomedical applications. The sample sintered at 450 °C exhibited the best overall performance, with the lowest corrosion rate (0.7306 mm/year) and the highest hardness (36.33 HV). Increasing the sintering temperature to 500°C and 550°C led to higher corrosion rates and reduced hardness.

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