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Investigated of Zn-1Mg Alloy Using Potentiodynamic Polarization and Weight Loss Methods in Kokubo's Solution

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Abstract

Zinc alloys are considered as candidates for bioabsorbable implant materials due to their corrosion rates and their outstanding combination of biodegradability and biofunctionality. This study compares the corrosion behavior of Zn-1Mg alloys prepared in as-cast, as-rolled, and as-extruded forms using two main methods: immersion testing (weight loss) in Kokubo's Simulated Body Fluid at 37 °C for up to 14 days, and potentiodynamic polarization (PDP). Microstructure characterization was performed using SEM/EDS to identify corrosion products after the corrosion test. The weight loss method showed the highest corrosion rate in the as-rolled sample 0.56 mm/year and the lowest in the as-cast sample 0.35 mm/year. In contrast, the potentiodynamic method showed the lowest corrosion rate in the as-extruded sample 0.019 mm/year. These results indicate that the extrusion process produces a refined and uniform grain microstructure, which contributes to improved electrochemical corrosion resistance. Overall, the corrosion rates of all samples were still within the safe daily intake limits of Zn allowed in the human body. It were concluded that mechanically processed Zn-1Mg alloys (rolling for plates and extrusion for pins) are suitable and promising for use as bioabsorbable metals for non-load-bearing applications such as craniofacial implant applications.

Keywords: Zn-1Mg, Corrosion Rate, Weight Loss, Potentiodynamic, Microstructure, Bioabsorbable.

1. Introduction

Bone fractures are common injuries that require internal fixation using implants such as plates and screws[1]. Conventional permanent implants made of corrosion-resistant metals, including titanium and stainless steel, typically necessitate secondary removal surgery after healing, increasing both clinical risk and healthcare costs[2]. To address this limitation, bioabsorbable metals have emerged as promising alternatives that can gradually degrade in vivo, eliminating the need for implant removal[3]. Among candidate materials, iron (Fe), magnesium (Mg), and zinc (Zn) have been extensively investigated[3]–[6]. However, Fe exhibits excessively slow degradation, whereas Mg degrades too rapidly, potentially compromising mechanical integrity before complete healing[7]. In contrast, Zn possesses an intermediate standard corrosion potential (−0.8 V), offering a more suitable degradation rate, along with good biocompatibility due to its essential role in physiological processes[8], [9]. Despite these advantages, pure Zn suffers from inherently low mechanical strength (<20 MPa), limiting its direct application as a load-bearing implant material[5], [8], [10]. Alloying with elements such as Mg and subsequent thermomechanical processing (e.g., rolling and extrusion) have been shown to significantly enhance its mechanical properties, particularly in Zn-1Mg alloys[9], [11]. However, these processing

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routes not only modify the microstructure but also strongly influence corrosion behavior, which remains insufficiently understood, especially when evaluated using different corrosion assessment techniques[8].

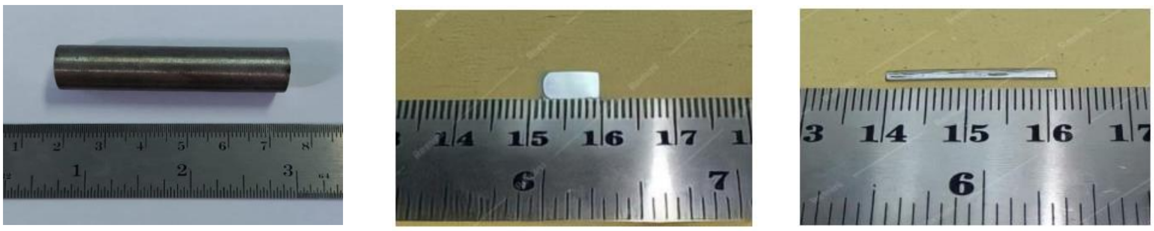
Corrosion characterization methods play a critical role in interpreting degradation behavior. Potentiodynamic polarization (PDP) provides instantaneous electrochemical kinetics, while weight loss measurements reflect long-term degradation, incorporating effects such as corrosion product evolution, passive film formation, and localized corrosion. Although both methods are widely used, their results are often not directly comparable, particularly for biodegradable metals where corrosion mechanisms are complex and time-dependent. Currently, there is a lack of systematic studies that directly compare PDP and weight loss methods for Zn-1Mg alloys under different processing conditions, leading to potential inconsistencies in evaluating corrosion performance.

Therefore, this study aims to systematically compare the corrosion behavior of Zn-1Mg alloys in as-cast, as-rolled, and as-extruded conditions using both potentiodynamic polarization and weight loss methods. This comparison is intended to clarify the relationship between short-term electrochemical responses and long-term degradation behavior, thereby providing a more reliable framework for assessing corrosion performance. The findings are expected to contribute to the optimization of Zn-based bioabsorbable implants, particularly for orthopedic applications, by enabling more accurate prediction of in vivo degradation and improving material selection for clinical implementation.

2. Material and Method

2.1 Material and Sample Preparation

Zn-1Mg alloy was prepared through induction melting vacuum casting process, then mechanically rolled and extruded as shows in Figure 1. As-cast samples were made by pouring the molten alloy into a cast iron mold to be cast into a cylindrical bar with a diameter of 8 mm and 55 mm of length. As-rolled samples were made by rolling and then cutting to produce samples with dimensions of 8 mm in length, 6 mm in width, and 2 mm in thickness. As-extruded samples were made by extrusion and cutting to dimensions of 20 mm in length and 2.5 mm in diameter. The sample surface was sanded in stages to grit #2000, polished, ultrasonically cleaned, and then dried.



(a) (b) (c)

Figure 1. Sample Zn-1Mg: a) As-Cast b) As-Rolled c) As-Extruded

2.2 Potentiodynamic Polarization (PDP)Test

Electrochemical tests were performed using a Metrohm Autolab potentiostat with a three-electrode configuration: the sample as the working electrode, platinum as the counter electrode, and AgCl as the reference electrode. The test medium was Kokubo's Simulated Body Fluid at 37°C. Before measurement, the sample was stabilized for 60 minutes until a stable open circuit potential (OCP) was obtained. Polarization curves were taken with a scan rate of 1 mV/s from -250 mV to +250 mV vs. OCP according to ASTM G5-94[12]. The corrosion current density (Icorr) value was obtained from Tafel extrapolation, and the corrosion rate was calculated based on that value.

2.3 Immersion (Weight-Loss) Test

The samples were immersed in Kokubo's Simulated Body Fluid (SBF)[13] at 37°C for 14 days. The ratio of the solution volume to the sample surface area was maintained at 1 ml/mm^2 according to ASTM G31-72[14]. After the immersion period, the samples were washed with deionized water, and the corrosion products were cleaned using a chemical solution according to ASTM G1-03[15]. The corrosion rate (CR) was calculated using the equation:

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$$CR = ((87{,}6 \; x \; \Delta W))/(A \; x \; \rho \; x \; t) \quad (1)$$

Where: ΔW = mass loss (mg), A = surface area (cm^2), t = time (hours), and ρ = material density (g/cm^3).

2.4 Microstructure and Chemical Composition (SEM and EDS)

Scanning Electron Microscope (SEM) was used to analyze and compare the microstructures of the cast Zn-1Mg alloy (as a control variable) and the rolled and extruded Zn-1Mg alloy (as dependent variables) and chemical composition was analyzed using Energy Dispersive X-ray Spectroscopy (EDS).

3. Result and Discussion

3.1 Potentiodynamic Polarization (PDP)

The potentiodynamic polarization method was employed to measure the current-potential response of a metal alloy in an electrolyte, characterizing its electrochemical and corrosion properties. Analysis uses a three-electrode configuration with a platinum auxiliary electrode, an AgCl reference electrode, and a working electrode on the sample. This potentiodynamic uses a scan rate of 1 mV/s in accordance with ASTM ID: G5-94 for the exploration table

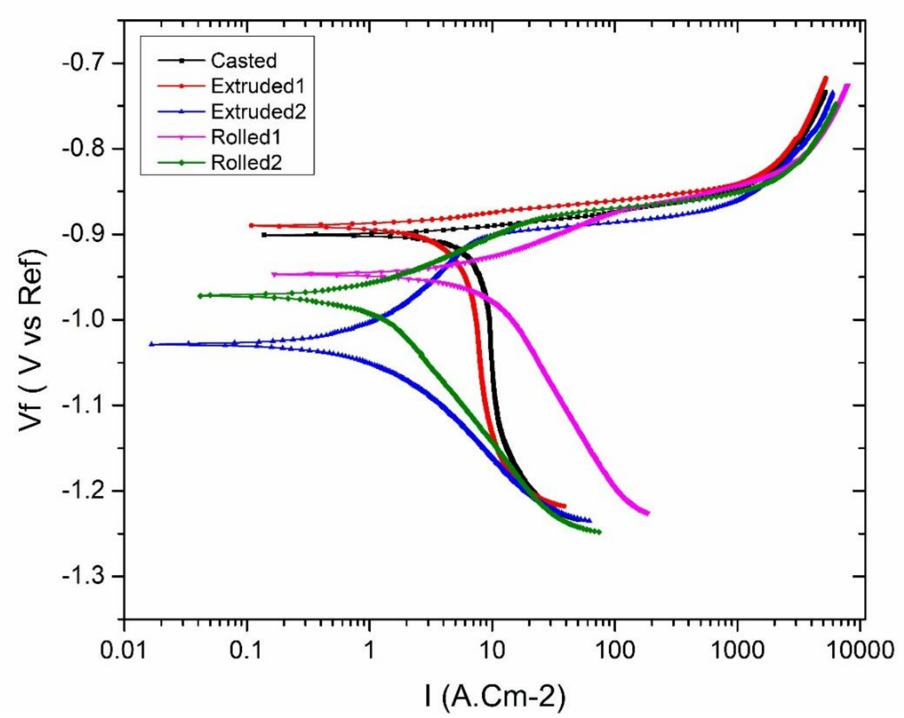

Figure 2. Curve Tafel Potentiodynamic Zn-1Mg alloy

Table 1. Analysis data Corrosion Rate

Sample (Zn-1Mg)	C.R(mm/year)	Eoc(V)	Ecorr(V)	Icorr(µA)
As-Cast	1.449	-0.901	-0.896	4.398
Rolled 01	0.0986	-0.947	-0.946	2.746
Rolled 02	0.0555	-0.972	-0.971	1.862
Extrusion 01	0.0801	-0.089	-0.088	2.242
extrusion 02	0.0189	-1.029	-1.033	0.531

The potentiodynamic polarization curve of the Zn alloy is shown in Figure 2. Tafel extrapolation analysis of the curve using the open circuit potential (Eoc), corrosion current density (Icorr), corrosion potential (Ecorr), and corrosion rate analysis for the Zn-1Mg alloy with variations in casting, rolling, and extrusion is shown in Table 1. As seen in the data table, the as-cast sample has the fastest corrosion

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rate with a value of 1.4 mm/year, which then decreases significantly in the as-rolled sample with a value of 0.08 mm/year, and the lowest corrosion rate results are found in the as-extrusion sample with a value of 0.01 mm/year. This proves that the rolling and extrusion processes can reduce the speed of the corrosion rate so that the implant installed in the patient will last longer with stability that helps the healing process more completely. The corrosion rate results on this as-cast sample are 8 times greater than the findings of Katarivas Levy et al. (2017) who obtained a corrosion rate value using the potentiodynamic method of 0.012 ± 0.002 mm/year[4]. According to Table 1, the lowest corrosion rate and close to the results obtained in extrusion data 2 with a value of 0.0189 ± 0.002 mm/year. The open circuit potential (Eoc), corrosion current density (Icorr), and corrosion potential (Ecorr) values are almost the same as the values reported by Katarivas Levy et al. (2017)[4]. This intersects with the equation found by Liu et al.[12].

Anodic Reaction: $$Zn \rightarrow Zn^2 + 2e^- \quad (2)$$

Cathodic Reaction: $$O_2 + H_2O + 4e^- \rightarrow 4OH^- \quad (3)$$

Formation Zn(OH): $$2Zn^2 + 4OH \rightarrow 2Zn(OH)^2 \quad (4)$$

Formation ZnO: $$Zn(OH)^2 \rightarrow ZnO + H_2O \quad (5)$$

Anodic reaction occurs in Zn metal in Kokubo Liquid, while the formation of Zn(OH) and ZnO is the result of the corrosion reaction of Zn metal when releasing hydrogen ions[12]. This is evidenced in Figure 3 which is the result of corrosion observed with an optical microscope on metal. It can be seen the Pitting Corrosion occurs on the surface with immersion on the 14th days of the rolled sample.

3.2 Immersion (Weight-Loss) Test

Corrosion rate data were conducted using the weight loss method from immersion tests can be obtained by immersing alloy samples in a suitable simulated solution for a period of 14 days. After immersion, the samples are removed, washed, and analyzed to measure the weight different, morphological characterization, or other changes in surface that indicate the degree of alloy degradation. Figure 3 shows the corrosion rate of the rolled alloys sample was a faster degradation rate than the as-cast and extruded samples. Based on Mostaed's research, the corrosion that occurs is pitting corrosion, which occurs in spherical atomic structures[9]. The results of the immersion test indicate that the extruded sample degrade more slowly than the rolled sample, but slightly faster than as-cast sample in the simulated solution. Weight loss in rolled alloys is more significant than that of extruded alloys, as the surface of rolled alloys tends to have a grain structure that is large and widely spaced. This is in contrast to the extruded alloys, which have a small and dense grain structure. The corrosion rate of as-extruded Zn-1Mg decreased quite slowly with a value of 0.35 ± 0.005 mm/year, compared to as-rolled which experienced a corrosion rate of 0.56 ± 0.005 mm/year, one and half times higher.

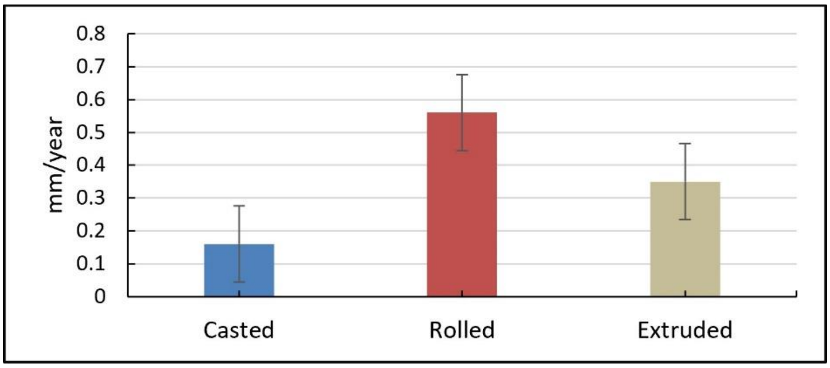

Figure 3. Corrosion Rate of Zn-1Mg Alloy (Weight Loss Method)

Figure 4 shown the pitting corrosion was observed after immersion for 14 days (336 hours),. This is consistent with the findings of Mostaed, who also observed pitting corrosion[9]. The Zn-1Mg alloy can be used as a bioabsorbable implant material because the chemical interaction between the alloy and the simulated solution leaves almost no residue during immersion and is within the permissible limits

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for Zn (RDI = 8-20 mg/day) and Mg (RDI = 240-420 mg/day)[13]. Considering the permissible RDI limits for Zn in the human body, this alloy has the potential to be used as a bioabsorbable metal because its corrosion is not too rapid and is still within the permissible limits for human use. Then, after immersion, the sample was washed and dried to re-examine the microstructure.

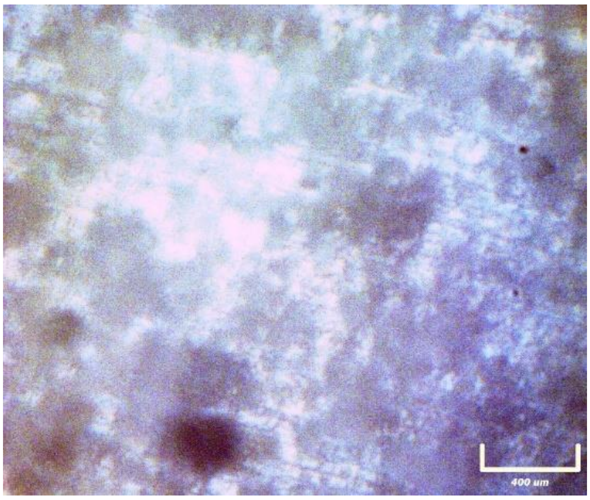

Figure 4. Pitting Corrosion in Zn-1Mg Alloy As-Rolled, Immersed in 14 Days

3.3 Microstructure and Chemical Composition

Scanning electron microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) were performed to characterize the microstructure of samples. The as-cast Zn-1Mg alloy was a control (independent) sample to compare with the as-rolled and as-extruded Zn-1Mg samples, which as dependent variables. Figure 5 shows the microscopic observations of the as-cast sample using at 4000x magnification. The EDS map shows that green represents O ions, yellow represents Mg ions, and red represents Zn ions. The black color represents the corrosion products indicated in the EDS results, which are green or produce O ions. The corrosion products have a large grain structure with very large spacing between the corrosion products. If the corrosion products were to represent the atomic grain structure that occurs during the casting process, they would be large grain structures with very large spacing between atoms.

Meanwhile, in Figure 6, the microstructural observations using the rolling process at the same magnification show slightly smaller corrosion (black color) results than the casting process, with closer spacing between the colors but producing a more rounded shape. The corrosion results as a grain shape, the structure found in the rolling process shows smaller, denser, and more rounded results compared to the casting process. However, this grain structure is slightly larger than the extrusion process. Interestingly, in Figure 7, the microstructural observations using the extrusion process at the same magnification show smaller but more evenly distributed black/corrosion spots. Considering the grain structure, the extrusion process produces smaller, denser, and more uniform shapes.

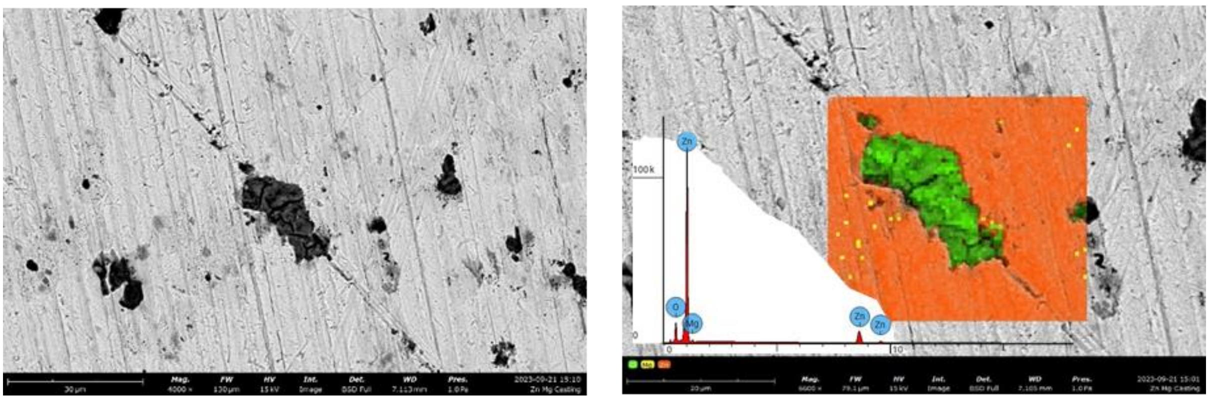

Figure 5. SEM and EDS of As-Cast Sample

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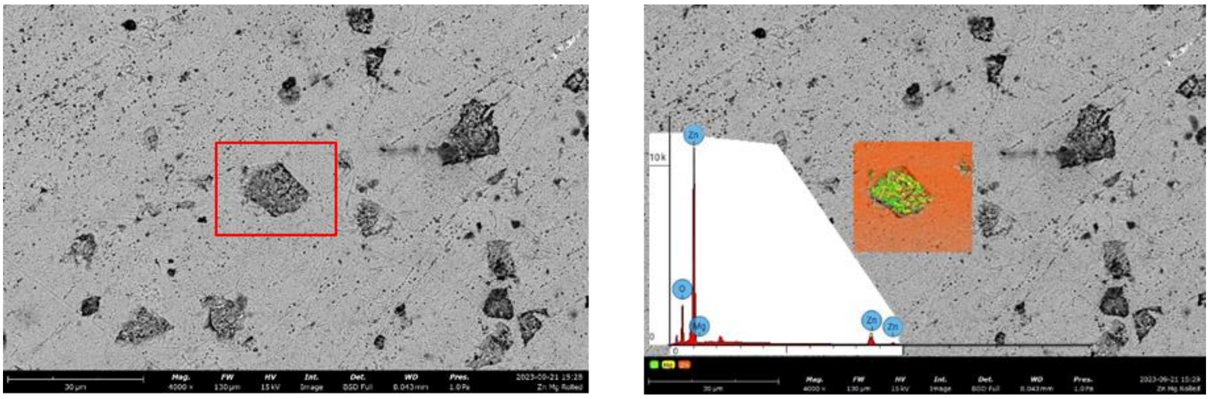

Figure 6. SEM and EDS of As-Rolled Sample

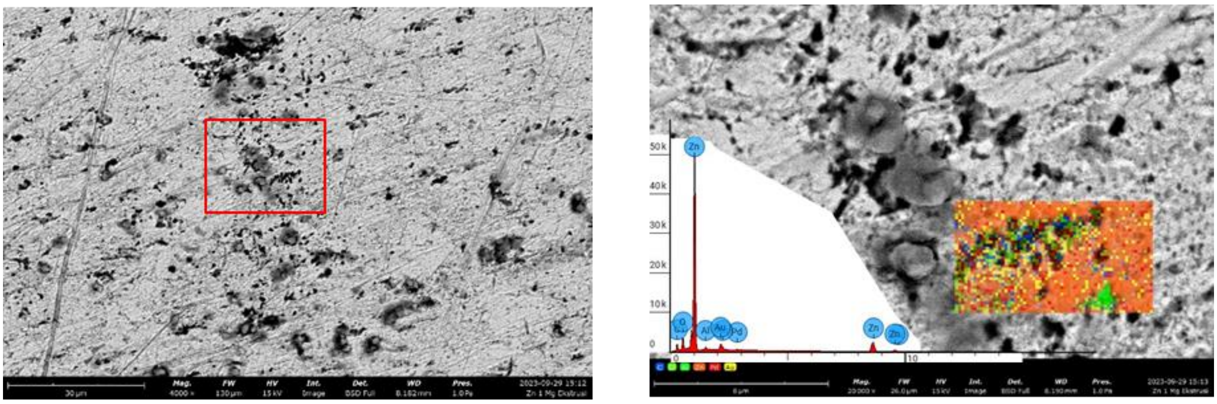

Figure 7. SEM and EDS of As-Extruded Sample

The as-rolled microstructure is somewhat consistent with research[9] that displayed the surface of Zn-1Mg using SEM after immersion for 336 hours. This Zn-1Mg alloy microstructure also aligns with Nurfitri's research, which found that the rolling process results in a material that becomes harder due to the smaller grain size after the process[14]. However, after the extrusion process, the particle grain size becomes smaller but the mechanical strength is slightly lower compared to the rolling process.

3.4 Discussion

The results showed a significant difference between the corrosion rates measured by the weight loss and potentiodynamic polarization methods. This difference can be explained by fundamental differences in the measurement principles of the two methods. The weight loss method measures corrosion cumulatively over a 14-days period, representing actual long-term degradation. The results showed that the as-rolled sample had the highest corrosion rate (0.56 mm/year), which can be attributed to the high strain energy stored during plastic deformation. This strain energy increases the material's Gibbs free energy, making it thermodynamically more reactive and susceptible to long-term corrosion attack[12].

In contrast, the potentiodynamic polarization method measures the instantaneous corrosion tendency over a short time scale. The results of this test actually showed the highest corrosion rate in the as-cast sample (1.449 mm/year). This phenomenon is caused by the presence of a large and continuous Mg2Zn1 intermetallic phase at the grain boundaries[15]. This phase acts as an efficient cathode to the Zn matrix (anode), forming a macro-galvanic cell that significantly accelerates initial corrosion. In terms of surface morphology, the as-extruded sample had smaller, denser, and more uniform grains compared to the as-rolled sample. This naturally affected the corrosion rate of the as-extruded sample, which was slower than that of the as-rolled sample, but still faster than that of the as-cast sample in the weight loss method. Meanwhile, in the potentiodynamic method, the as-extruded sample had the lowest corrosion rate compared to the as-cast and as-rolled samples.

The as-extruded sample performed best in the potentiodynamic test with the lowest corrosion rate (0.050 mm/year). This was due to the fine and homogeneous microstructure resulting from dynamic

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recrystallization during the extrusion process, which facilitated the formation of a uniform and protective passive layer[16]. In conclusion, these mechanical changes did not show significant changes, but there were changes in hardness, surface microstructure, and even corrosion rate. Additionally, the Zn-1Mg alloy has potential as a bioabsorbable implant, but in areas that do not require significant strength, such as the skull, fingers, ribs, or in the medical world for maxillofacial implants and external fixator implants. The difference in results between the two methods provides valuable information for implant applications:

- Initial corrosion resistance (represented by potentiodynamic results) is important to prevent premature implant failure.
- Long-term corrosion resistance (represented by weight loss results) determines the duration of implant degradation.

Based on those results, the as-extruded sample is the best candidate for implant applications because it exhibits excellent initial corrosion resistance and moderate long-term degradation rates. The as-rolled sample is suitable for applications requiring faster degradation, while the as-cast sample is not recommended due to its very poor initial corrosion resistance. All samples demonstrated corrosion rates within the safe daily Zn intake (8-11 mg/day) for adults, confirming the suitability of the Zn-1Mg alloy as a bioabsorbable implant material.

4. Conclusion

The results showed a significant difference between the corrosion rates measured using the weight-loss and potentiodynamic polarization methods. This difference can be explained by fundamental differences in the measurement principles of the two methods. This study demonstrated that processing methods determine the corrosion behavior of Zn-1Mg alloys.

- Potentiodynamic: The lowest instantaneous corrosion rate occurred for extruded alloys, while the highest occurred for as-cast alloys.
- Weight-loss: The highest cumulative corrosion rate occurred for as-rolled alloys, while the lowest was for extruded alloys.

Potentiodynamic reflects the instantaneous surface condition, while weight-loss reflects the total degradation and exfoliation of corrosion products during the immersion time. Overall, the as-extruded Zn-1Mg sample exhibited the most stable corrosion behavior, making it the most suitable for bioabsorbable applications. The as-extruded sample performed best, with excellent initial corrosion resistance (0.01 mm/year) and a moderate long-term degradation rate (0.35 mm/year). All samples had corrosion rates within the safe limits of daily Zn intake, confirming the suitability of the Zn-1Mg alloy for bioabsorbable implant applications, particularly in the as-extrusion condition.

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