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To cite this article: M A F Romzi *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1068** 012004

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240th ECS Meeting ORLANDO, FL

Orange County Convention Center Oct 10-14, 2021



Abstract submission due: April 9

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Recent progress on the corrosion characterization of magnesium (Mg) prepared by powder metallurgy technique

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Abstract. This paper reviews the progress on the corrosion characterization of magnesium (Mg) based material prepared by the powder metallurgy (PM) technique. In recent years, Mg alloys and composites produced by the powder metallurgy technique have been gaining interest in many industries especially in biomedical applications. Mg is also being used to improve battery electrodes and be considered for next-generation secondary batteries with remarkable energy and capacity density. Nevertheless, bare Mg is known to have poor corrosion resistance in most environments with occurrences such as non-uniform corrosion attack, high and fast reaction, microgalvanic corrosion of Mg-matrix and intermetallic particle and formation of non-protective hydroxide film after longer exposure in corrosive environment. New fabrication methods of Mg, such as the PM method are likely to benefit future Mg's mechanical and corrosion properties. Utilizing the latest corrosion characterization method is needed to ensure accurate and precise methods for investigating corrosion behaviour. The recent approach and progress in characterizing Mg's corrosion behaviour, specifically that have been prepared by the PM technique is considered in this paper.

1. Introduction

Magnesium (Mg) is a current lightweight material which is relevant in a wide scope of modern fields, from aviation, automobiles to biomedical applications. Its principle points of interest are good strength to weight proportion, biocompatibility, in blend with biodegradability. In any case, because of the high reactivity of pure Mg in most environment and the poor room temperature strength, making it not so much adequate for building applications. Nevertheless, for the most part, Mg composites and alloys are utilized [1–3]. Unfortunately, even though Mg might have been stable in an atmospheric environment, it is not stable in an aqueous environment since it is more anodic than the other metals, referring to the electrochemical series (EMF) table. Because of that, pitting is likely to occur on Mg due to galvanic corrosion happens on it. Corrosion occurs on Mg when it is in a room temperature environment where a film of oxide occurs on Mg. The moisture in the atmosphere will then converts the MgO to Mg (OH)₂ [4]. Mg is known to improve the mechanical properties of materials and it would be a disadvantage if the corrosion resistance of Mg cannot be improved [5–7]. It is proven that the corrosion resistance of pure Mg does not suit some biomedical and technical applications because of its high porosity values [8–14]. High porosity means the materials will corrode faster. Therefore, this paper will be discussing the recent techniques used by the researchers to characterize the corrosion behaviour of Mg prepared by powder metallurgy technique.



2. Powder metallurgy of Mg

Powder metallurgy (PM) is characterised as the pressing of fined powders to bond a strong piece or material. There are five procedures in the powder metallurgy including the powder creation, blending, compaction, sintering and secondary processing. PM technique is used in Mg production to form a layer of thermodynamically stable corrosion products on its surface. The layer produced prevents diffusion processes which is needed for the densification of material during the PM process. Usually, argon and nitrogen are to be used as a protective atmosphere, especially during the sintering process since Mg is highly reacted to oxygen [15].

There have been some discussions regarding the suitability of the PM technique towards Mg alloys production. It is known that the PM technique is effective since it can help to combine metals with different densities and melting points of materials. The PM technique will affect the microstructure of Mg and then will also affect the corrosion behaviour of the alloys produced. Besides, the limitations are also being discussed, for example, the hot pressing process in producing Mg powders caused the powder particles to undergo high plastic deformation which leads to the leakage of the corrosion layer which usually present on the surface of particles [7,16]. When this thing happens, the contact area of the particles will increase, enhancing the diffusion process and then will result in decreased porosity of the materials when processing in bulk production.

Zhao et al. [17] reported that there was improvement in terms of compressive strength when alloying Mg-Zn-Zr-3Y using the PM technique. It was happened due to strain, precipitation and fine crystal strengthening which caused the performance improvements as highlighted by [18,19]. Shi et al. [20] proved that during the compression test which was done according to CMT4305 at ambient temperature, the compressive strength will be improved as well as the matrix alloy which shows the role when suppressing twinning deformation.

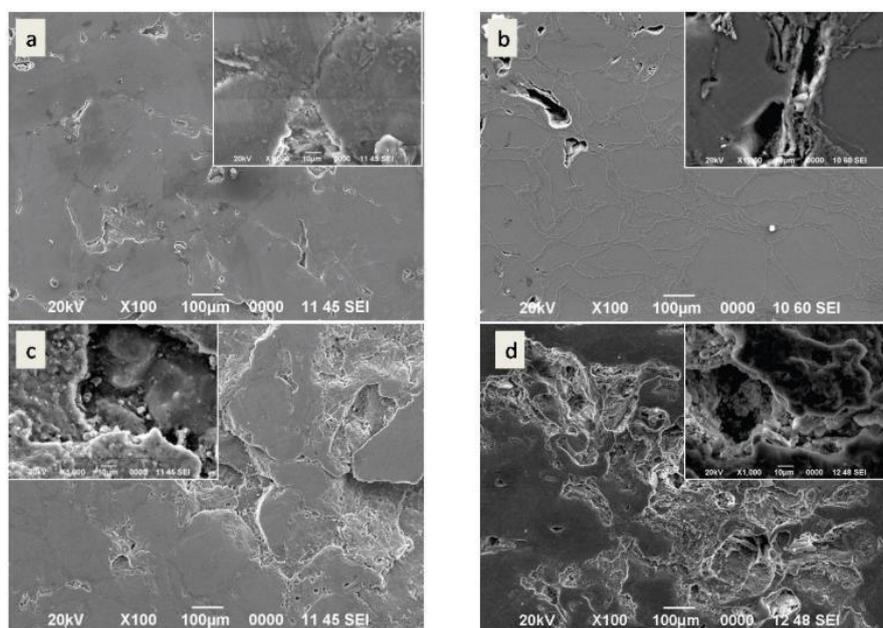


Figure 1. SEM images of a) Mg-Zn-Ca-580, b) Mg-Zn-Ca-630, c) Mg-Zn-Ca10Carb-580, and d) Mg-Zn-Ca-10Carb-630 [21].

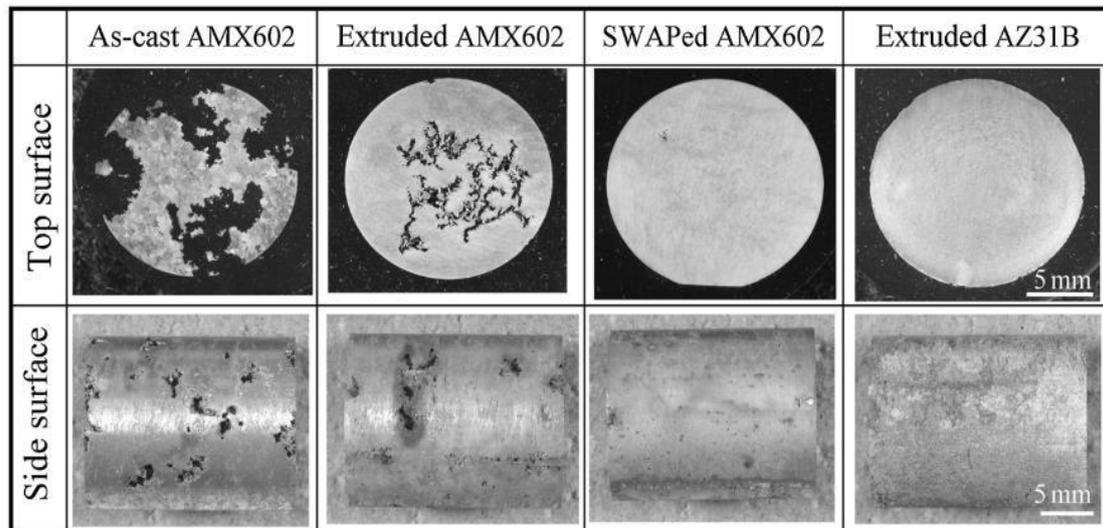


Figure 2. Micrographs of Mg alloys after immersed in 0.1M of NaCl solution for two weeks [22].

Meanwhile, Annur et al. [21] used a space holder agent to create porous Mg-Zn-Ca which was also used by [23–27]. By adding Mg-Zn Ca mixture powder with carbamine particles, the porosity of the Mg alloy was increased even though the mechanical properties were decreased. Figure 1 shows the scanning electron microscopy (SEM) images of the as-sintered alloys. Aghion et al. [24] also found the same effect where the addition of space holder resulted in the increase of porosity, but they also reported that a high porosity resulted decreasing in corrosion resistance when exposed to human body fluid. Liao et al. [22] observed the pitting occurs on the as-cast and extruded AMX602 alloys, whereas for the SWAPed AMX602 and AZ31B alloys, the corrosion is not obvious. Figure 2 shows the micrographs indicating pitting and filiform corrosion occurs in the samples immersed in 0.1M of NaCl solution for two weeks.

Čapek et al. [28] performed a sintering process under two types of atmosphere with 0.1L/min flowrate which are argon gas with 99.996% purity and 55 mm thick of Mg chips purified argon which acted as a getter. The results suggested that sintering under getted argon atmosphere improve the Mg particles diffusion, hence slightly increase the porosity. Rashad et al. [29] added Al-GNP into Mg by mixing Al and Mg using ethanol solution using mechanical agitator. The graphene nanoplatelets (GNP) were ultrasonicated for 60 minutes in ethanol and then being dropped into the mixture of Al and Mg, which later the mixing process will be continued for 60 minutes to obtain homogeneity. The addition resulted in improvements in tensile, strain and yield strength of Mg.

3. Corrosion behaviour of PM Mg and the applied techniques

Some researchers use variation of techniques to characterise the corrosion behaviour of Mg and Mg alloys specifically produced by the PM method. The techniques including the electrochemical characterization, potentiostatic and potentiodynamic polarization, electrochemical impedance spectroscopy (EIS), immersion test, hydrogen evolution test and scanning vibrating electrode technique (SVET). Some of the researchers in the biomedical field use electrochemical tests where the test was carried out in simulated body fluid including Hank's Balanced Salt Solution (HBSS), enriched Hank's Balanced Salt Solution (HBSS) and also in NaCl solution highlighted by [2,30–32].

The EIS provides information about the characteristics of material degradation when going through a corrosion medium. EIS can also be evaluated from other solution such as simulated body fluid (SBF) which was highlighted by [2]. From the data obtained from Nyquist plots, equivalent circuit (EC) is used to determine the characteristic of the material corrosion as shown in Figure 3. Figure 4 shows the EIS plots of the electrochemical behaviour of PM Mg under the different conditions highlighted by [16].

Brezina et al. [16] proved that for hot pressed Mg materials prepared under 500 MPa in a corrosion medium, the highest corrosion resistance was achieved. This was proven by higher polarization resistance after longer corrosion experiment duration compared to other material conditions. More pronounced corrosion attack of samples in HBSS compared to enriched HBSS is shown from experiments. Zhang et al. [33] discussed that when Mg alloy was put into HBSS, the Cl⁻ presence will increase the reaction rate when reacting to Mg(OH)₂ and creating MgCl₂ which were highlighted by [34]. From their findings, they suggested that the increase of Zn content resulted in the polarization curve becoming longer which also meant that Zn is beneficial as a protective film for Mg. Yan et al. [1] also used Ringer's solution to get the pH values of Mg-Zn against time. The samples were immersed for 24 hours and the pH increased slowly until the peak of stabilized pH values. The increment of pH values means extra OH⁻ is generated and slower increasing of pH values means a lower rate of corrosion as highlighted by [35].

Zheng et al. [36] plotted three Nyquist plots of Mg/Ca composites in DMEM for different immersion times. In the curves obtained, the capacitance loop at high frequency and the other at low frequency are set as constants. For Mg/10Ca composite sample, within 12 hours of immersion, R_f and R_t increased and slowly decreased after being immersed for 72 hours. It shows that the protection effect of the corrosion product layer is enhanced at the early stage of immersion and becomes worse when duration of immersion is increased which was agreed by [37].

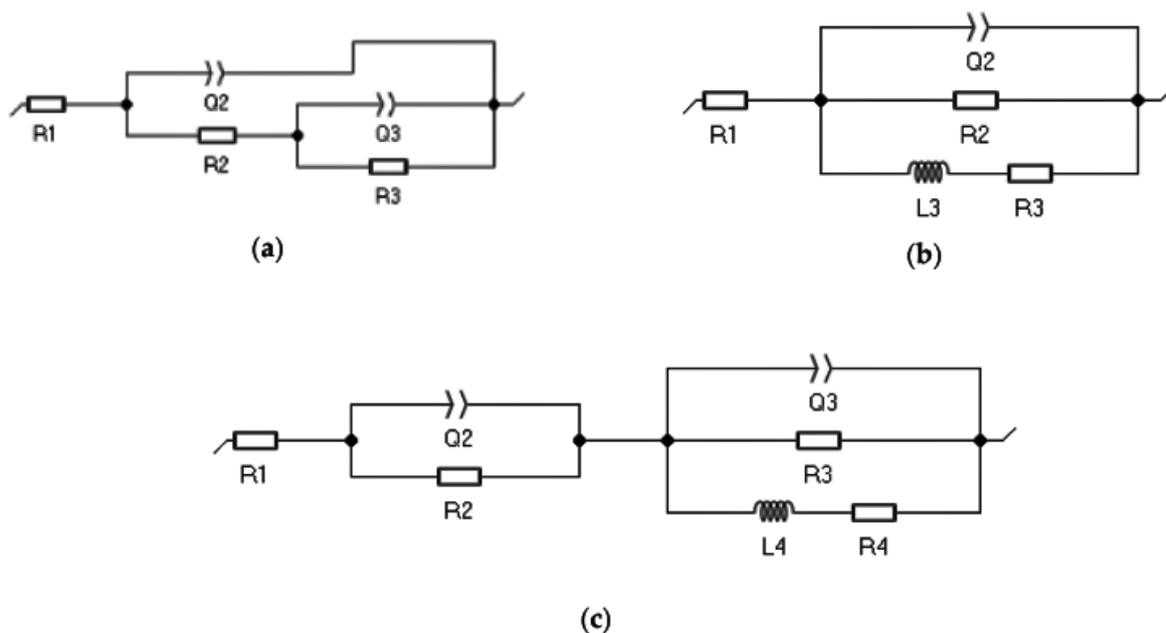


Figure 3. EC used for Nyquist plots evaluation [16].

Liao et al. [22] meanwhile use NaCl solution as a medium to immerse three AMX602 alloys and AZ31B alloy for the electrochemical test. The samples were immersed in 500 ml NaCl solution with ratio of the area of the samples and the volume of NaCl 1:35. After the immersing process, the corrosion products were cleaned away using a solution containing a boiling 10% CrO₃ chromic acid, and later rinsed with water. Then, the samples are cleaned ultrasonically using acetone, dried and then weighed to measure the weight loss. Figure 5 shows the plot of weight loss (mg/cm²) of AMX602 and AZ31B Mg alloys due to corrosion.

Hydrogen evolution (HE) accompanied Mg dissolution in aqueous solutions. HE controls oxygen reduction at all values of pH where the potentials at Mg dissolves are low. In aqueous solution, Mg and all its alloys exhibited open circuit potential values less than about -1.45 V_{SCE} [38]. Thomas et al. [39] defined that the negative different effect (NDE) is a phenomenon of cathodic hydrogen evolution at anodically polarised Mg which is categorised by hydrogen evolution reaction (HER) rate increasing with anodic polarisation which is mentioned by [36],[40]. Zheng et al [36] found in cathodic polarization

current that hydrogen evolution of Mg/1Ca composites is much lower than Mg/5Ca and Mg/10Ca samples. The data was obtained from potentiodynamic polarization curve which is also highlighted by [1,2,22,32,36,40,41]. Figure 6 shows the potentiodynamic polarization curve of Mg/1Ca, Mg/5Ca and Mg/10Ca composite samples in Dulbecco's Modified Eagle Medium (DMEM).

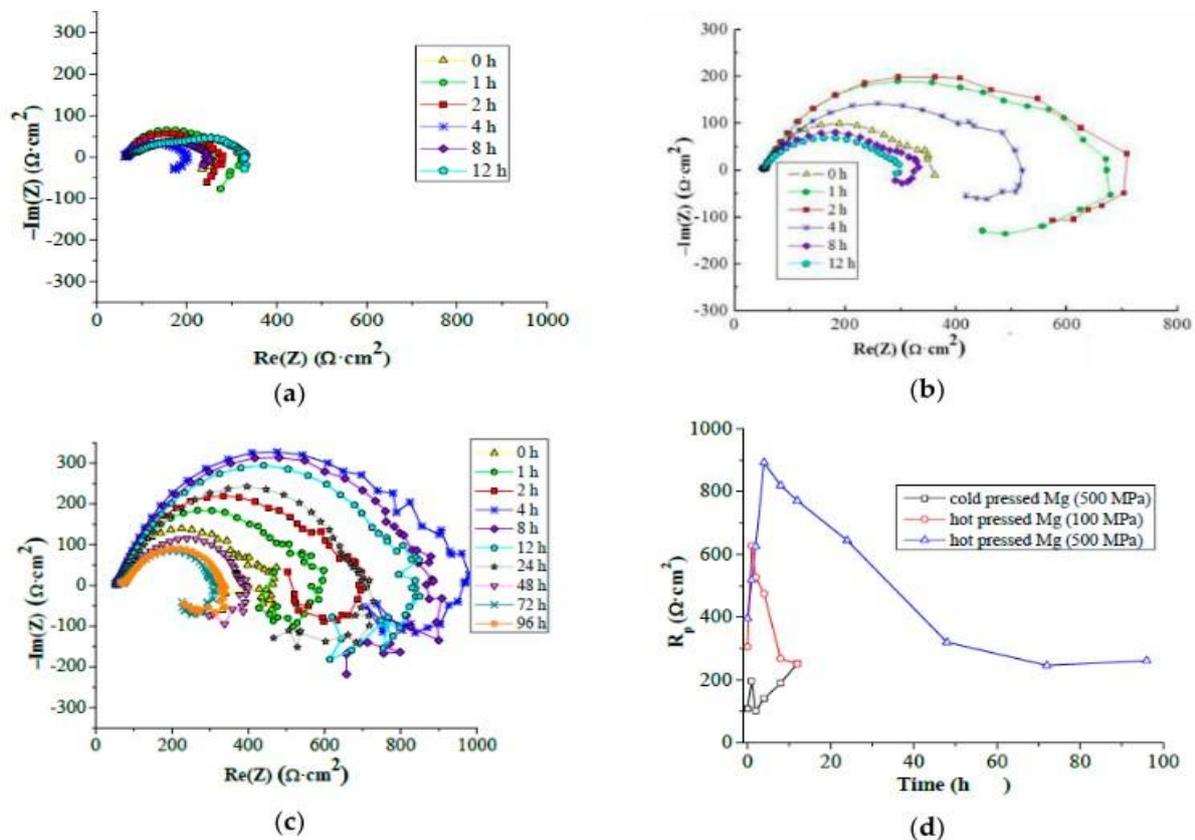


Figure 4. PM Mg EIS behaviour under different condition of pressing temperature and power [16].

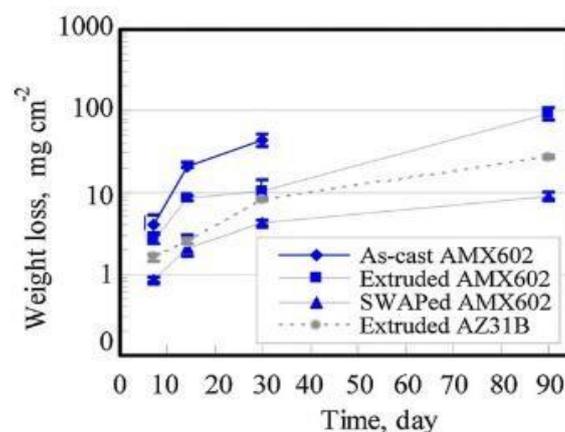


Figure 5. Plot of weight loss against time for three AMX602 and AZ31B alloys during immersion test in NaCl solution [22].

Potentiodynamic polarization curve is also an electrochemical test which is normally done for Mg in SBF. Kubásek et al. [2] performed the potentiodynamic and EIS test under SBF at 37°C which resulted that WE43 PM alloy is characterised by slightly higher corrosion potential than the WE43

IM. Pereda et al. [32] immersed the Mg (PM) in PBS solution for 1 hour to 168 hours. The PBS consists of 8g l^{-1} NaCl, 0.2g l^{-1} KCl, 0.2g l^{-1} KH_2PO_4 , 1.15g l^{-1} Na_2HPO_4 . Akinwekomi et al. [40] revealed that materials that exhibit more positive corrosion potential (E_{corr}) and lower corrosion current density (i_{corr}) show that there were enhanced corrosion resistance compared with materials that exhibit opposite behaviours as shown on Tafel curves on Figure 7.

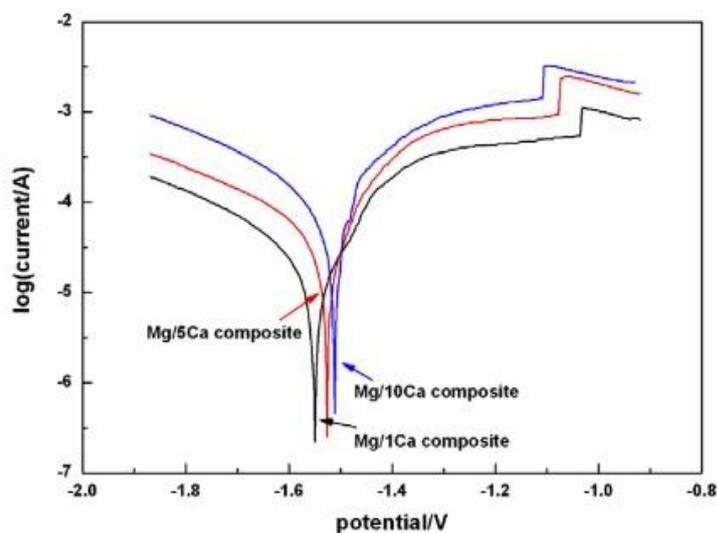


Figure 6. Potentiodynamic polarization curve of Mg/1Ca, Mg/5Ca and Mg/10Ca composite samples in DMEM [36].

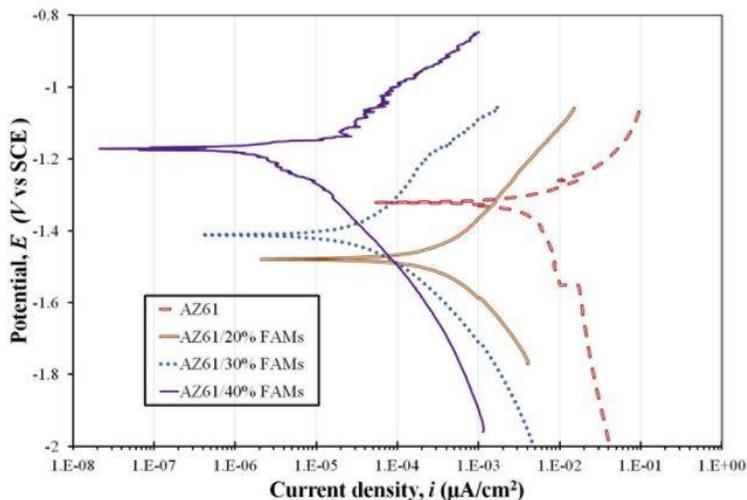


Figure 7. Potentiodynamic polarization curves of AZ61 and AZ61/FAMs syntactic foams [40].

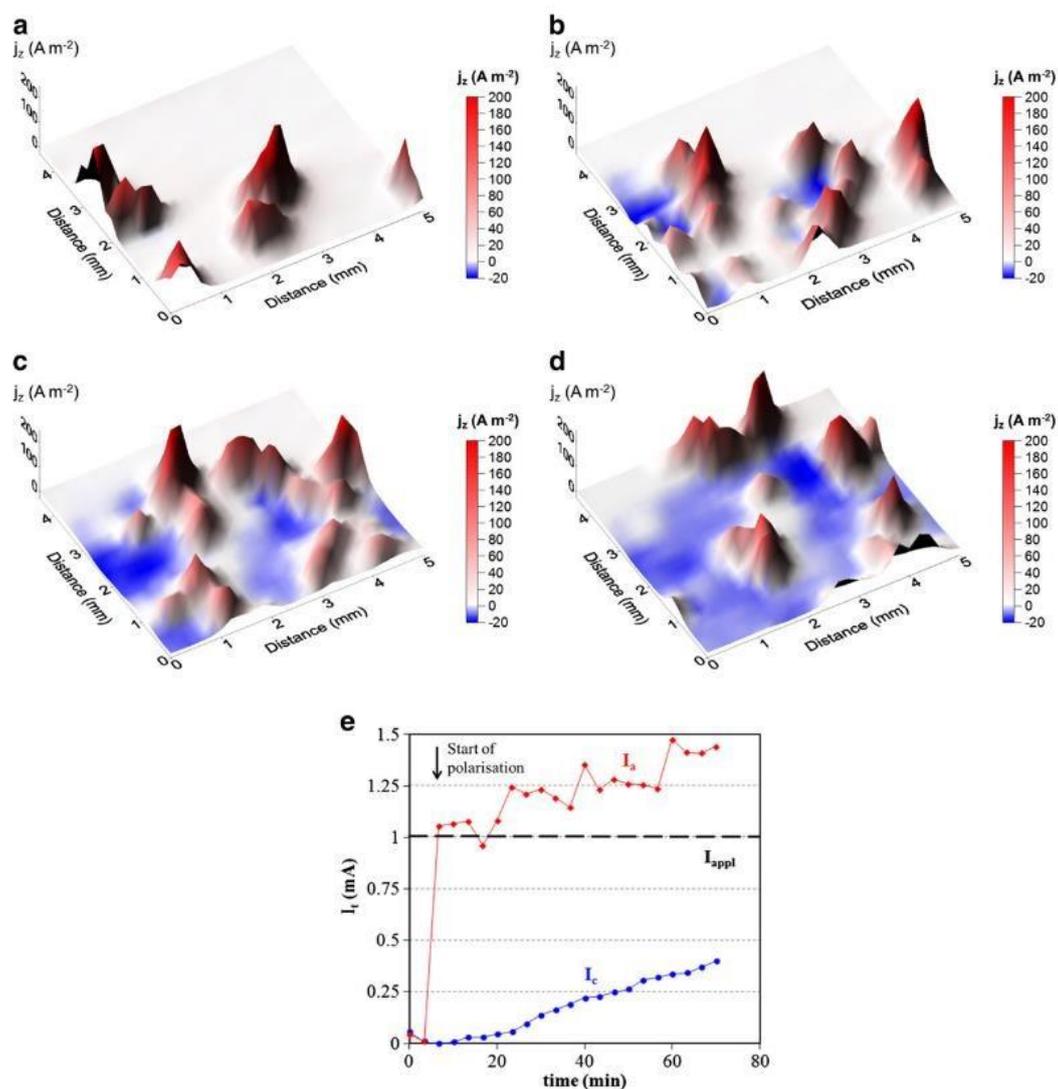


Figure 8. SVET maps on Mg surface immersed in NaCl (aq) electrolyte, a) 7 mins, b) 28 mins, c) 48 mins and d) 68 mins after starting galvanostatic polarisation at $+1 \text{ mA cm}^{-2}$ and e) SVET-derived integrated current (I_t), normalised for a 1 cm^2 area, galvanostatically [42].

Scanning vibrating electrode technique (SVET) is used to characterise the anodes and cathodes distributions on pure Mg during anodic polarisation in NaCl solution. Williams et al. [42] use electrical contact which was made to high purity Mg rod of 10 mm diameter and 8 mm length. The specimen was then cold mounted in two-part epoxy resin so that one of the circular faces of the cylinder was exposed. Based on Figure 8, it was observed that some Mg areas become anodes, which later changes to cathode hence sustaining the HER which is agreed by [43]. Table 1 shows the list of researches of PM Mg and Mg alloys with the corrosion characterization technique.

Table 1. Lists of the corrosion characterization technique applied for PM Mg and Mg alloys.

Author	Year	Alloys	PM routes	Corrosion characterization methods
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Balog et al. [44]	2019	Ti-Mg	Compacted by CIP at 200MPa-extruded at 400-500°C	<ul style="list-style-type: none"> • Immersion test
Akinwekomi et al. [40]	2019	AZ61/fly ash	Sintered for 20 minutes at 550°C	<ul style="list-style-type: none"> • Electrochemical characterization • Microstructural characterization
Kubásek et al. [2]	2017	Mg-4Y-3RE-Zr	Compacted under HVP at 425MPa and 350°C for 1 hour-extruded at 400°C and 0.2mm/s rate	<ul style="list-style-type: none"> • Immersion test • Potentiostatic and potentiodynamics test • Open circuit potential measurements
Březina et al. [16]	2017	Pure Mg	Compaction under nitrogen atmosphere-compacted at pressure 100MPa, 200MPa, 300MPa, 400MPa and 500MPa	<ul style="list-style-type: none"> • Microstructure observation • Potentiostatic • Electrochemical impedance spectroscopy (EIS)
Yan et al. [1]	2016	Mg-Zn	Mix for 8 hours-cold pressed-sintered at 500550°C for 2 hours in vacuum sinter furnace under argon gas	<ul style="list-style-type: none"> • Immersion test • Electrochemical test
Razak et al. [45]	2013	Al-Mg	Compact using manual hydraulic press at 210 MPa- sintered at 610°C argon gas for 6 hours	<ul style="list-style-type: none"> • Electrochemical characterization
Liao et al. [22]	2012	Mg-AlMn-Ca	Atomized by spinning water atomization process-compacted at 400MPa-hot extruded at 573-623K	<ul style="list-style-type: none"> • Immersion test • Microstructural characterization • Electrochemical characterization
Zheng et al. [36]	2010	Mg-Ca	Cold pressed at 400MPahot pressed at 350MPa at 320°C-hot extruded at 300°C	<ul style="list-style-type: none"> • Immersion test • Electrochemical test • Potentiodynamic test
Pereda et al. [32]	2010	Pure Mg	Cold-pressed 310MPa extruded at 420°C-	<ul style="list-style-type: none"> • OCP measurements • Electrochemical characterization and EIS • Potentiodynamic and potentiostatic • Surface characterization of corroded Mg (PM)

4. Conclusion

- The review offered here recognize the application of corrosion test to characterize the corrosion behavior of Mg that is specifically produced by PM technique. The use of PM technique provides the route to create a high strength and better properties of Mg, while at the same time pertaining cost effective and less complex production of Mg alloys and composites.
- Accurate results of corrosion behaviour can help to verify the performance of Mg alloys in a corrosive medium, hence producing Mg with the best properties. The most common and reliable strategy to assess the corrosion behaviour is performing electrochemical test such as EIS and potentiodynamic polarization that can tailor the surface properties of Mg to the microstructure developed during the PM route. The use of SVET test also effectively provides important information on the anodic and cathodic reactions, that is best approach to understand the corrosion mechanism of Mg.

Acknowledgement

The authors wish to acknowledge the financial support of Universiti Malaysia Pahang (RDU180333) and Ministry of Higher Education (FRGS/1/2019/TK05/UMP/02/5) research grants.

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