Electrodeposition onto magnesium in air and water stable ionic liquids: From corrosion to successful plating

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Abstract

Magnesium is very reactive and therefore magnesium electroplating in aqueous solutions is hazardous. Mg is classified as a water sensitive substrate from the electrodeposition point of view. Therefore, it was suggested that ionic liquids be used as electrolytic solvents for electrodeposition onto Mg and its alloys. Five air and water stable ionic liquids based on choline chloride (ChCl) were investigated to deposit Zn onto several Mg alloys in air. The ChCl/urea mixture was found to be the most feasible liquid for successful electrodeposition. Other liquids produced powdery deposits or resulted in corrosion of Mg substrate. Application of a pulsed current was superior to a constant current in producing smooth, sealed and more corrosion-resistant Zn layers.
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1. Introduction

Magnesium is unable to form a self-healing protective passive film, and suffers high micro-galvanic corrosion when it contains noble phases or impurities. Macro-galvanic deterioration occurs if it is in contact with cathodic metals in a wet environment [1–5]. Consequently, widespread use of magnesium alloys to save weight in applications such as automobiles, aerospace, electronics, etc. necessitates the successful application of appropriate protective coatings, which can withstand service conditions.

The authors contribution [6,7] was laser cladding of a coating of a Al–Si eutectic alloy on Mg surfaces. The corrosion rate was reduced by two orders of magnitude compared to the un-coated surface. However, the process parameters for this were narrow and control of the power input was necessary to prevent overheating of the Mg substrate and formation of pores in the clad layer. Recently

[8,9], surface modification with hydrogen, by electrochemical ion reduction or by plasma immersion ion implantation, has been developed as a technique for producing a magnesium hydride coating on magnesium and its alloys. It was shown that a significant improvement in the corrosion resistance took place but was strongly dependent on the alloying elements. A detailed review [10] of the possible technologies currently available was carried out. Also, a number of patents, which claim to have coating processes for Mg, have been made recently. It has been concluded, however, that no single coating technology has been developed, which functions adequately to protect magnesium from corrosion in harsh service conditions. Thus much research is still required to develop better, simpler and cheaper coatings for Mg and its alloys.

One of the coating technologies which is claimed to be successfully applied for magnesium is electroplating. However, magnesium and its alloys have been classified as difficult to electroplate [10] because of: (1) extreme corrosion attack on Mg substrates by aqueous electrolytes, (2) high reactivity of Mg which rapidly forms loose immersion layers on the surface by replacement, which inhibit successive

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electrodeposition, and (3) rapid formation of MgO and/or Mg(OH)$_2$ film, as soon as the Mg is exposed to air or water, which prevent adhesion of the electrodeposited layers [5,10,11].

Consequently, Mg and its alloys are considered to be water sensitive substrates, and thus the importance of ionic liquids as non-aqueous electrolytes in electrodeposition. The objective of the present study was the electrodeposition of Zn onto magnesium and some of its alloys by using two recently invented types of ionic liquid solvents at ambient temperatures [12,13]. One type of these novel ionic liquids is formed, as stated in the invention [12], by mixing choline chloride (ChCl) – as an amine salt – with a hydrogen bond donor such as urea, ethylene glycol, malonic acid, or glycerol with appropriate molar ratios. The other type is formed by mixing ChCl with a metal halide [13]. These ionic liquids are not only non-aqueous electrolytes but they are stable in air and water, and thus electrodeposition is possible in a room atmosphere at ambient temperatures. The physical and chemical properties of these ionic liquids are given in many references [12–17].

To the authors knowledge, electrodeposition onto Mg substrates in ionic liquids has not been studied. The present paper aimed to show the feasibility of electrodeposition of Zn onto Mg alloys in five ionic liquids. The results of successful electrodeposition experiments, as well as unsuccessful ones, are presented. Also, the microstructure and corrosion behaviour of successfully deposited Zn layers are described.

2. Experimental details

The ionic liquids were prepared and provided by Scionix Ltd. The chemical compositions and abbreviations used are given in Table 1. The water content was measured by Karl–Fisher Titration method at 60 °C. The preparation of the electrodeposition baths involved heating the ionic liquids to 90 °C and dissolving 0.5 M of ZnCl$_2$ with gentle stirring until a clear colourless liquid was formed. All electrochem-

ical measurements and electrodeposition experiments were carried out open to air.

The magnesium alloys used as substrates for electrodeposition were: Cp Mg (99.99 Mg), AZ31 (3 Al, 1 Zn, 0.2 Mn), AZ61 (6 Al, 3 Zn, 0.15 Mn), AZ91 (8.7 Al, 0.7 Zn, 0.13 Mn), AS41 (4.37 Al, 0.93 Si, 0.35 Mn), AE42 (4 Al, 2.5 RE, 0.1 Mn), WE43-T6 (4 Y, 3.4 RE), QE22 (2.5 Ag, 2.1 RE), MgGd5Sc1 (4.64 Gd, 0.26 Sc, 1.53 Mn) and MgY4Sc1 (3.88 Y, 0.73 Sc, 1.11 Mn). The numbers before each element express the weight percentage.

Mg specimens were cut into $20 \times 20 \times 6$ mm blocks. A hole in one of the 20×6 mm sides was drilled and tapped so that a copper rod could attached to it. Each specimen was ground on 2400 grit SiC paper on a wide side. The specimen was then subjected to standard industrial pretreatment, including alkaline degreasing, acidic pickling and activation. The specimen was finally fixed to the copper rod and coated by an appropriate lacquer exposing a free surface of $\sim18 \times 18$ mm to be electroplated.

Electrochemical measurements to study the corrosion of Mg in ionic liquids were carried out in a conventional three-electrode cell using the model "Wenking LB 94 L (Auto Range) Laboratory Potentiostat", controlled by a PC. The Mg specimen was immersed in 50 ml of the ionic liquid for 15 min prior to polarisation. A stable free corrosion potential (open circuit potential "OCP") was monitored in this time. A Pt wire electrode, as quasi-reference electrode, was used. The polarisation was obtained by scanning from ~400 mV more negative than the OCP to more positive potentials at a rate of 20 mV/min. A Pt sheet of 1 cm^2 was used as counter electrode. The corrosion potential "E_{corr}" and corrosion current "I_{corr}" were typically determined by extrapolation of the Tafel lines of each polarisation curve using the soft ware program CPCDAU 41.

For comparison purposes, the electrochemical behaviour of Cp Mg and low carbon steel (grade A 516, with nominal wt% composition: 0.21 C, 0.13–0.45 Si, 0.55–0.98 Mn, 0.035 P and 0.040 S) was studied in a classic aqueous zinc plating electrolyte (~500 ml) at 25 °C. This electrolyte contains 150 g/l KCl and 23 g/l H$_3$BO$_3$ [18]. The procedure applied was the same as for measuring the corrosion of Mg in ionic liquids, but the reference electrode was saturated calomel electrode (SCE).

The cyclic voltammogram measurements were performed using the same potentiostat and the same set of electrodes demonstrated above to measure the electrochemical behaviour of Mg in ionic liquids. The potential was scanned from ~200 mV more positive than the OCP in the negative direction with a rate of 1 mV/s. Scanning continued until a definite potential (-2000 or -2500 mV vs. Pt) , and then reversed in the noble direction and continued up to 500 mV vs. Pt. Thereafter, it was reversed again in the negative direction down to the initial potential.

In the case of galvanostatic electrodeposition constant and pulsed currents were generated by a Potentio-Galvano-scan (model Wenking PGS 95) controlled by a PC. The Mg

Table 1
Chemical compositions, abbreviations and water contents at 60 °C of ionic liquids used

Ionic liquid mixture	Abbreviation	Water content (wt%)
1 Choline chloride (HOC$_2$H$_4$N(CH$_3$)$_3$$^+Cl^-$):2 urea (NH$_2$CONH$_2$)	ChCl/urea	3.5
1 Choline chloride:2 ethylene glycol (HOCH$_2$CH$_2$OH)	ChCl/EG	5.6
1 Choline chloride:2 glycerol (HOCH$_2$CH(OH)CH$_2$OH)	ChCl/Gl	2.9
1 Choline chloride:1 malonic acid (HOOCCH$_2$COOH)	ChCl/Mal	4.6
1 Choline chloride:2 zinc chloride (ZnCl$_2$)	ChCl/ZnCl$_2$	7.2

specimen was connected as a cathode and immersed in a 50 ml electrolyte. The anode was a Pt sheet of 17×17 mm^2. The distance between the electrodes was maintained at 35 mm.

Microstructural investigations of both planar outer surfaces and cross sections of electroplated specimens were conducted using an optical microscope and a scanning electron microscope (SEM) coupled with an energy dispersive X-ray analyser (EDX).

Aqueous corrosion testing of final deposited layers was performed by electrochemical potentiodynamic polarisation, using a laboratory potentiostat (model Wenking LB 94L), with an Avesta-cell [19] containing 0.1 M NaCl solution. Prior to polarisation, the specimen was exposed to about 0.5 l of the solution for 15 min. During this time, the OCP was obtained. The polarisation was carried out with reference to SCE, by scanning at a rate of 20 mV/min from ∼400 mV more negative than the (OCP), and the current passing from the specimen to the counter electrode (Pt) was monitored by a PC. The E_{corr} and I_{corr} were determined using the same soft ware program mentioned above to determine the corrosion parameters of Mg in ionic liquids. The corrosion parameters exhibited below are the average of two experiments.

3. Results and discussion

3.1. Electrochemical behaviour of Mg in the ionic liquids

Apart from a recent study [20], which dealt with the corrosion behaviour of pure Mg and AZ91-Mg alloy in a ionic liquid 1-butyl-3methyl-imidazolium trifluoromethylsulfonate ([BMIm]CF$_3$SO$_3$), there was no research, to date, on the corrosion of Mg in ionic liquids. The study showed that the corrosion of Mg is strongly dependent on the water content in the ionic liquid. An addition of 100 ppm H$_2$O increased the corrosion rate of AZ91 Mg alloy to by one order of magnitude. The contamination by water reduced also the resistance of Mg to pitting in the ionic liquid.

The preliminary experiments to electroplate Mg using ionic liquids showed no successful plating and corrosion of Mg substrates. Therefore, it was necessary to study the electrochemical behaviour of Mg in various ionic liquids. Fig. 1 and Table 2. show that Mg has the lowest corrosion rates in ChCl/urea and ChCl/Gl liquids. However, Mg suffers pitting in ChCl/Gl; when the anodic polarisation potential increases, the current density increases sharply. Consequently, ChCl/urea was suggested to be the most likely suitable ionic liquid used as a solvent for electrodeposition onto Mg substrates.

The electrochemical characteristics of Cp Mg and low carbon steel in an aqueous electrolytic solvent of ZnCl$_2$ for electrodeposition of Zn is shown in Table 3. In a comparison observation of Tables 2 and 3, it is clear that the corrosion rate of Mg in the aqueous electrolyte is extremely higher than that in the ionic liquids. The I_{corr} of Cp Mg in the aqueous electrolyte is 7732 times higher than that in the
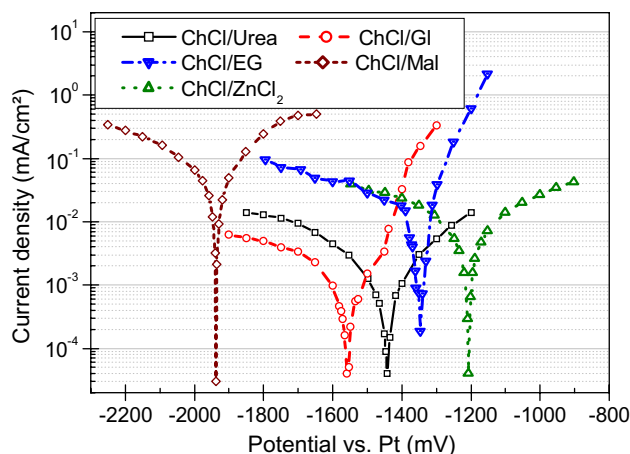

Fig. 1. Potentiodynamic polarisation curves of Cp Mg in different ionic liquids at 60 °C.

ChCl/urea ionic liquid. Also, the I_{corr} of Cp Mg in the ChCl/urea is about 0.004 of the I_{corr} of low carbon steel in the aqueous electrolyte. This means that, in spite of presence of small water contents in the ionic liquids studied, the corrosion rate of Mg is still greatly lower than that of low carbon steel, the most common successfully Zn-electroplated substrate, in the aqueous electrolyte.

3.2. Voltammetric studies

Fig. 2 shows typical cyclic voltammograms of Pt and WE43 Mg alloy electrodes in ChCl/urea liquid containing 0.5 M ZnCl$_2$, and also Mg alloy in pure ChCl/urea. The reduction potentials for Zn from ChCl/urea +0.5 M ZnCl$_2$ on Pt sheet and on Mg alloy are different (−1.2 V and −1.7 V, respectively). This suggests that the kinetics of Zn reduction differ on the two substrates. The forward cathodic polarisation part of Mg alloy in ChCl/urea (encompassed in a separated diagram in Fig. 2) indicates that no significant decomposition occurs less cathodic than −2 V (vs. Pt). However, in closely related experiments, the decomposition potential of ChCl/urea on Pt was determined at −1.2 V (vs. Pt). This wide difference between the decomposition potentials on Pt and Mg alloy can be ascribed to the slower kinetics of decomposition on Mg. A significant difference of decomposition potential values for ChCl/urea has been also reported on Pt and Zn substrates [16].

The cyclic voltammogram recorded on Mg in ChCl/urea +0.5 M ZnCl$_2$ shows that the forward–reverse scan after a reduction potential peak of Zn forms a clear current loop which is typical for the nucleation process. This suggests that, on the Mg substrate, the rate of deposition process is mostly nucleation controlled. In a comparison observation between the voltammograms on Mg alloy polarised down to different negative potentials, it is clear that the more the negative sweep, i.e. the higher the reductive charge, the higher is the oxidative charge and the wider is the stripping peak. This presumes that the bulk

Table 2
Electrochemical parameters obtained from the polarisation curves of Cp Mg in different ionic liquids at 60 °C

Ionic liquid	pH	OCP (mV vs. Pt)	E_{corr} (mV vs. Pt)	I_{corr} (μA/cm^2)
ChCl/urea	6.90	−1531	−1446	0.48
ChCl/Gl	4.55	−1594	−1557	0.49
ChCl/ ZnCl$_2$	0.50	−1253	−1207	3.86
ChCl/EG	4.30	−1489	−1347	12.72
ChCl/Mal	0.45	−1962	−1940	31.58

Table 3
Electrochemical parameters of Cp Mg and low carbon steel obtained by polarisation in aqueous acid chloride zinc plating electrolyte at 25 °C, pH 5

Specimen	OCP (mV vs. SCE)	E_{corr} (mV vs. SCE)	I_{corr} (μA/ cm^2)
Cp Mg	−1863	−1857	3711.17
Low carbon steel	−664	−651	109.42

deposition of Zn on Mg continues with increasing the negative charge regardless the decomposition of the ionic liquid. The successive increase of current, followed its gradual decrease after oxidation peaks for Zn on Mg, is due to the corrosion of Mg alloy substrate. Note that the cyclic voltagrams on WE43 Mg alloy in ChCl/urea + 0.5 M ZnCl$_2$ are not shown entirely but only up to 0.0 V to avoid the complicating of the figure.

3.3. Galvanostatic electrodeposition

The possibility of electrodeposition of Zn onto Mg from ionic liquids based on ChCl/EG was investigated. No successful deposition was obtained, but rather significant corrosion of the Mg substrate resulted. The severity of corrosion increased on increasing the applied current density and the concentration of ZnCl$_2$, as shown in Fig. 3.
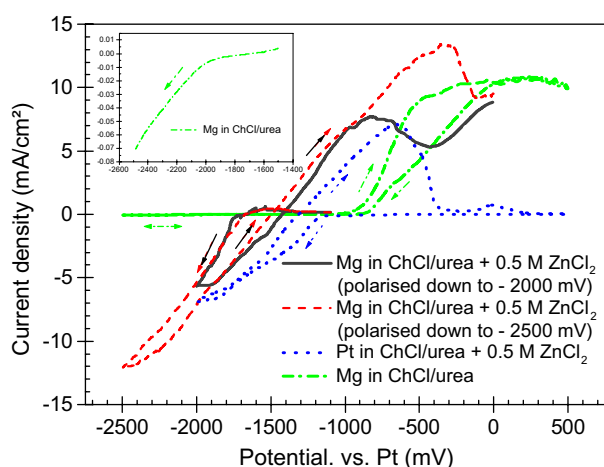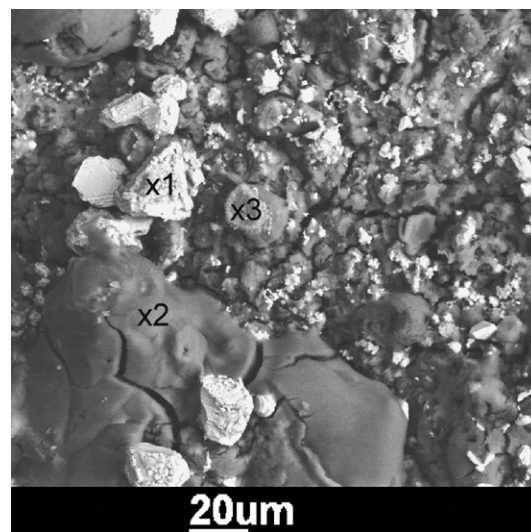

Fig. 2. The voltammograms for Pt sheet electrode and WE 43 Mg alloy in the ChCl/urea with and without 0.5 M ZnCl$_2$. Scan rate = 1 mV/s and $T = 60$ °C.

Fig. 4. SEM micrograph of electrodeposited Zn onto AZ91 Mg alloy from the ChCl/ZnCl$_2$ at 5 mA/cm^2, $T = 60$ °C.
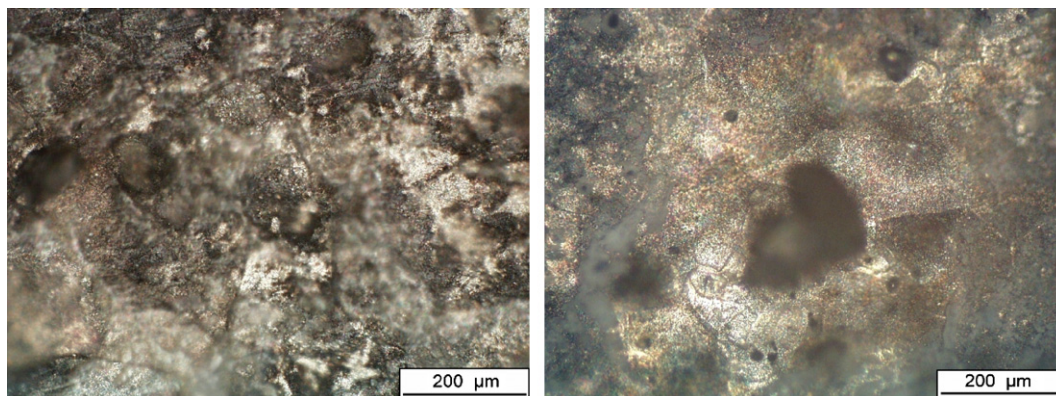

Fig. 3. The micrograph of WE43-Mg substrate after deposition trial of Zn from ChCl/EG + 0.5 M ZnCl$_2$ at 5 mA/cm^2 (left), and from ChCl/EG + 1 M ZnCl$_2$ at 10 mA/cm^2 (right), $T = 60$ °C.

Table 4
EDX elemental analysis of selected points revealed in Fig. 4

	Zn	Mg	Al	O	Cr	F
X1	94.66	01.27	00.45	02.57	0.08	00.85
X2	00.67	33.16	01.06	56.61	0.06	08.25
X3	08.97	35.69	18.68	25.49	0.45	10.12

Also, the ionic liquid $ChCl/ZnCl_2$ was used to deposit Zn onto many Mg alloys including WE43 Mg alloy. In all alloys tested, a powdery dull layer was obtained. Fig. 4 shows a representative micrograph of the obtained layer on a Mg alloy. The SEM/EDX compositional analysis, Table 4, indicates that only few Zn particles, indicated by "×1" in Fig. 4, are electrodeposited on Mg substrate. The substrate is covered with a layer of corrosion products consisting mainly of Mg oxide rich in F and Cr, the main elements of the chemical pre-treatment.

Electrodeposition from the ionic liquid $ChCl/urea + 0.5\ M\ ZnCl_2$ by applying constant current densities onto pure magnesium and different magnesium alloys was attempted. It was found that the success of electroplating depends strongly on the chemical composition of the Mg alloy substrate. The Mg alloys WE43, QE22, MgGd5Sc1 and MgY4Sc1, which contain rare earth (RE) elements and are free of Al, have been successfully electroplated and had dense, compact and adherent deposited Zn layers.

Different values of constant current densities (2.5, 5, and $10\ mA/cm^2$) were applied. Both a low current density ($2.5\ mA/cm^2$) and a high current density ($10\ mA/cm^2$) produced porous deposited layers. Application of a current density of $5\ mA/cm^2$ produced dense layers and was used to test the possibility of Mg alloys to be electroplated. Fig. 5 shows the microstructure of the outer surface of Zn layers deposited at $5\ mA/cm^2$ for 4 h. The Zn particles deposited in the shape of sharp-edged parallelogram. A relatively finer microstructure was obtained by adding EDTA to the electrolyte as an agent to make it complex, see Fig. 5a and b. Thus, the EDTA addition only reduced

the size of Zn particles, maintaining the shape and type of microstructure.

The cross-sectional micrographs showed good adherence of the deposited Zn layers onto Mg alloy from the $ChCl/urea + 0.5\ M\ ZnCl_2$ at 60 °C and CD = $5\ mA/cm^2$, see Fig. 6. However, microscopic observations at high magnification indicated the presence of micro-cracks perpendicular to the deposit surface within the Zn layer. The presence of these micro-cracks explains why some Zn-coated specimens have an electrochemical behaviour,
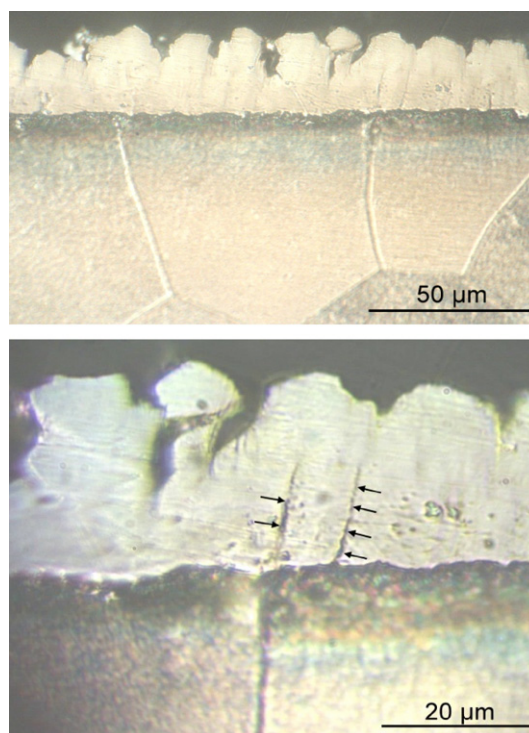

Fig. 6. Cross-sectional micrograph of electrodeposited Zn on WE43 from $ChCl/urea + 0.5\ M\ ZnCl_2$, with two magnifications. The micro-cracks are pointed out by arrows.

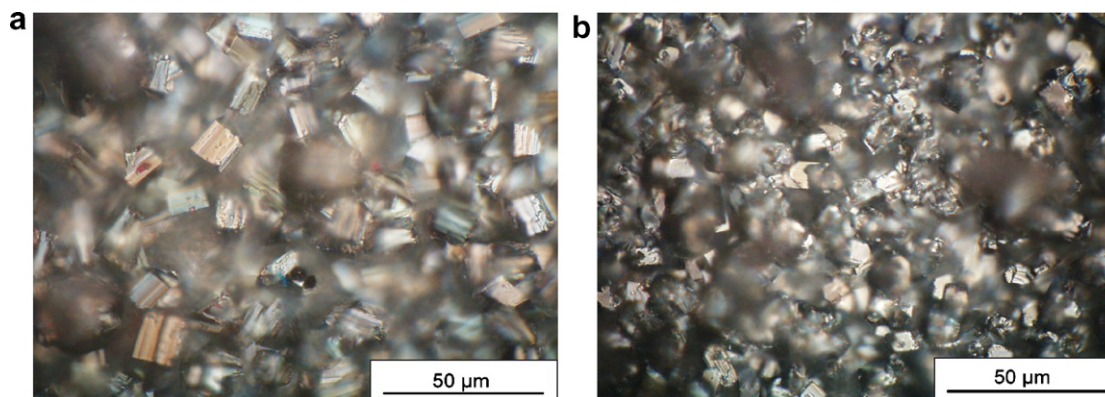Fig. 5. Optical micrograph of electrodeposited Zn onto QE 22-Mg alloys at $5\ mA/cm^2$, $T = 60$ °C: (a) from $ChCl/urea + 0.5\ M\ ZnCl_2$, (b) from $ChCl/urea + 0.5\ M\ ZnCl_2 + 10\ g/l\ EDTA$.
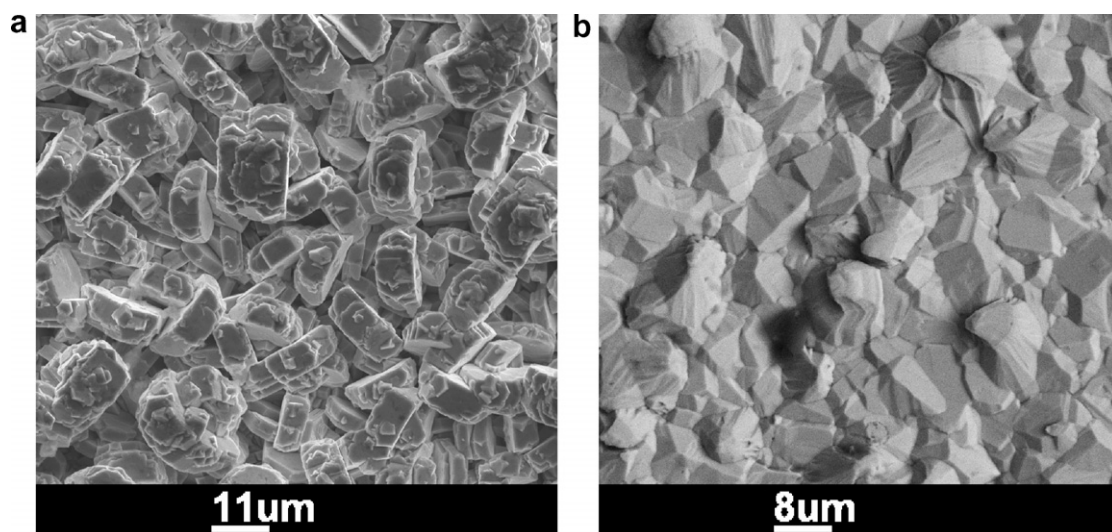
Fig. 7. SEM micrograph of electrodeposited Zn onto WE43 Mg alloy from ChCl/urea + 0.5 M ZnCl$_2$ at CD = 5 mA/cm^2, T = 60 °C (a) Constant current, with adding 10 g/l EDTA (b) Pulsed current (2 s on-time, 1 s off-time).
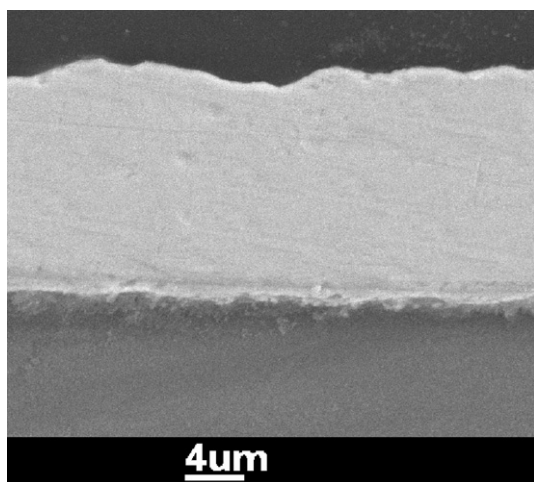

Fig. 8. SEM Cross-sectional micrograph of Zn coat electrodeposited by application of pulsed current (2 s on-time, 1 s off-time).

in 0.1 M NaCl aqueous solution, similar to that of the Mg substrate, as can be seen below. Therefore, the electrochemical polarisation test was to be used as indication of the presence of cracks in the deposited Zn layers.

3.4. Pulsed current vs. constant current

In order to eliminate the presence of micro-cracks, which render the Zn layers not corrosion resistant against immersion in aqueous chloride solutions, pulsed currents were to be applied in electrodeposition. The application of pulsed cathodic current was suggested to increase the nucleation rate of the electrodeposited atoms at the expense of the growth rate, thereby producing smoother deposits with a smaller grain size.

A cathodic current pulse between -5 mA/cm^2 and 0.0 mA/cm^2 was applied, and various pulse widths were used forming the loops:
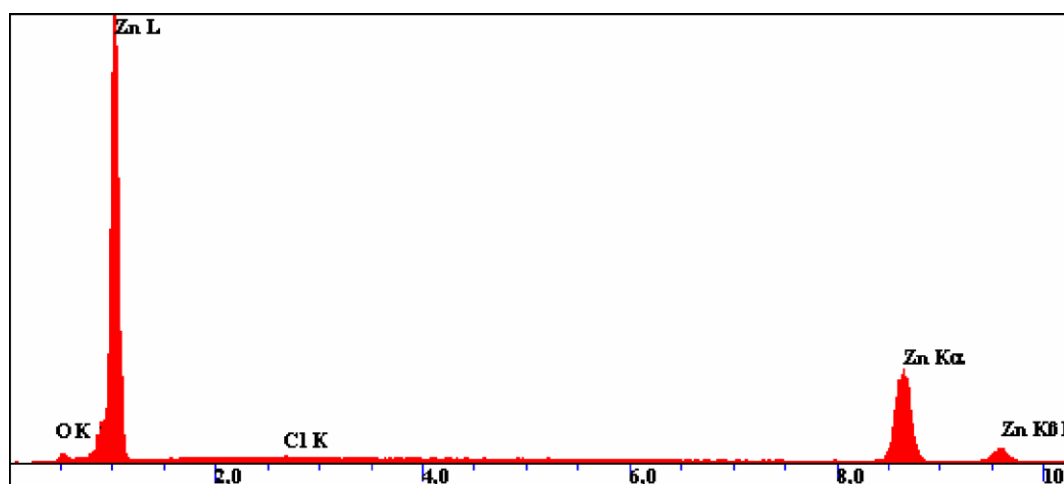

Fig. 9. EDX spectrum of electrodeposited Zn layer from ChCl/urea + 0.5 m ZnCl$_2$ at 5 mA/cm^2, T = 60 °C.
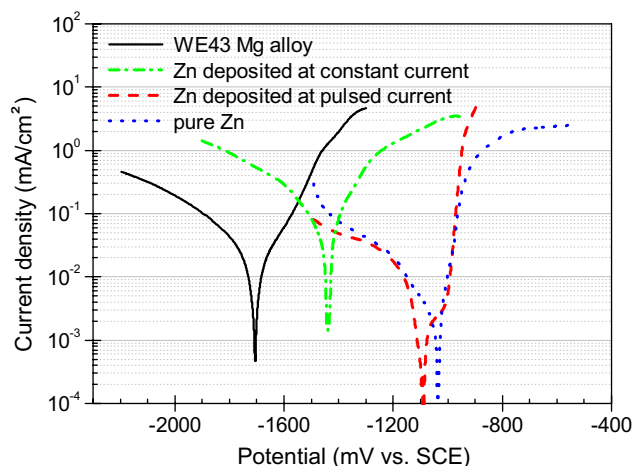
Fig. 10. Potentiodynamic polarisation diagrams of Mg alloy substrate, Zn deposits at constant and pulsed currents, and pure Zn sheet in 0.1 M NaCl aqueous solution.

Table 5
Corrosion characteristics, obtained by polarisation in 0.1 M NaCl solution, of Mg alloy substrates, Zn coats, and Zn standard sheet

Specimen	OCP (mV vs. SCE)	E_{corr} (mV vs. SCE)	I_{corr} ($\mu A/cm^2$)
WE43 Mg substrate	−1725	−1705	14.67
Zn on WE43 (IL without EDTA)	−1551	−1420	38.68
Zn on WE43 (IL + 20 g/l EDTA)	−1526	−1437	40.09
Zn on WE43 (IL previously dried)	−1515	−1402	30.7
Zn on WE43 (deposited by pulsed current)	−1129	−1093	1.38
Zn standard sheet	−1006	−1037	1.89
QE 22 Mg alloy substrate	−1543	−1489	6.69
Zn on QE 22 (IL + 10 g/l EDTA)	−1520	−1398	40.36

(i) 1 s on-time–0.5 s off-time (i.e. 1 s for–5 mA/cm^2 and 0.5 s for 0.0 mA/cm^2).
(ii) 1 s on-time–1 s off-time.
(iii) 2 s on-time–1 s off-time.

It was found that the best pulsing loop is 2 s on –1 s off to obtain an adequately uniform plating. In a comparison between Fig. 7a and b, it is clear that the morphology of the Zn coating electrodeposited by pulsed current is different. The pulsed current produced smoother and finer Zn grains, without pores. On the other hand, the microstructure of the deposit obtained by constant current can be imagined as piles of quasi parallelogram-shaped grains placed on top of each other, between which wide crevices are formed. The depth of these crevices reaches, occasionally, one half of the total thickness of the deposited layer, see the cross-section in Fig. 6. Also, the cross-sections of specimens electrodeposited by pulsed current showed uniform bulk Zn layers free of micro-cracks, as depicted in Fig. 8, on contrary to the cross-section revealed in Fig. 6.

The EDX spectrum originating from the deposits produced at either constant or pulsed current indicated that the coat layer is pure Zn. Fig. 9 depicts the typical EDX spectrum of the deposited layer revealed in Fig. 7b. The notably small oxygen peak is due to atmospheric oxidation of the Zn surface during exposure to air. The Cl content, whether it is co-deposited or not in the Zn layer, did not exceed 0.05 wt% for many of the deposits tested. Also, the EDX quantitative elemental analysis showed no evident difference in the composition regarding the type of applied current.

3.5. Corrosion testing

The corrosion behaviour of all successfully deposited Zn coats, both produced from liquids with and without additions of EDTA and at constant and pulsed currents, compared with that of a Mg alloy substrate and pure Zn sheet, were obtained by potentiodynamic polarisation in a 0.1 M NaCl solution (Fig. 10 and Table 5). The results illustrated that the Zn coats deposited at constant current densities have corrosion potential (E_{corr}) values close to those of their Mg alloy substrates. Moreover, these deposits have higher corrosion current densities (I_{corr}). This is attributed to the presence of very fine defects (micro-cracks) which permit the chloride solution to reach to the Mg substrate and decrease the corrosion resistance as a result of the galvanic corrosion arising at the interface between Mg and Zn. However, the Zn coats deposited at pulsed current densities exhibited a corrosion behaviour similar to that of the pure Zn sheet.

4. Conclusions

A detailed experimental study of the electrodeposition of Zn onto several Mg alloys in five air and water stable ionic liquids has been presented. These liquids are: 1 ChCl:2 urea, 1 ChCl:2 ethylene glycol, 1 ChCl:1 malonic acid, 1 ChCl:2 Glycerol, and 1 ChCl:2 ZnCl$_2$.

The electrochemical behaviour of Mg was studied in the five ionic liquids. The results showed that Mg has the lowest corrosion rate in the 1 ChCl:2 urea and 1 ChCl:2 Glycerol liquids. The latter, however, resulted in pitting on the Mg surface. Therefore, the ionic liquid 1 ChCl:2 urea was suggested to test the possibility of electroplating of several Mg alloys.

Successful electrodeposition of metallic Zn layers onto Mg-RE alloys free of Al was obtained in the ionic liquid 1 ChCl:2 urea. The other ionic liquids produced either powdery deposits or corrosion of the Mg substrate.

Application of pulsed cathodic current was found to be superior than constant current. The former produced uniform, shiny and dense deposits free of defects. Potentiodynamic polarisation in 0.1 M NaCl aqueous solution, which has been stated as a test for indication of the presence of defects in the deposited Zn layers, showed that the Zn coats deposited at pulsed current densities exhibit a corrosion behaviour similar to that of pure Zn.

Acknowledgements

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