



Discharge behavior and electrochemical properties of Mg–Al–Sn alloy anode for seawater activated battery

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Abstract: Mg–Al–Sn alloy is one of the new developed anode materials for seawater activated batteries. The potentiodynamic polarization, galvanostatic discharge and electrochemical impedance spectroscopy of Mg–6%Al–1%Sn and Mg–6%Al–5%Sn (mass fraction) alloys in seawater were studied and compared with the commercial AZ31 and AP65 alloys. The results show that the Mg–6%Al–1%Sn alloy obtains the most negative discharge potential of average -1.611 V with a electric current density of 100 mA/cm^2 . EIS studies reveal that the Mg–Al–Sn alloy/seawater interfacial electrochemical process is determined by an activation controlled reaction. The assembled prototype batteries with Mg–6%Al–1%Sn alloy as anodes and AgCl as cathodes exhibit a satisfactory integrated discharge properties.

Key words: magnesium anode material; galvanostatic discharge; anodic dissolution; seawater activated batteries

1 Introduction

Seawater activated batteries are important power sources for underwater instruments including military devices and commercial equipments [1]. One of the key materials applied in such batteries is the metal anode which increases the cell voltage and current capacity [2]. Magnesium alloy is one of the important anode materials used in seawater activated battery because magnesium offers a high electrode potential of -2.37 V vs normal hydrogen electrode (NHE), a high Faradic capacity and appropriate corrosion rate [3]. Previous investigations show that Mg–3%Al–1%Zn (AZ31) alloy is applied as a sacrificial anode but it hardly meets the requirements of seawater activated battery because of its bad electrochemical discharge properties [4]. The most serious disadvantage of such commercial AZ31 magnesium alloy is that it cannot provide a steady corrosion potential during the discharge process. Some special anode alloys, such as Mg–6%Al–5%Pb (AP65) and Mg–4%Ga–2%Hg, are developed to provide enough electrochemical activity to meet the requirements of seawater activated battery [5,6]. The AP65 alloy can provide high potential values during the discharged process as an anode of magnesium/silver chloride

(Mg/AgCl) battery with seawater electrolyte [7]. And the specific energy of the battery of Mg–Ga–Hg anode can exceed $100 \text{ W}\cdot\text{h/kg}$ so that it also can be one of the candidates for using as an anode [8,9]. But such two anode alloys contain the poisonous elements lead or mercury so that they are restricted within military applications and can hardly be used as commercial materials for battery. Therefore, in this work, the element tin is selected as a substitute for lead in Mg–Al–Pb alloy to study its effects on the electrochemical characterization of Mg–Al series alloys. As a result, a novel Mg–Al–Sn ternary alloy is developed to investigate its discharge behavior and evaluate its electrochemical properties as an anode for seawater activated battery. And a simple prototype battery is assembled with Mg–Al–Sn alloy sheet as anode and AgCl as cathode to evaluate its discharge properties in seawater.

2 Experimental

Two experimental alloys with the nominal compositions of Mg–6%Al–1%Sn and Mg–6%Al–5%Sn (mass fraction) were melted with pure magnesium ($>99.9\%$), pure aluminum ($>99.9\%$) and pure tin ($>99.99\%$) at 973 K with CO_2 and SF_6 gas protection.

Then, the melted alloy was cast in the water-cooled copper mold. The analyzed chemical compositions of the experimental alloy ingots were Mg–6.13%Al–1.23%Sn and Mg–6.20%Al–5.07%Sn. The as-cast two Mg–Al–Sn alloy ingots were homogenized at 673 K for 16 h and hot rolled many passes at 673 K to plates with 0.6–0.8 mm in thickness to produce the experimental specimens. The Solartron SI1287 potentiostat/galvanostat system was used in a three-electrode configuration for testing the half-cell characterization of the experimental alloys. The working electrode was the experimental alloy specimen. A saturated calomel electrode (SCE) served as the reference electrode, and the counter electrode was a platinum plate. The electrolyte was artificial sea water with temperature range of 289–291 K. The polarization, galvanometrical test and electrochemical impedance spectroscopy (EIS) of experimental alloy anodes were measured and the data were acquired and processed by the Zplot/Zview software version 2.6. Three samples of the all these alloys were tested for the electrochemical properties and the minimum values were reported. And the simple prototype batteries were assembled with ten pieces of Mg–Al–Sn experimental alloy sheets with the sizes of 160 mm×100 mm×0.4 mm as anodes and silver chloride (AgCl) plates as cathodes. The discharged properties of such prototype battery were measured in flowing artificial seawater with a temperature of (289±1) K. The microstructures of the Mg–Al–Sn alloys were observed by using a FEI-Quanta200 scanning electron microscope (SEM) and the compositions were analyzed with an energy-dispersive X-ray spectroscopy (EDX). The phase identification was investigated by using the DMAX-2500X X-ray diffractometer by using a Cu K α radiation with a wavelength of 1.5406 Å.

3 Results and discussion

3.1 Microstructure and phases in experimental Mg–Al–Sn alloys

The phases in experimental Mg–6%Al–1%Sn alloy includes α (Mg) matrix and a ternary $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ phases which can be identified by X-ray (Fig. 1(a)). Another binary Mg_2Sn phase forms in the Mg–6%Al–5%Sn alloy (Fig. 1(b)).

The morphology of these phases can be observed with the SEM and their compositions can be measured by the EDX (Fig. 2). The experimental alloys show dendritic structure grains with different morphology phases both at the grain boundary and in the matrix. According to Refs. [9,10], the morphology, size, and distribution of such phases influence the electrochemical characterization of magnesium alloy anode because of

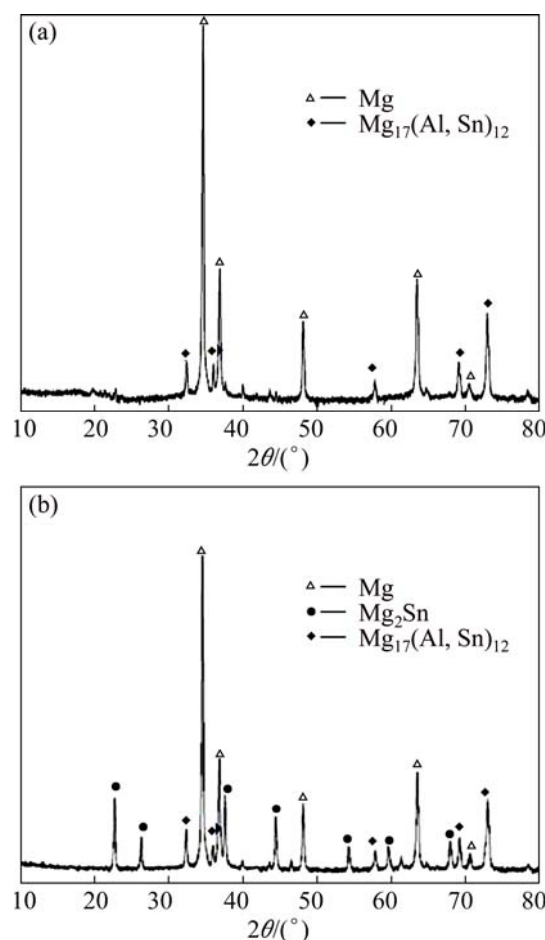


Fig. 1 XRD patterns of Mg–Al–Sn alloys: (a) Mg–6%Al–1%Sn alloy; (b) Mg–6%Al–5%Sn alloy

their cathodic qualities in the corrosion galvanic cell. Two distinct shape phases can be observed in the microstructure of experimental Mg–Al–Sn alloys. One is dispersed point shape at the grain boundaries, the other is large block with 30–40 μm in size in the α (Mg) matrix. With a composition analysis of EDX by using SEM (Figs. 2(b) and (d)) and the XRD phase identification (Fig. 1), it is found that the dispersed point shape phase is ternary $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ phase, and the block shape phase is Mg_2Sn phase. The maximum solid solubility of tin in α (Mg) matrix is 14.5% at 561 °C and decreases greatly to almost zero at room temperature. So, the $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ compound particles precipitate with the reduction of solid solubility of tin in α (Mg) matrix in Mg–6%Al–1%Sn alloy. With the increase of tin in the Mg–6%Al–5%Sn alloy, another Mg_2Sn phase can form during the solidification process with the reference of Mg–Al–Sn ternary phase diagram [11]. Obviously, the homogenous and refined distribution of $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ phase particles will exhibit different discharge behaviors with the coarse Mg_2Sn phase in the matrix during the electrochemical discharge process.

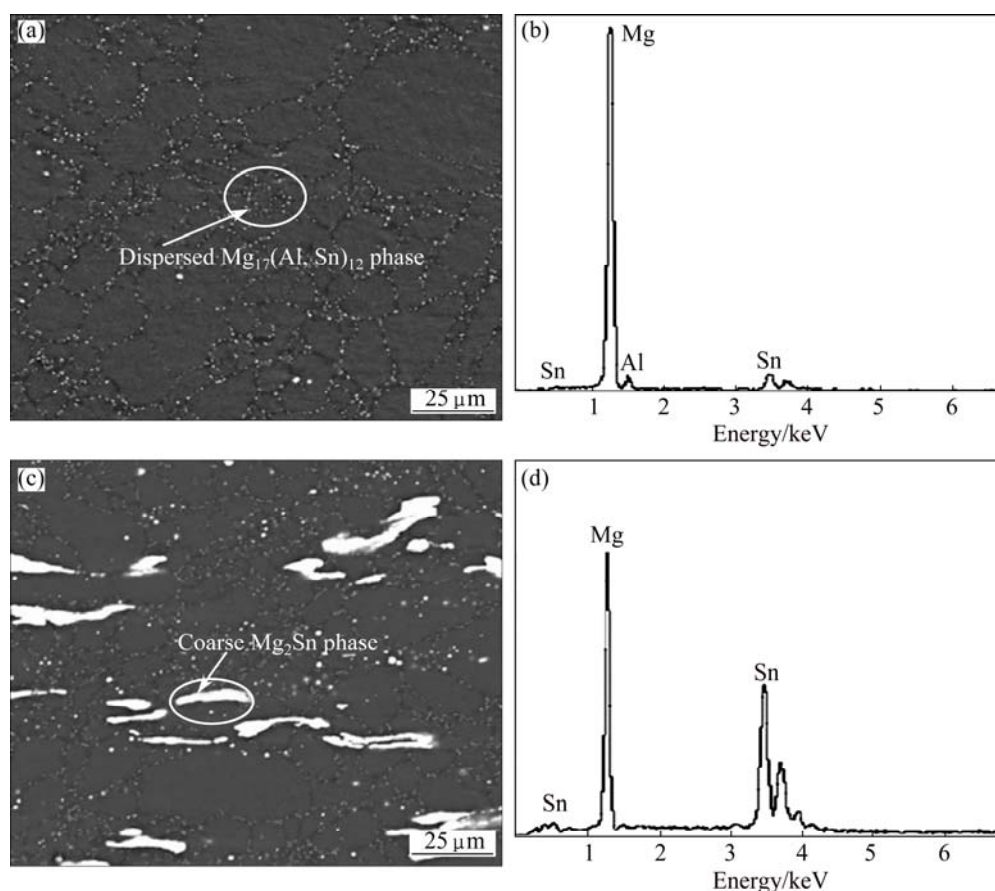


Fig. 2 Morphologies and composition of different phases in Mg–Al–Sn alloys: (a, b) Mg–6%Al–1%Sn alloy; (c, d) Mg–6%Al–5%Sn alloy

3.2 Discharge behavior of experimental Mg–Al–Sn alloys compared with AZ31 and AP65 alloys

The potentiodynamic polarization behaviors of two Mg–Al–Sn alloys compared with that of commercial AZ31 and AP65 alloys are shown in Fig. 3. The results show apparent variations in the corrosion potential and the corrosion current of different alloys. The corrosion potentials of experimental Mg–Al–Sn alloys are more negative than those of AZ31 and AP65 alloys. Compared with the commercial AP65 anode alloy, the corrosion potentials shift negatively by 0.05 V for Mg–6%Al–5%Sn alloy and 0.1 V for Mg–6%Al–1% alloy. Although the standard potential of the magnesium electrode is -2.37 V (vs NHE), the steady-state working potential is generally about -1.40 V (vs NHE) which is close to the value of AZ31 alloy. This deviation in potential is due to the formation of a magnesium hydroxide film which improves the corrosion resistance of magnesium on the metal surface [12]. As it is well known, the more negative corrosion potential with higher corrosion current is beneficial to the utilization of anode in the seawater activated battery. Therefore, the AP65 alloy is more suitable than the AZ31 alloy as a commercial anode material because it provides the

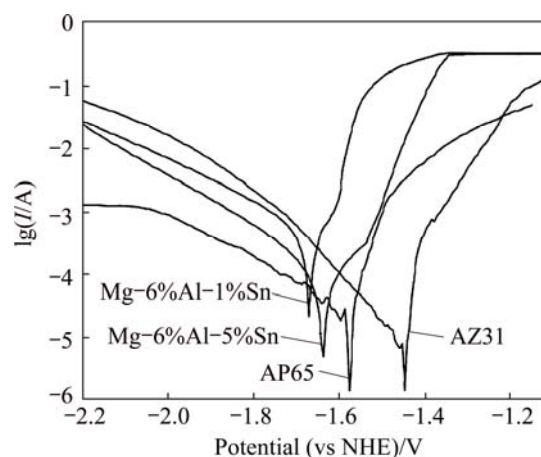


Fig. 3 Potentiodynamic polarization curves of experimental alloys

potential about -1.6 V. And the more negative corrosion potential of Mg–Al–Sn alloy with a value of about -1.7 V than that of AP65 shows such experimental alloys can be also treated as a suitable candidate anode for seawater activated battery.

In order to further compare the corrosion voltage variations of these alloys during the discharge process, the galvanostatic behavior of these alloys is measured

with a steady electric current densities of 100 mA/cm² and the results are shown in Fig. 4. The detail discharge voltage parameters are listed in Table 1. The high current density of 100 mA/cm² aims to evaluate the discharge behavior of power source anode material for long-term high-power applications [13]. The results show that the corrosion potentials of AZ31 alloy are more positive and unstable than those of AP65 and Mg–Al–Sn alloys. Such results confirm that AZ31 alloys can hardly meet the requirements of a seawater activated battery anode. The AP65 alloy can be used as battery anode material due to its comparatively negative corrosion potential value of average about –1.409 V with such high current density of 100 mA/cm² during the discharge process. The Mg–6%Al–5%Sn alloy obtains almost the same discharge potential values as AP65 alloy. And the Mg–6%Al–1%Sn alloy provides the most negative voltage values of all the alloys, especially it provides an average –1.611 V with the electric current density of 100 mA/cm². Such different discharge behaviors of these alloys are due to the different electrochemical reactions of Mg matrix with the second phases in the alloy. The reactions for magnesium matrix can be described as the following [14]:



Therefore, the anode will undergo hydrogen evolving corrosion:

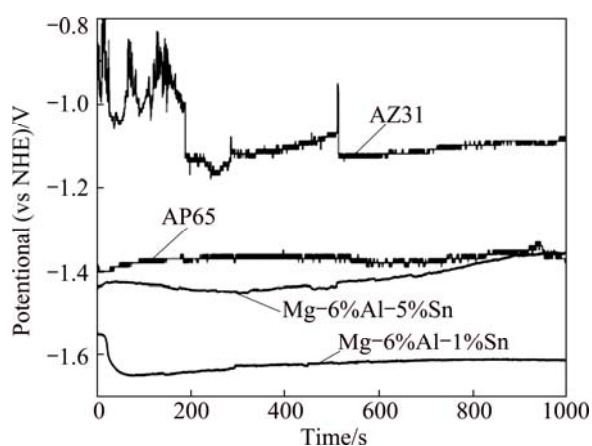
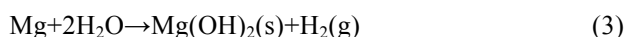


Fig. 4 Galvanostatic curves of experimental alloys with electric current densities of 100 mA/cm²

Table 1 Galvanostatic discharge parameters of experimental alloys

Alloy	Open circuit voltage/V	Average voltage/V
AZ31	–1.462	–1.151
AP65	–1.584	–1.409
Mg–6%Al–5%Sn	–1.643	–1.424
Mg–6%Al–1%Sn	–1.703	–1.611



The discharge reaction can be minimized by the corrosion product Mg(OH)₂ which pastes on the surface of alloy specimens and results in the reducing of anode reacting area [14,15].

With the observation of the corrosion surface of discharged Mg–6%Al–1%Sn (Fig. 5(a)) and Mg–6%Al–5%Sn alloy (Fig. 5(b)), it shows obviously that the corrosion starts at the grain boundary where the second phase Mg₁₇(Al,Sn)₁₂ distributes in Mg–6%Al–1%Sn. And the dispersed Mg₁₇(Al,Sn)₁₂ phases make the electrochemical reactions more uniform and let the corrosion penetrate into the interior of alloy through the corrosion crack at the grain boundaries. Such phenomenon is beneficial to the continuation corrosion of the Mg–6%Al–1%Sn anode alloy in the discharge reaction. So, the surface morphology of specimen becomes a rimous view as shown in Fig. 5(a). Such unprotected surface is beneficial to maintaining continuous corrosion reaction and provides stable discharge potential of the Mg–6%Al–1%Sn alloy anode.

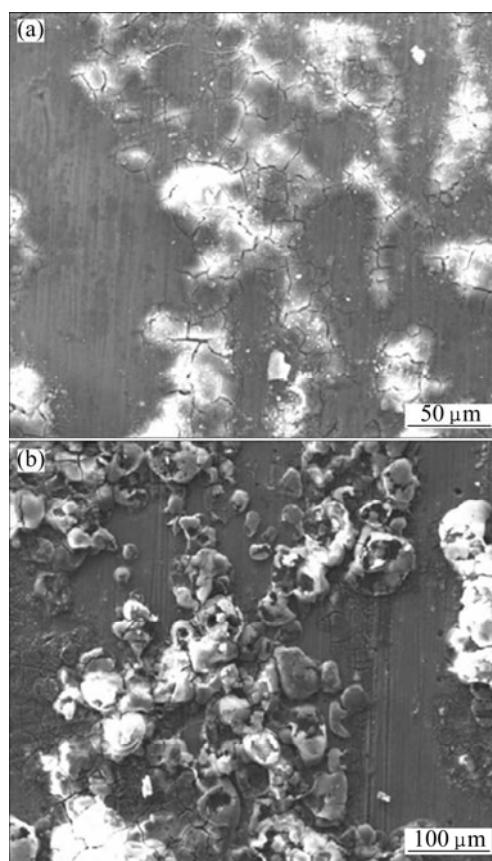


Fig. 5 Corrosion morphologies of Mg–Al–Sn alloys: (a) Mg–6%Al–1%Sn alloy; (b) Mg–6%Al–5%Sn alloy

The Mg₁₇(Al,Sn)₁₂ phases in Mg–6%Al–1%Sn alloy obtain a great electrode potential difference with the α(Mg) matrix. Such compound will accelerate the corrosion of α(Mg) matrix and induce the corrosion

penetrating into the internal of alloy. The dissolving of $\alpha(\text{Mg})$ matrix with $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ will produce Sn ions, which combine with Mg to produce amalgams that react severely with water and maintain the activation reaction circle. On the surface of Mg–6%Al–5%Sn alloy, such corrosion crack into the interior of matrix also can be observed but some corrosion product pastes on the surface of Mg–6%Al–5%Sn alloy and results in the reducing of anode reacting area. The Mg_2Sn phase obtains a coarse size and does not distribute as dispersed as the $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ phase, therefore, it makes less effect on the corrosion properties of Mg matrix as $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ does. And coarse Mg_2Sn phase causes the solid corrosion products to impede the corrosion penetrating into the internal of Mg matrix.

Generally, the electrochemical impedance spectroscopy (EIS) is used to study the discharge behavior and corrosion mechanism of magnesium alloy because the use of an AC signal can provide more information than that obtained from DC polarization technique [16]. In the present investigation, a simple electrochemical system consists of a double-layer capacitance (C_{dl}), a solution resistance (R_s) and a charge-transfer resistance (R_{ct}) [17,18]. The impedance locus diagrams of two experimental Mg–Al–Sn alloys, AP65 alloy and AZ31 alloy are obtained by applying a frequency range of 0.01–100 kHz with a AC amplitude of 10 mV. The Z-phase (θ) relation of the impedance is given by $Z' = Z \cos \theta$ for the real part and $Z'' = Z \sin \theta$ for the imaginary part. Typical Nyquist plots for the four magnesium alloys are shown in Fig. 6.

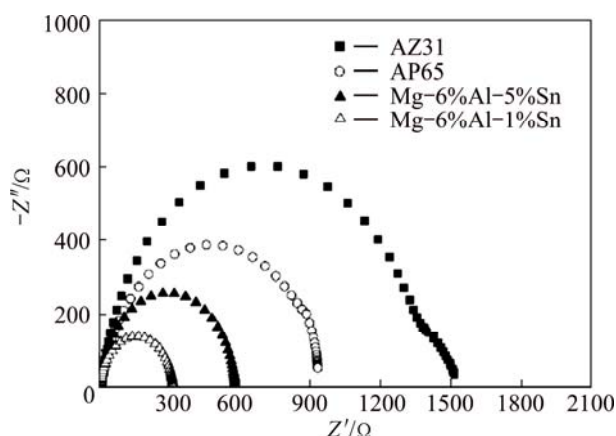


Fig. 6 Electrochemical impedance spectroscopy measurements of different magnesium alloys

The Nyquist plot shows that the curves of four alloys have a capacitive loop at all frequencies. The nature of the curves indicates that the system is activation-controlled because the semicircles have been obtained and this indicates that the electrode/electrolyte interface is controlled predominantly by activation-

controlled processes [17]. The diameter of the semicircle gives the charge-transfer resistance (R_{ct}) at the electrode/electrolyte interface which is related to the corrosion rate. The intercept on the x-axis at the higher frequencies gives the solution resistance (R_s). In order to enable a detail analysis of the impedance diagram, the equivalent circuit model is reported in Fig. 7 and the obtained parameters such as R_s , C_{dl} and R_{ct} are shown in Table 2. The EIS spectra of four alloys are similar except the diameter, which show that the corrosion mechanism is the same but the corrosion rate is different. The least diameter is obtained with Mg–6%Al–1%Sn alloy, indicating that the corrosion resistance of such alloy is the lowest. The corrosion rate is inversely related to R_{ct} . The higher the R_{ct} value, the lower the corrosion rate. And the order of the R_{ct} values with respect to four experimental alloys is: Mg–6%Al–1%Sn < Mg–6%Al–5%Sn < AP65 < AZ31. Such result reflects that the corrosion rate of Mg–6%Al–1%Sn alloy is the highest. The low C_{dl} value for the magnesium alloy implies the formation of relatively thick and compact protective film on the alloy surface. As expected, the Mg–6%Al–1%Sn alloy obtains the highest C_{dl} value and AZ31 alloy obtains the lowest value. In the present study, it is also seen that increasing R_{ct} is accompanied by the reduction of C_{dl} due to the totally different corrosion behaviors among these four alloys. In general, the intermetallics in the alloy make great effects on the electrochemical corrosion behavior. During corrosion process, the Mg–6%Al–1%Sn alloy cannot form effective protective film in chloride environment. The $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ phases in this alloy are activated easier and accelerate the corrosion of $\alpha(\text{Mg})$ matrix. Meanwhile, the dispersed and small size $\text{Mg}_{17}(\text{Al},\text{Sn})_{12}$ phases can adjust the corrosion current density and let the alloy exhibit an appropriate corrosion rate during the discharge process [19]. Such results match with the electrochemical measurements of polarization and galvanostatic discharge behaviors.

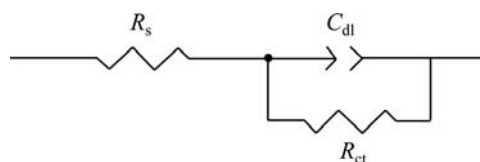


Fig. 7 Equivalent circuit of magnesium alloys in seawater

Table 2 Corrosion parameters obtained from EIS measurement of different alloys

Alloy	$R_s/(\Omega \cdot \text{cm}^2)$	$C_{dl}/(\text{F} \cdot \text{cm}^2)$	$R_{ct}/(\Omega \cdot \text{cm}^2)$
Mg–6%Al–1%Sn	6.51	1.25×10^{-3}	309
Mg–6%Al–5%Sn	7.12	1.38×10^{-3}	575
AP65	9.08	2.20×10^{-4}	910
AZ31	14.11	9.11×10^{-5}	1453

3.3 Discharge properties of a prototype battery assembled by Mg–Al–Sn alloy as anode

Two simple prototype batteries are assembled respectively with ten pieces of Mg–6%Al–1%Sn and Mg–6%Al–5%Sn alloy sheets with the sizes of 160 mm × 100 mm × 0.4 mm as anodes and silver chloride (AgCl) as cathodes (Fig. 8). And the electrolyte is artificial seawater at (289±1) K. The discharge properties of the prototype battery activated by seawater are shown in Table 3.

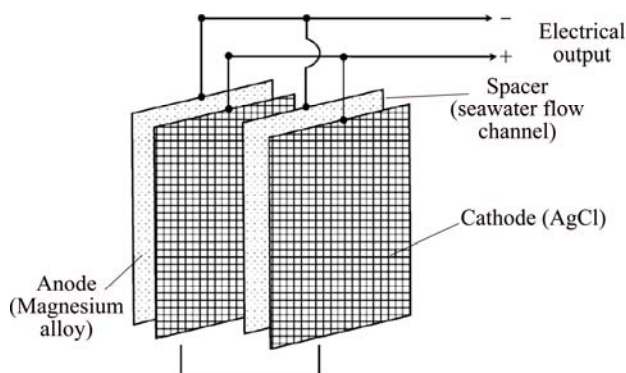


Fig. 8 Schematic diagram of prototype battery

Table 3 Typical discharge properties of prototype battery

Property	Mg–6%Al–1%Sn	Mg–6%Al–5%Sn
Activation time/s	7.7	8.1
Maximum voltage/V	1.530	1.488
Average voltage of entire discharge process/V	1.463	1.302
Hydrogen emission rate/(mL·cm ⁻² ·min ⁻¹)	0.32–0.47	0.23–0.39
Specific energy/(W·h·kg ⁻¹)	94.2	79

The activation time was measured as the time required by the battery to reach 70% of the average voltage. For both Mg–Al–Sn alloy anodes, the activation time was less than 10 s. The total discharge time of the battery persists 30 min. The average and maximum voltages of the cell with Mg–6%Al–1%Sn anode are higher than those of cell with Mg–6%Al–5%Sn anode. The hydrogen emission rate value is acceptable for the application in seawater activated battery. Especially, the specific energies of the simple prototype battery are 94.2 W·h/kg and 79.7 W·h/kg, respectively. Compared with the published data of 30 W·h/kg for lead acid battery or 88 W·h/kg for AP65/AgCl seawater activated battery [20,7], the discharge performance of Mg–6%Al–1%Sn alloy is also acceptable.

4 Conclusions

1) There are two typical secondary phases in Mg–Al–Sn alloys with different morphologies. One is dispersed Mg₁₇(Al,Sn)₁₂ phase in Mg–6%Al–1%Sn alloy, the other is coarse Mg₂Sn phase in Mg–6%Al–5%Sn alloy. Such two kinds of phases influence the discharge properties of alloy on different electrochemical tests.

2) The Mg–6%Al–1%Sn alloy is more negative than other experimental alloys, such as AZ31 and AP65 alloys, during the potentiodynamic polarization measurement. It provides an average –1.611 V with high electric current density of 100 mA/cm² during the galvanostatic discharge process.

3) The dispersed Mg₁₇(Al,Sn)₁₂ phases make the electrochemical reactions penetrate into the interior of alloy through the corrosion crack at the grain boundaries. And coarse Mg₂Sn phase causes the solid corrosion products to impede the corrosion penetrating into the Mg matrix. Hence, the Mg–6%Al–1%Sn alloy provides more negative discharge voltages than the Mg–6%Al–5%Sn alloy does during the electrochemical reaction.

4) EIS studies reveal that the electrode/electrolyte interfacial process is determined by an activation controlled reaction. The C_{dl} values which imply the formation of relatively protective film on the Mg alloy surface follow the following sequence: Mg–6%Al–1%Sn > Mg–6%Al–5%Sn > AP65 > AZ31. This result matches with the phenomenon that the corrosion rate of Mg–6%Al–1%Sn alloy is the highest.

5) The assembled prototype batteries with Mg–6%Al–1%Sn and Mg–6%Al–5%Sn alloys as anodes and AgCl as cathodes exhibit a satisfactory integrated discharge properties because of the discharge behavior and microstructures of the Mg–Al–Sn alloy. And the electrochemical parameters show that the Mg–6%Al–1%Sn alloy can be used as a candidate for seawater activated battery anode.

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海水激活电池阳极材料 Mg–Al–Sn 合金的 放电行为及电化学性能

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摘 要: Mg–Al–Sn 合金是海水激活电池阳极用的新型材料。针对 Mg–6%Al–1%Sn 和 Mg–6%Al–5%Sn(质量分数)两种合金, 进行动电位极化曲线实验、恒电流放电实验和交流阻抗实验等电化学实验研究, 并将研究结果与常规的 AZ31 和 AP65 镁合金进行对比。结果表明: Mg–6%Al–1%Sn 合金在 100 mA/cm² 恒电流放电实验中获得最低电位–1.611 V。在 Mg–Al–Sn 阳极材料和海水界面阳极的反应过程由活化反应控制。Mg–6%Al–1%Sn 阳极材料与 AgCl 阴极材料装配成原型电池后, 在电池放电过程中合金表现出满足使用要求的电化学性能。

关键词: 镁阳极材料; 恒电流放电; 阳极溶解; 海水激活电池

(Edited by Yun-bin HE)