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# Electrochemical noise characteristics in corrosion process of AZ91D magnesium alloy in neutral chloride solution

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Abstract: The corrosion process of AZ91D magnesium alloy in neutral 1% (mass fraction) sodium chloride aqueous solution was investigated by electrochemical noise(EN), SEM and EDX. Fractal theory was primarily used to depict the corrosion process of the alloy. The fast wavelet transform(FWT), as well as the fast Fourier transform(FFT), was employed to analyze the EN data. The results show that the overall corrosion process can be described by three stages. The first stage corresponds to the pit nucleation and growth; the second stage involves the growth of a passive oxide layer; and the third stage involves reactivation. With increasing immersion time, fractal dimension increases fast initially, fluctuates in the medium and increases again at last. Pitting corrosion and fractal dimension increase due to the initiation and formation of pits in the initial and the end of immersion, while depresses due to the passivation in the medium period. The results of SEM and EDX support the above conclusions.

Key words: AZ91D magnesium; corrosion; electrochemical noise; fractal dimension

# **1** Introduction

Magnesium-based alloys exhibit an attractive combination of low density and high specific strength associated with good castability and workability. Thus, they offer substitutes to ferrous alloys and aluminium in automotive and aeronautical applications[1]. However, the application of magnesium alloys has been limited due to the undesirable properties, including poor corrosion and wear resistance[2–10].

Among the magnesium alloys, AZ91D is one of those having the best mechanical properties/corrosion resistance balance on its corrosion behaviour in corrosive environments. Recently, the corrosive behavior of AZ91D magnesium alloy has been studied[11–23]. For example, MATHIEU et al[11], LI et al[12] and AMBAT et al[13] studied the influence of elements and the main constituent phase on corrosion of AZ91D magnesium

alloy. ANIK and CELIKTEN[14] studied the electrochemical behavior of AZ91D in phosphate/potassium hydroxide solution with the method of EIS. JÖNSSON et al[15-16], WINZER et al[17] and UNIGOVSKI et al[18] studied the atmospheric corrosion and stress corrosion cracking(SCC)of Mg-Al alloys, respectively. SONG et al[23] studied the corrosion behavior of AZ21, AZ501 and AZ91 magnesium alloys in 1 mol/L sodium chloride at pH=11. However, our knowledge was still insufficient and unsystematic for better understanding the corrosion behavior of AZ91D magnesium alloy.

Electrochemical noise(EN) has gained popularity in the recent years and has emerged as a promising technology for corrosion analysis. EN shows some advantages compared with the common techniques. First, EN is able to instantaneously monitor the rate of the corrosion process[24–25]; second, EN is carried out without artificial disturbance of the system[26]; and at

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last, EN can provide more information of localized corrosion than conventional techniques[27]. In the last decade, some researchers have used EN in corrosion study of magnesium alloy. LAFRONT et al[25] studied the pitting corrosion of AZ91D and AJ62x magnesium alloys in alkaline chloride medium using electrochemical noise. ZHANG et al[10] studied the electrochemical behavior of anodized Mg alloy AZ91D in chloride containing aqueous solution. ZHANG et al[28] analyzed the electrochemical noise of the corrosion of AZ91D magnesium alloy in alkaline chloride solution and deduced the corrosion process of the material according to the analysis. To date, there has been no detailed study about the EN utilized in magnesium alloy in neutral chloride medium. For understanding the corrosion progress of AZ91D magnesium alloy better, it is necessary to investigate the EN characteristics of the materials in neutral chloride medium.

 $D_{\rm f}$  (fractal dimension) is a quantitative parameter for analysis of fractal objects which is widely used for different purposes. In addition, it is one of the most important and useful parameters for analysis of the structure of rough surfaces and can provide additional valuable insight into the properties and dynamics of the studied system[29]. Fractals have been characterized by several methods that can be classified to be physical, chemical and electrochemical. Electrochemical methods supported by mathematical simulations are perhaps most useful and reliable for the determination of fractal dimensions of surfaces[30].

The aim of this work is to study the electrochemical noise characteristics of AZ91D magnesium alloy in neutral chloride solution and the time-dependent evolution of  $D_{\rm f}$  calculated by EN.

### 2 Experimental

The commercial Mg-based AZ91D alloys, of which the chemical compositions are listed in Table 1, were employed.

**Table 1** Chemical composition of AZ91D magnesium alloy(mass fraction, %)

Al	Zn	Mn	Ni	Cu
8.77	0.74	0.18	0.001	0.001
Са	Si	K	Fe	Mg
< 0.01	< 0.01	< 0.01	< 0.01	Bal.

Each electrode was embedded in epoxy with working surface exposed. In order to ensure the same surface roughness, each exposed surface was polished sequentially using abrasive papers from 400- to 1 000-grade and 2.5  $\mu$ m diamond paste, then rinsed

successively in acetone and distilled water, finally dried in air.

EN was monitored as a function of time between working electrode and reference electrode(SCE), using Powerlab/4sp (made in Australia) electrochemical interface through a GP amplifier controlled by Chart 5 software for Windows XP operating system. This instrument is equipped with analog/hardware filters including AA filter to remove high frequency components before the signal is digitized, and the fallacious components yielded from data acquiring can be avoided. The EN records were collected at 4 points/s, which means that the sampling interval,  $\Delta t$ , is 0.25 s, the low pass is 10 Hz and the high pass is DC. Tests were carried out in neutral 1.0% sodium chloride solution at ambient temperature and repeated at least three times for reproducibility.

SEM and EDX (SIRION, FEI Company, made in Holland) were utilized to examine the corrosion morphologies and composition of the corroded area of specimen surfaces.

All tests were conducted in a traditional three-electrode system (Fig.1). Counter electrode was platinum gauze and a saturated calomel electrode(SCE) was used as reference electrode separated by a salt bridge from the cell solution. The experimental temperature was controlled at  $(25\pm1)$  °C.



Fig.1 Schematic diagram of three-electrode glass cell

#### **3 Results and discussion**

#### 3.1 EN time records

The electrochemical noise of AZ91D magnesium alloy is shown in Fig.2. As shown in Fig.2(b), potential rises quickly at first (< 1 ks). Probably, the initial potential—time relationship should be connected with the soakage process of electrode surface and/or electrostatic effect that gives rise to the charge/discharge of interface electric double layer[10]. During the period of 1 to 2 ks, fast and small transients are seen on a background of slow event (the gradual change of the



**Fig.2** EN plots of AZ91D magnesium alloy in 1% sodium chloride solution: (a) Long-time immersion (1–15 represent random picked potential noise patterns); (b) Short-time immersion

baseline). During the period from 1 to 1.5 h, the similar feature of potential transients shown in Fig.2(b) is observed, but in a larger amplitude. According to Butler-Volmer equation, this phenomenon indicates that anodic dissolution takes place on AZ91D alloy surface. Meanwhile, such transients could be associated with the hydrogen evolution[28]. When the immersion time increases from 1.5 to 2 h, a characteristic pattern of abrupt drift with approximately exponential rise is observed and shown in Fig.2(b), which implies that pitting corrosion occurs on AZ91D alloy surface. With further increasing immersion time (corresponding pattern number 1-6), the similar transients are repeated many times, almost periodically. The fast transients reflect the competition process of metastable pitting and repassivation of AZ91D alloy. Between 28 to 56 h (corresponding pattern number 7-13), large transients appear consecutively in Fig.2(a) and subsequently potential falls down fast. In the end of immersion (from 64 to 72 h, corresponding pattern number 14-15), potential transients are similar to those in the initial immersion. According to the study of LAFRONT et al[25], there was a transient of "passivation to activation"

in corrosion progress of AZ91D alloy in alkaline chloride medium. In passive zone, the low current noise corresponds to a more noble potential, which is reversed in active zone. In this study, the electrolyte is neutral chloride medium without buffer. In long-time immersion, with magnesium dissolution and hydrogen evolution, pH value of the solution will increase, namely basification[31]. Basification should be propitious to the formation of passive film which can protect the base alloy. So the potential large transients in the period from 28 to 56 h may be related to the passivation due to basification. In the end of immersion, partial area of AZ91D alloy breaks off because of dissolution, which leads to the exposure of fresh base alloy to corrosive medium. Therefore, the electrode is activated and corroded badly, which presents the similar potential transients as those before 28 h.

#### 3.2 Power spectral density

Power density spectroscopy, which is calculated by the mean of fast Fourier transform(FFT), is one of the most frequently used methods to analyze EN. Power spectral density(PSD) plots provide information on the shape of the time record, which is supposed to be related to the corrosion mechanism[32–33]. The potential power spectral density is calculated as follows:

$$\lg D_{\rm v} = A_{\rm v} + S_{\rm v} \lg f \tag{1}$$

where  $A_v$  and  $S_v$  are the noise intensity and the roll-off slope of potential PSD plots, respectively. In this work, the original data were first subtracted by its Gaussian fit result (calculated by Origin 7.0 software) to remove the trend of the ensemble potential change, which were thought to be accorded with Gaussian distribution, and then all above parameters were calculated by the FFT software written by ourselves.

The PSD plots of AZ91D alloy during various immersion periods are illustrated in Fig.3, and the parameters are summarized in Table 2.



Fig.3 PSD plots of AZ91D magnesium alloy in 1% sodium chloride solution

Pattern No.	$W/(dB \cdot V^2 \cdot Hz^{-1})$	$k/(dB \cdot decade^{-1})$	$f_{\rm c}/{\rm Hz}$
1	-65.235 6	-16.139 8	5.260 6
2	-65.238 5	-13.106 9	7.699 8
3	-79.386 3	-13.030 8	51.397 1
4	-63.194 2	-14.485 5	6.857 7
5	-58.372 2	-14.219 5	5.940 8
6	-67.106 7	-15.901 7	33.841 3
7	-52.130 5	-15.989 9	1.292 1
8	-59.658 3	-16.840 6	2.337 4
9	-59.108 3	-15.392 8	2.540 2
10	-54.945 2	-15.463 3	5.280 6
11	-62.075 0	-14.783 4	11.098 9
12	-53.498 8	-16.754 2	2.415 1
13	-60.891 8	-18.754 2	3.053 5
14	-75.652 0	-13.091 1	19.212 9
15	-68.954 0	-14.905 7	6.189 4

 Table 2 PSD parameters of AZ91D magnesium alloy in 1%

 sodium chloride solution

Three important parameters can be obtained from PSD plots: 1) the slope of high frequency linear region (k), 2) the critical frequency or the cut-off frequency  $(f_c)$  and 3) the low-frequency plateau or white noise level (W). Generally, the values of k,  $f_c$  and W of potential SPD can be related to the severity of corrosion in some extent, namely, the more positive the values of k,  $f_c$  and W are, the more easily the pit initiates. For example, the slope of high frequency linear region(k) of PSD has been proposed as an indicator of the type of corrosion[34].

The more smoothly the curve changes, the more possibly the pitting corrosion occurs. As shown in Fig.3 and Table 2, the slopes of high-frequency linear part are greater than -20 dB/decade. In the period of 0-25 h, the values of k,  $f_c$  and W are mainly larger than those in the period of 28–56 h, which implies that pitting corrosion is more possible to occur and develop in this period. Moreover, the slopes change steeply in the period of 28–56 h, which means that the electrode surface might be in the state of passivation[34]. In the end of immersion time (64–72 h), the values of k,  $f_c$  and W increase again, which indicates that the more severe corrosion might happen.

The fractal dimension( $D_f$ ) of time sequence can take values between 1 for classical time sequence, to values less than 2 for abnormity of the time sequence. The larger the  $D_f$  is, the abnormity the electrode system will be. Namely, the alloy will be corroded more severely.

As shown in Fig.4, in the time scale of 1-6 (0-25 h),  $D_{\rm f}$  increases with the immersion time, which indicates that under the attack of aggressive ions pitting corrosion has occurred and developed/repassivated ceaselessly on

the electrode surface. And in the time scale of 7–13 (28–56 h),  $D_{\rm f}$  fluctuates to some extent due to the protection of the passive film and corrosion products. In the end of immersion,  $D_{\rm f}$  increases again, which means that the severe corrosion occurs on the local region of the surface.



**Fig.4** Fractal dimension of AZ91D magnesium alloy in different immersion time

As the above discussion, the results of PSD agree with those of EN record analysis very well. However, the information obtained only from PSD is insufficient for better understanding the corrosion process of AZ91D alloy during the immersion. The wavelet transform has been carried out and will be discussed later.

#### **3.3 SEM and EDS**

The corroded morphologies of AZ91D magnesium alloy in 1% sodium chloride solution for different immersion time are shown in Fig.5. It can be obviously seen that in the initial time, there is only one pit on the surface of the electrode, and the pit develops into the base gradually (Figs.5(a)-(c)). With the time increasing, the number of pits increases and pits connect with each other (Figs.5(d)-(f)), which reflects the propagation/ development process of the pitting corrosion. When immersion time increases to about 48 h, the number of pits increases seldom and the corrosive intensity is depressed to some extent. In the end of immersion, severer corroded morphology can be seen in Fig.5(h). From EDS spectra in Fig.6(b), it can be seen that a strong peak of oxygen is detected in the corroded region of alloy in 48 h (labeled with the arrow in Fig.5(g)), which implies that a passive film constituted of magnesium oxide may exist in the corroded region. The protection of this film baffles the process of the pitting corrosion to some extent. However, as shown in Figs.6(a) and (c), there are a strong peak of magnesium and a weak peak of aluminium without oxygen peak detected in the corroded region (labeled with the arrow in the Figs.5(f) and (h)),



**Fig.5** SEM images of AZ91D magnesium alloy in 1% sodium chloride solution for different immersion time: (a) 1 h; (b) 3 h; (c) 5 h; (d) 8 h; (e) 15 h; (f) 23 h; (g) 48 h; (h) 72 h



**Fig.6** EDS spectra of corrosion areas labeled with arrows in Fig.5: (a) 23 h; (b) 48 h; (c) 72 h

which indicates that the main corroded region is  $\alpha$  phase (magnesium base) and without passive film  $\alpha$  phase corrodes badly. The results of SEM and EDS prove strongly the conclusions obtained from the analysis of the EN records.

#### 3.4 Wavelet analysis

According to the fast wavelet transform(FWT) analysis algorithm, a time record  $S_n(t)$  ( $n=1, 2, \dots, n$ )  $\in L^2(\mathbf{R})$  can be represent by linear combinations of basis

 $\phi_{J,k}$ ,  $\varphi_{J,k}$  and amplitude coefficients of each function  $S_{J,k}$ ,  $D_{J,k}$ ,  $\cdots$ ,  $D_{l,k}$ .

$$S(t) \approx \sum_{k} S_{J,k} \phi_{J,k}(t) + \sum_{k} D_{J,k} \varphi_{J,k}(t) + \sum_{k} D_{J-1,k} \varphi_{J-1,k}(t) + \dots + \sum_{k} D_{1,k} \varphi_{1,k}(t)$$
(2)

$$S_{J,k} = \int S(t)\phi_{J,k}^{*}(t)dt$$
(3)

$$D_{l,k} = \int S(t)\varphi_{l,k}^*(t)\mathrm{d}t \tag{4}$$

where  $\phi_{J,k}^*$  and  $\varphi_{l,k}^*$  are the complex conjugates of the basis functions father wavelet  $\phi(t)$  and mother wavelet  $\varphi(t)$ , which are the bases for this decomposition by translating in time and dilating in scale.

$$\phi_{J,k}(t) = 2^{-J/2} \phi(2^{-J}t - k) = 2^{-J/2} \phi(\frac{t - 2^J k}{2^J})$$
(5)

$$\varphi_{l,k}(t) = 2^{-l/2} \varphi(2^{-l}t - k) = 2^{-l/2} \varphi(\frac{t - 2^{l}k}{2^{l}}), \quad J, l, k \in \mathbb{Z}$$
(6)

where  $k=1, 2, \dots, N/2$ , N is the number of data record.  $l=1, 2, \dots, J, J$  is often a small natural number.  $2^{l}$  acts as scale factor and  $2^{l}k$  as translation parameter. Every coefficient  $S_{l}$  or  $D_{l}$  is called a crystal, and is related to the features of the original signal of a particular time-scale that can be computed roughly[35].

In order to characterize the FWT results in more details, the contribution of each crystal to the overall signal,  $E_l$ , named as a relative energy of a crystal, is plotted with the crystal name:

$$E_{J}^{S} = \frac{\sum_{k=1}^{N/2^{J}} S_{J,k}^{2}}{\sum_{n=1}^{N} S_{n}^{2}}, E_{J}^{D} = \frac{\sum_{k=1}^{N/2^{J}} D_{l,k}^{2}}{\sum_{n=1}^{N} S_{n}^{2}}, l=1, 2, \dots, J$$
(7)

Fig.7 shows the evolution features of  $E_l$  versus time in 1% sodium chloride solution. Each  $E_l$  is calculated from a range of EN records according to Fig.2(a), which contains 1 024 points and registered from different exposure time (the selected batches are the first 1 024 (n = 1 024) points of the second quarter in each hour of tests). Generally, it is widely accepted that the initiation, growth and death of metastable pitting always occur prior to the other types of localized corrosion, and are much faster than processes such as diffusion of aggressive ions, removal of corrosion products and bubble evolution[36]. There are fast processes corresponding to smaller and larger time-scales. The time-scale of crystal D can be ranked in an increasing series:  $D_1 < D_2 < \cdots < D_7 < D_8$ .

As shown in Fig.7, the whole corrosion process of alloy in neutral chloride solution can be divided into



**Fig.7** Time evolution of potential  $E_l$  plots for AZ91D magnesium alloy in 1% sodium chloride solution

three stages.

In the initial stage, from 0 to 25 h, the energy mainly accumulates in  $D_2$  crystal, which may correspond to the fast localized corrosion process such as pitting, and  $D_7$ – $D_8$ , which may correspond to the slow diffusion process of mass or charge around the active corrosion points due to corrosion products.

In the second stage from 28 to 56 h, the energy accumulates gradually into the medium time-scale and finally occupies predominantly the coarse crystals  $(D_6-D_7)$  due to the formation of a passive film that covers on the electrode surface. This indicates that the whole corrosion process is mainly controlled by the diffusion process. Because of basification, the formation of the passive film makes the transfer of mass of charge more difficult, which baffles the propagation and development of pits to some extent (Fig.5(g)). As a result, the energy is mainly accumulated in the coarse crystals  $(D_6-D_7)$ .

In the last stage from 64 to 72 h, the energy contribution mainly accumulates in small time-scale  $(D_1-D_2)$ . In this stage, the passive film is damaged by attack of chloride ions and the corrosion products are saturated gradually by solution and become expanding. This means the protection of passive film, as well as corrosion products, to magnesium alloy is weakened a lot. Meanwhile, with the dissolution and departure of the products from the base, fresh region of electrode with more vigor is exposed to corrosive medium, which will speed up the pitting corrosion. Consequently, it is understandable that the energy is mainly accumulated in the coarse crystals  $(D_1-D_2)$ .

#### **4** Conclusions

1) The time dependent corrosion potential obtained in the experiments, as well as power spectral density and contribution of each crystal to overall signal, shows that the overall corrosion processes can be described by three stages. The first stage corresponds to the pit nucleation and growth; the second stage involves the growth of a passive oxide layer; and the third stage involves the reactivation.

2) With increasing immersion time, the fractal dimension increases fast initially, fluctuates in the medium and increases again at last. This indicates that in the initial and end of immersion, the pitting corrosion and fractal dimension are increased due to pitting, but depressed due to the passivation in the medium immersion.

3) The results of SEM and EDS show that the alloy is mainly suffered from pitting corrosion in neutral chloride solution, and the corrosion is buffered to some extent in the medium immersion time due to the formation of passive film.

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