



Influence of processing factors on properties of anodic coatings obtained on Mg–1.0Ca alloy



S.B. Guo^a, Y.Q. Zhang^a, R.F. Zhang^{b,c,*}, S.F. Zhang^{b,c}, H.H. Zhang^{b,c}

^aHunan University of Science and Technology, Xiangtan 411201, PR China

^bJiangxi Key Laboratory of Surface Engineering, Jiangxi Science and Technology Normal University, Nanchang 330013, Jiangxi Province, PR China

^cSchool of Material Science and Engineering, Jiangxi Science and Technology Normal University, Nanchang 330013, PR China

HIGHLIGHTS

- NaOH can result in the development of thin coatings with large microcracks.
- Increasing sodium phytate concentration improves the coating corrosion resistance.
- Treatment time has a minor influence on the coating composition.
- Duty cycle is the least important factor on coating thickness.

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ABSTRACT

The influences of four factors including NaOH concentration, sodium phytate concentration, treatment time and duty cycle on properties of anodic coatings formed on Mg–1.0Ca alloy were systematically investigated using the Taguchi experiment. The effect order of factors on the corrosion resistance is ranked as sodium phytate concentration > treatment time > duty cycle > NaOH concentration, while the sequence on the coating thickness is sodium phytate concentration > treatment time > NaOH concentration > duty cycle. The increasing sodium phytate concentration is benefit for the corrosion resistance of the anodized magnesium alloys by developing thick coating with stable composition.

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1. Introduction

Magnesium alloys have been one of the hot topics in the field of degradable biomaterials due to their many advantages, such as low elastic modulus, similar density to natural bone and in vivo degradation via corrosion obviating the requirement of a second surgery for implant removal [1–3]. However, the too rapid degradation rate of magnesium alloys in body fluids restricts their clinical applications. Therefore, alloying [3–5] and various surface treatments [1–3,6–24] are applied to improve their corrosion resistance. Calcium is a major component in human bone and is beneficial to the bone healing. As an alloying element, calcium can

improve the corrosion resistance [3,4], mechanical property [3] and biocompatibility [5]. However, the in vivo corrosion rate of Mg–Ca alloy is still too high to prevent local gas accumulation and match the bone healing process [5]. Therefore, surface treatments, which can improve the biocompatibility of magnesium alloys by reducing the degradation rate and inducing better bone–implant interfaces [6], are widely used [6–24].

Micro arc oxidation (MAO) is an effective treatment technology on magnesium alloys and can improve the corrosion resistance of the anodized magnesium alloys [7–22]. In addition, the porous structure of anodic coatings developed on magnesium alloys by MAO is helpful to cell adhesion [7]. The coating properties depend on several factors, such as the composition of the substrate [8], electric parameters [7,9–14] and concentrations and compositions of the electrolytes [9,13–15,17–22]. Among these influencing factors, the used electrolytes play a main role in determining the coating property. Because phosphorus is an essential mineral for all living organisms, phosphates are usually used as the main

* Corresponding author. Box 124, Jiangxi Key Laboratory of Surface Engineering, Jiangxi Science and Technology Normal University, Nanchang 330013, Jiangxi Province, PR China. Tel./fax: +86 791 83831266.

E-mail addresses: rfzhang-10@163.com, zhyq8767@126.com (R.F. Zhang).

electrolytes of MAO [8,13–19]. However, the obtained coatings in a solution with inorganic phosphates are not ideal [15]. Therefore, to meet the demand of magnesium alloys used for biomaterials, selecting a high efficiency and environmentally friendly organic phosphates is necessary.

Recently, phytic acid ($C_6H_{18}O_{24}P_6$), which originates in most cereal grains, legumes, nuts, oilseeds and has a variety of benefits on human health including anti-cancer agent, inhibitor for renal stone development and anti-oxidation agent [25], was used as a main electrolyte in MAO on magnesium alloys [20,21]. The coatings obtained in a solution containing phytic acid achieve better corrosion resistance than those in a solution containing silicate [20]. Sodium phytate ($Na_{12}Phy$), a salt derivative of phytic acid, has some more advantageous properties than phytic acid, such as convenient to transportation and storage. In addition, phytic acid was not given the status of “generally recognized as safe (GRAS)” by the Food and Drug Administration of the United States in its 1995 list [25], while sodium phytate was listed as a GRAS substance by 1997 and has been used as a preservative for baked goods in the U.S. [25]. However, to the best of the authors’ knowledge, there are no reports about the applications of sodium phytate in MAO on magnesium alloys.

In order to achieve the optimum processing conditions under which the anodized sample achieves the best corrosion resistance, a large number of experiments will be carried out by the conventional approach, namely, one factor is varied while at the same time, all the other factors are kept constant. The Taguchi method can minimize the number of experiments to a practical level for optimization purposes through the use of a statistical design of experiments at a relatively low cost [9]. In this paper, the Taguchi method was used to investigate the effect order of factors on the thickness and corrosion resistance of anodic coatings. In addition, field emission scanning electron microscope (FE-SEM), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) were used to reveal the influencing mechanism of processing factors on the coating corrosion resistance.

2. Experimental

An ingot of Mg–1.0Ca alloy with nominal 1 wt.% calcium and balance Mg was used as the substrate. Samples for MAO treatment were masked with silicone sealant leaving an area of 50 mm × 60 mm × 10 mm exposed, with a 3 mm diameter hole drilled for connection with the anode by using a screw. High quality sodium phytate (purity ≥ 98%) and analytical reagent grade NaOH were used in the experiment. The pH values of solutions were measured by PHS-3C pH Meter (Yoke Instrument Co. Ltd, Shanghai).

Samples were anodized after they were ground successively on SiC paper up to 1200 grit, degreased by acetone, washed with distilled water and dried in a cool air stream. The equipment for MAO consisted of a MAOI-50C power supply (Chengdu Pulsetech Electrical Co., Ltd, China), a stainless steel barrel and a stirring and cooling system that controlled the solution temperature below 40 °C. The power supply can provide a unipolar (positive) pulse or bipolar pulses (with a positive pulse and a negative pulse). The frequency and duty cycle of the power supply can be adjusted from 100 to 3000 Hz, 5%–40%, respectively. In the experiment, the unipolar pulse was used under a constant current control mode. According to the obtained results as before, current density and frequency have minor influences on the coating corrosion resistance [12]. Therefore, in the experiment, current density and frequency were separately fixed as 40 mA cm⁻² and 2000 Hz.

Surface and cross-sectional morphologies of the anodized samples were observed using a ΣIGMA FE-SEM after they were rinsed with distilled water, dried in a cool air stream and coated with gold. The coating compositions were determined by EDS attached to FE-

SEM. After anodic coatings were scrapped off from magnesium alloys with a razor, they were analysed using a D8 ADVANCE X-ray diffractometer with Cu K α radiation and a Nicolet 460 FT-IR spectrometer in the range of 400–4000 cm⁻¹. In comparison, the original $Na_{12}Phy$ powder was also analysed by KBr pellet technique. The corrosion resistance of the anodized samples in 3.5% NaCl sodium chloride solution at 37 °C was carried out using a hydrogen evolution method [23]. Three duplicates were measured.

3. Results

3.1. The arrangement of the Taguchi experiment and result analysis

The Taguchi experiment of four factors with three levels including NaOH concentration, sodium phytate concentration, treatment time and duty cycle was used to comprehensively investigate the effects of processing factors on the coating thickness and corrosion resistance. The factors and levels of the Taguchi experiment are listed in Table 1.

The Taguchi experimental array and the pH values of nine solutions are listed in Table 2. The cross-sectional morphologies of anodic coatings obtained in nine solutions above are shown in Fig. 1. The coating thickness was achieved by Fig. 1 and the results are listed in Table 2.

As shown in Table 2, the pH values of nine solutions are from 12.94 to 13.23. In these strong alkaline solutions, NaOH can slightly increase the pH values of the solutions, while sodium phytate has a minor influence on them. In the experiment, the volume of hydrogen evolved after a 24 h immersion in 3.5% NaCl sodium chloride solution at 37 °C was used as the evaluation standard of the corrosion resistance. It is clear that among all the anodized samples, the coating on No. 3 poses the best corrosion resistance (listed in Table 2). In order to systematically investigate the effect of processing factors on the coating corrosion resistance, the experimental data were treated by the method of intuitionistic analysis, which was synthesized by calculating the total volume of hydrogen evolved on all anodized samples at each level for various factors. The maximum difference between the total volume of hydrogen at two levels for each factor indicated the general effect of that factor. Based on the value of the differences, the rank of influencing factors on corrosion resistance of the anodized samples was sodium phytate concentration > treatment time > duty cycle > NaOH concentration. The optimum processing conditions for the corrosion resistance are NaOH 3 g L⁻¹, sodium phytate 15 g L⁻¹, treatment time 210 s and duty cycle 35%.

Table 2 also shows that among all the anodized samples, the coating on No. 3 is the thickest. The data about the coating thickness (listed in brackets) were dealt with the same method as corrosion resistance and the order of influencing coating thickness was sodium phytate concentration > treatment time > NaOH concentration > duty cycle.

Fig. 2 shows the effects of the four factors on the thickness and corrosion resistance of anodic coatings plotted based on the results in Table 2.

Table 1
Factors and levels of the Taguchi experiment.

Levels	Factors			
	NaOH concentration (g L ⁻¹)	Sodium phytate concentration (g L ⁻¹)	Treatment time (s)	Duty cycle (%)
1	3	5	150	15
2	6	10	180	25
3	9	15	210	35

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