



Fabrication and corrosion property of conversion films on magnesium alloy from deep eutectic solvent

Jialei Zhang^{a,b}, Changdong Gu^{a,b,*}, Wei Yan^{a,b}, Jiangping Tu^{a,b}, Xiangdong Ding^{c,**}

^a School of Materials Science and Engineering, State Key Laboratory of Silicon Materials, Zhejiang University, Hangzhou 310027, People's Republic of China

^b Key Laboratory of Advanced Materials and Applications for Batteries of Zhejiang Province, Hangzhou 310027, People's Republic of China

^c State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, People's Republic of China

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ABSTRACT

A facile anodic treatment for magnesium alloy was proposed in a choline chloride-ethylene glycol based deep eutectic solvent (DES). Conversion films with interconnected porous networks and jagged nanorod arrays can be formed, which depends on the applied anodic current density. Crystallographic orientation of the substrate is accordingly changed. The conversion films are mainly composed of MgCO₃ which results from the interaction between the Mg alloy and the released species from the decomposition of DES. A higher anodic current density produces a better corrosion resistant conversion film. Superhydrophobic and slippery surfaces can be achieved on the as-prepared conversion films to further improve the corrosion resistance.

1. Introduction

Magnesium and its alloys have been extensively used in the fields of automotive, aerospace, electronics and biomedicine owing to their attractive properties including high specific strength, considerable ductility, improved machinability, good castability, excellent biocompatibility and available recyclability [1–4]. However, a fatal flaw of Mg alloys is the poor corrosion resistance caused by the inherently high activity of Mg. Natural corrosion products such as oxides, hydroxides and carbonates on Mg alloys cannot provide a uniform and large-area passive film [5]. Artificial conversion coatings seem to be one of the effective ways to enhance the corrosion resistance [6–8]. In recent years, various conversion coatings on Mg alloys have been developed with advantages of facile procedure, tunable texture and high adhesion to substrate, e.g. phytic acid [9], chromate [10], phosphate [11–13], stannate [14,15], vanadate [16,17], permanganate [18], fluoride [2], and hydrotalcite coatings [19–21]. However, most of these coatings or films are synthesized from conventional aqueous reaction media, which results in hydrogen evolution and uncontrollable preparation [22]. In comparison with the widely studied water-based system, non-aqueous solvents show prospective alternatives for preparing superior coatings or films on Mg alloys [23].

Ionic liquids are room-temperature molten salts composed entirely of cations and anions, which are regarded as a new type of solvent except aqueous and organic solvents. Active metals such as magnesium

and lithium remain stable for a long time without being corroded in specific ionic liquids owing to no free H⁺ or other metal cations, which provides favorable conditions for the control of film formation on active metals [24–29]. M. Forsyth's group proposed a surface treatment of the AZ31 Mg alloy in biocompatible phosphate-based ionic liquids to mitigate its degradation in human body [24]. They evaluated the cytotoxicity and corrosion resistance of the ionic liquid films and found the corrosion resistance was depended on the treatment time. When a potential bias of –200 mV was applied on the ZE41 Mg alloy during exposure to the ionic liquid of trihexyl(tetradecyl)phosphonium diphenylphosphate, a more uniform ionic liquid film would be formed compared with the one obtained at open circuit potential [29].

Choline chloride (ChCl) based deep eutectic solvents (DESs) are a new class of ionic liquids or ionic liquid analogues sharing most of their remarkable qualities while possessing other advantages such as easy synthesis, low cost facile application and so on [30]. They have been applied for coating electrodeposition, surface electropolishing, nano-material synthesis, and biotransformation [31,32]. Our group has reported that the ChCl-urea based DES would decompose under thermal conditions to generate ammonia/amide and carbonate in the presence of a little water [33]. Upon heating, the ChCl-urea mixture could react with the AZ31B Mg alloy to form a promising anti-corrosion conversion film [23]. It was also demonstrated that the trivalent chromium conversion coating with self-healing effect could be fabricated by utilizing the interaction between the ChCl-ethylene glycol-CrCl₃ solution and the

* Correspondence to: C. Gu, School of Materials Science and Engineering, State Key Laboratory of Silicon Materials, Zhejiang University, Hangzhou 310027, People's Republic of China.

** Corresponding author.

E-mail addresses: cdgu@zju.edu.cn (C. Gu), dingxd@mail.xjtu.edu.cn (X. Ding).

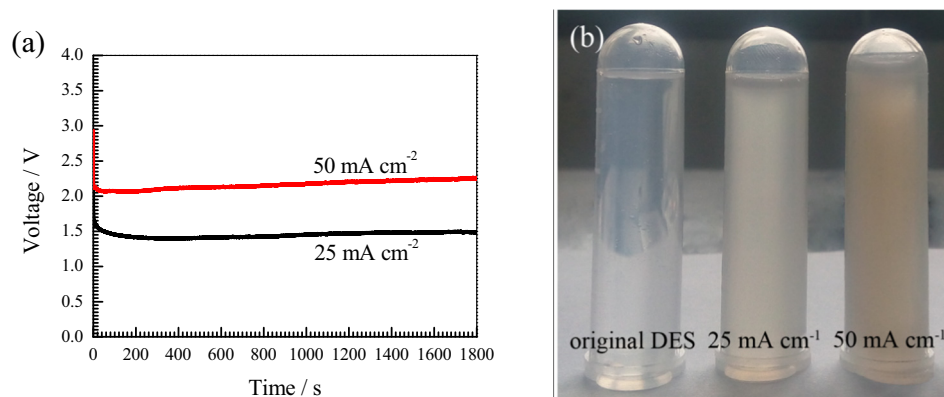


Fig. 1. (a) Chronopotentiometric curves at different current densities during the anodic treatments. (b) Visual inspections of the original DES and the electrolytes after anodic treatments at different current densities.

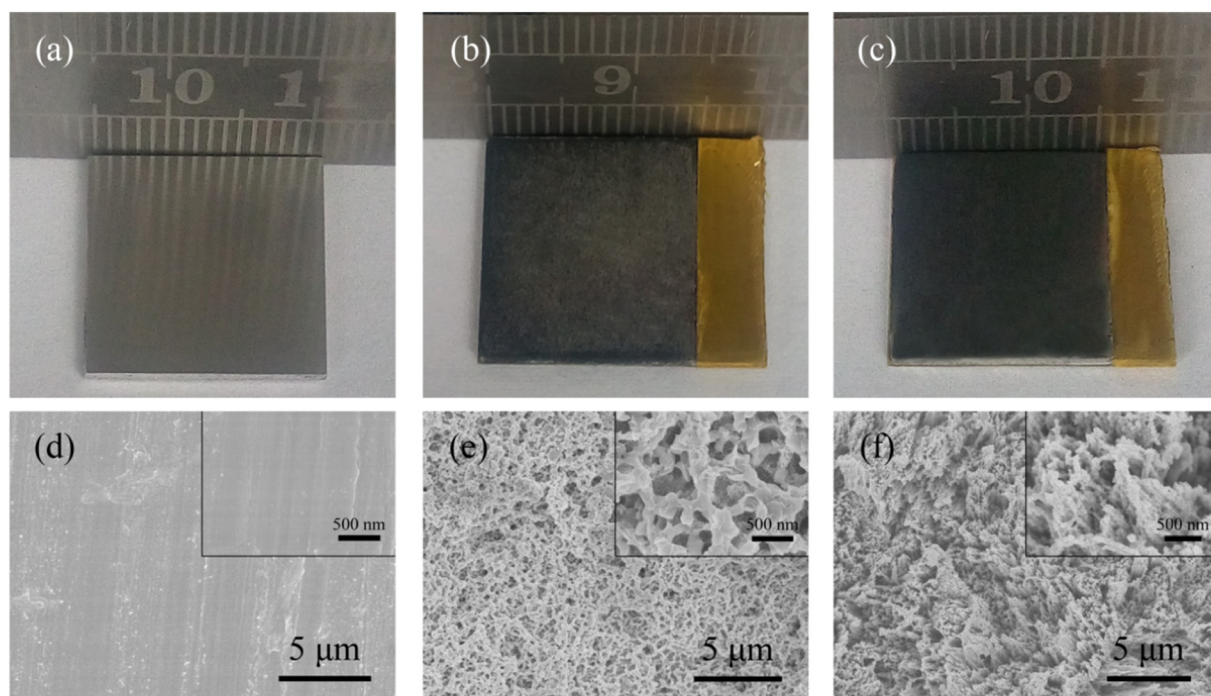


Fig. 2. Optical photographs and SEM images of the bare and anodic treated Mg alloy substrates. (a, d) bare Mg alloy, (b, e) CF-25 and (c, f) CF-50. Insets of the SEM images show the magnified views.

Mg alloy substrate under an ultrasonic condition [34]. Therefore, it would be promising that more effective coatings or films might be produced on Mg alloys by adopting different external fields at the ionic liquid/substrate interface. DESs would decompose at both electrodes over long periods of electrolysis to form chlorinated products and organic compounds via Hoffman elimination [35]. The decomposition of DESs would produce anions and cations which have different affinity for absorption on the Mg alloy surface. Therefore, conversion films could form by interactions between the decomposition products and the Mg alloy [23].

An electric field was applied to stimulate the decomposition of DES and facilitate the reaction of DES/Mg alloy interface in the present work. Interestingly, conversion films with various nanostructures were formed on the Mg alloy substrates by the proposed anodic treatment in the DES. Superhydrophobic surface (SHS) and slippery lubricant-infused porous surface (SLIPS) were further endowed to the conversion films under the inspiration of natural *Nepenthes* pitcher plants. The composition, structure and corrosion behavior of the conversion films were investigated. Moreover, a possible film formation mechanism was

proposed. This work is anticipated to stimulate the practical application of DESs in the surface treatment of Mg alloys for corrosion protections.

2. Experimental

2.1. Preparation of conversion films and the following surface modifications

Choline chloride [$\text{HOC}_2\text{H}_4\text{N}(\text{CH}_3)_3^+ \text{Cl}^-$] (analytical reagent, purity $\geq 98.0\%$) and ethylene glycol [$(\text{CH}_2\text{OH})_2$] (analytical reagent, purity $\geq 99.0\%$) were mixed in a 1:2 mol ratio without further purification and subsequently stirred at 80°C until a homogeneous DES formed. AZ31B Mg alloy substrates ($45 \text{ mm} \times 17 \text{ mm} \times 1 \text{ mm}$) were previously abraded by SiC papers up to 1000 grit, and then ultrasonically washed in an acetone (analytical reagent, purity $\sim 99.7\%$) bath. Each pretreated substrate was sealed by polyimide tapes leaving a single side of $15 \text{ mm} \times 17 \text{ mm}$. A facile anodic treatment was performed in a chronopotentiometry mode using a two-electrode electrochemical cell (CHI660e, Chenhua Instruments Inc., China). The anodic voltage was enabled through tuning current density. An unsealed Mg

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