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# Corrosion protection of AZ91D magnesium alloy with sol-gel coating containing 2-methyl piperidine

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#### ABSTRACT

Sol-gel coatings were prepared using 3-glycidoxypropyltrimethoxysilane (GPTMS) and tetraethoxysilane (TMOS) as precursors, diethylentriamine as curing agent. Inhibition effect of 2-methyl piperidine on AZ91D in 0.005%, 0.05% and 0.5% (wt.%) NaCl solution is investigated. Potentiodynamic polarization tests revealed that 2-methyl piperidine significantly decreased the anodic activity of AZ91D especially at high concentration. Corrosion behaviors of sol-gel coatings incorporated with 2-methyl piperidine on AZ91D in the Harrison's solution were analyzed using electrochemical impedance spectroscopy (EIS). EIS results showed the corrosion resistance of sol-gel coatings and sol-gel sealed phosphate conversion coating on AZ91D were significantly improved through addition of 2-methyl piperidine. Fourier transform infrared spectra (FT-IR) confirmed that the 2-methyl piperidine was compatible with sol-gel matrix.

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#### 1. Introduction

The low density and high specific strength, stiffness and elastic modulus of magnesium alloy make it attractive for traditional aerospace industry. Another impulse to improve energy efficiency and reduce environmental pollution makes it also imperative for increasing potential use of magnesium alloys in electronics, automobile and telecommunication industries. However, magnesium alloys are poor in corrosion resistance. Magnesium is the most active one in all the industrial metals, which has the lowest corrosion potential, compared with other metals such as Fe, Ni and Zn. Magnesium alloys are susceptible to corrosion with existence of H<sub>2</sub>O and Cl<sup>-</sup>. It is necessary for magnesium alloys to have surface treatment and subsequent organic coatings to prevent them from corrosion.

The surface treatment of magnesium alloys is mainly comprised of the following technologies. The first is chemical conversion coating, which consists of chromate conversion coating and phosphate conversion coating. The former is under stricter pressure to be used due to the harmful effect on human body [1]. Phosphate conversion coating is an environmentally compliant technology and is attached of more importance [2]. The second is anodizing, which utilizes electrolysis to produce an oxidized film. Generally, the anodized

In recent years, upon the wide application of sol-gel conversion coatings, a few reports of sol-gel coatings on magnesium alloy can be found. Tan [7] reported the effect of sealing of double-layer silane-based sol-gel coating on anodized film. Supplit [8] found that after acid pickling and subsequent sol-gel coating, the corrosion rate of AZ31 magnesium alloy was reduced to 1/60 as much as that before treatment. This work aims at developing environmental friendly sol-gel coatings with good corrosion resistance. This work investigated the corrosion resistance and sealing ability on phosphate conversion coatings of silane-based coatings with addition of 2-methyl piperidine. Heterocyclic compounds, especially which have lone pair electrons, were considered effective inhibitors in electrolyte or sol-gel coatings through adsorption or complexation with metal surface [9,10]. It is of significance to determine the inhibition effect of 2-methyl piperidine (molecular structure as shown in Fig. 1) in sol-gel coatings on magnesium alloy, which has nitrogen atom with lone pair electrons.

film of magnesium alloy tends to be porous and needs sealing. Micro-arc oxidizing is newly developed technology upon traditional anodizing treatment of magnesium alloys, i.e., the ceramic film is produced by high-voltage current, which is more corrosion and wear resistant [3,4]. The third is electrolessplating. The most widely researched type is Ni–P plating, which is characterized by excellent corrosion- and wear-resistance [5,6]. After surface treatment, organic coatings, i.e., epoxy, vinyl, and polyurethane coatings are applied to protect the substrate from corrosion. The research of organic coatings was less than that of surface treatment on magnesium alloys because the latter is key issue to protect magnesium alloy.

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Fig. 1. 2-Methyl piperidine.

#### 2. Experimental

#### 2.1. Substrate preparation and coating procedure

The substrate is commercial AZ91D. The samples with size of  $10\,\mathrm{mm}\times10\,\mathrm{mm}\times1.5\,\mathrm{mm}$  were gradually polished with 400#, 600#, 800# and 1000# emery paper, then washed by distilled water for 2 min, finally dried by cold air for use. The preparation of phosphate conversion coating was applied on substrate treated by polishing with 800# emery paper and alkaline washing. Five grams of  $\text{Ca}(\text{NO}_3)_2$  was added to a base formulation. The substrates were immersed in base solution

(2)

HO 
$$\stackrel{\text{OH}}{=}$$
 OH  $\stackrel{\text{OH}}{=}$  HO  $\stackrel{\text{OH}}{=}$  OH  $\stackrel{\text{H}^+/\text{H}_2\text{O}}{=}$  OH  $\stackrel{\text{OH}}{=}$  OH  $\stackrel{\text{NH}_2}{=}$  OH

HRN 
$$H_2C$$
 OH  $O$   $Si - OX$   $OX$   $OX$   $OX$   $OX$   $OX$   $OX$ 

 $X=H, (SiO_2)n (From\ hydrolysis\ of\ TMOS) \quad RNH2=NH_2CH_2CH_2NHCH_2CH_2NH_2 \eqno(3)$ 

$$\begin{array}{c} \text{OX} \\ \text{I} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OH} \\ \text{OH} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OH} \\ \text{OH} \\ \end{array} \\ \begin{array}{c} \text{OX} \\ \text{I} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OX} \\ \text{I} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OX} \\ \text{I} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \text{OX} \\ \end{array} \\ \begin{array}{c} \text{OH} \\ \text{OX} \\ \text$$

**Scheme 1.** Film forming process of silane sol–gel coating.

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