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Smart self-healing anti-corrosion vanadia coating for magnesium alloys

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ABSTRACT

Eco-friendly vanadia based chemical conversion coating was applied for improving the corrosion resistance of a newly developed magnesium AZ31 HP–O alloy. The effect of vanadia solution concentrations (10, 30 and 50 g/l) and pH (neutral pH 7 and pH 9) on the corrosion protection performance of a magnesium substrate were investigated. EIS and linear polarization techniques were used to evaluate the electrochemical behavior in 3.5% NaCl. The results showed a marked increase in the localized corrosion resistance after applying vanadia surface treatment of 50 g/l due to self-healing effect. The optimum conditions to obtain protective coatings for AZ31 HP–O with a self-healing ability were determined. Changes in surface morphology, composition and microstructure of the conversion coatings were followed by SEM-EDS and macroscopic imaging techniques.

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1. Introduction

Sustainable energy saving and reducing CO_2 emissions from transportation by development of advanced technologies for highperformance light-metal-alloy materials for fuel-efficient vehicles is a most important topic for all manufacturers and scientists working in transportation industry. Improving the mechanical properties, formability and corrosion resistance of magnesium alloys has got special attention.

Magnesium possesses the lowest density among all metallic construction materials, high specific strength as a substrate, and good castability and weldability characteristics. Unfortunately, magnesium has high chemical affinity to react with atmospheric oxygen and water resulting in the formation of a porous film on the surface which does not offer protection.

In earlier days, hexavalent chromates were widely used for producing conversion coatings [1,2] to protect magnesium alloys. In spite of its toxicity, chromium (VI) has remained an essential ingredient in the metal finishing industry for corrosion control. But in view of the economic impact of corrosion damage, the environmental and health problems caused by chromium (VI), and the increasing regulatory restrictions, scientists have an increasing inducement to develop a new generation of protective coating systems.

The advanced magnesium alloys that are being developed and used in modern automotive and aerospace industries require high performance and more durable coating systems. The current conventional anti-corrosion coatings behave mostly as passive barriers that delay or minimize but not prevent the interaction of corrosive species with the metallic substrate. Other coating schemes are based on nanotechnology (nano-containers, capsules, reservoirs, etc.) that can be filled with inhibitor to protect the substrate from corrosion upon damage or scratch in the coating layer. However, more studies should be done to optimize the coatings conditions because such so called 'smart' coating technology is industrially unattractive as long as multi-steps (around 8-12 steps) have to be used to prepare the coatings and the raw materials are quite expensive. Moreover, the presence of tank/reservoir containing "liquid" inhibitors in the coating will may affect negatively the adhesion performance, wear and abrasion resistances of the coatings over the metallic substrates. The healing efficiency of such "smart" coatings at low temperature (>0 °C) is unknown as the inhibitors inside the reservoirs might freeze. The overall coating quality is also unclear at high temperature >40°C where swelling and micro-cracks due to partial inhibitor evaporation can be done [3].

This paper addresses part of our research activities at Max Planck Institute (MPI), focuses on a simple two-layer coating system, including a non-chromate conversion coating layer and a commercially available, self-priming top coating containing nontoxic corrosion inhibiting materials for some newly developed magnesium alloys and high-strength aluminum alloys [4–6].

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Vanadate solution is a well-known corrosion inhibitor and has already been used in many paint or coating systems for aluminum alloys and composite materials [7–12]. However, the research work reported on the introduction on vanadia as an anti-corrosion surface pretreatment on magnesium alloys is very scarce [4–6,12–17]. Introducing eco-friendly surface treatment such as vanadia coatings prior to a commercially available top coating (such as polyurethane, epoxy, and fluoropolymer) is an attractive alternative to existing systems, as it would reduce the complexity of the coating system by combining the primer and topcoat into a single layer while at the same time reducing volatile organic compounds. Furthermore, ease of application of vanadia surface treatment at low cost in a very short time (about 10 min) is another advantage for future industrial application [4–6].

The recent research work done at MPI, together with other previous research work [4–17] have identified vanadium oxide-based conversion coatings (VCCs) as potentially viable corrosion inhibitors to replace toxic chromate on magnesium and aluminum alloys. The VCCs show promise for corrosion protection to aluminum alloys, such as AA 6061 and aluminum metal matrix composites [7–12] and, very recently, the 2024-T3 used in military and commercial aircraft.

Vanadia coatings are deposited from aqueous solutions containing sodium vanadate using a free immersion processing technology. A thin layer of thickness about $1.5-2.5 \,\mu\text{m}$ composed mostly of vanadium oxide phases is deposited in 10 min on the magnesium alloy surface by a chemically driven deposition process [4–6].

2. Experimental

2.1. Materials and surface preparation

Specimens of AZ31 HP–O alloy (a modified version of standard Mg alloy AZ31D) provided by EADS, Germany, in the form $30 \text{ mm} \times 60 \text{ mm}$ taken from a sheet 3 mm thick were abraded to 800 # finish with SiC grit papers, degreased in acetone, washed with distilled water, and dried in dry air.

2.2. Solutions and surface treatment

The coatings are deposited from aqueous solutions containing sodium vanadate using a very simple spontaneous free immersion processing technology that is very similar to that currently used for the toxic chromate system. A thin layer of vanadium oxide conversion coatings is deposited on the magnesium alloy surface by a chemically driven deposition process. Typically, uniformly distributed compact coatings of thickness about 1.5–2.5 μ m were deposited. Depositions are achieved in 10 min and use commercially available chemicals, materials and equipment, making the overall process compatible with industrial operations such as those employed by current aircraft manufacturers.

Solutions used in this work were prepared using sodium vanadate salt with different vanadia solution concentrations. The magnesium AZ31 HP–O substrates untreated and treated directly with vanadia (by simple free immersion in vanadate solutions) were prepared under the following conditions:

- 1. As polished (untreated with vanadia coatings)
- 2. Treatment in 10 g/L NaVO3 solutions for 10 min
- 3. Treatment in 30 g/L NaVO₃ solutions for 10 min
- 4. Treatment in 50 g/L NaVO₃ solutions for 10 min

3. Testing

3.1. Electrochemical impedance spectroscopy (EIS)

EIS was used to evaluate the electrochemical behavior of the uncoated (as-polished) and coated magnesium samples in 3.5% NaCl solution open to air and at room temperature for up to seven days. A three-electrode set-up was used with impedance spectra being recorded at the corrosion potential E_{corr} . A saturated calomel electrode (SCE) was used as the reference electrode. It was coupled capacitively to a platinum wire to reduce the phase shift at high frequencies. EIS was performed between 0.01 Hz and 65 kHz frequency range using a frequency response analyzer (Electrochemical analyzer instrument: CompactStat Ivium Soft 1.805 Release IVIUM Technologies Netherlands). The amplitude of the sinusoidal voltage signal was 10 mV. The exposed surface area was 4 cm². All curves were normalized to 1 cm².

3.2 Linear polarization tests

Linear polarization measurements were performed for the samples previously immersed for 30 min in 3.5% NaCl solution. The scan rate was 0.05 mV/s using the electrochemical analyzer; Compact-Stat Ivium Soft 1.805 Release IVIUM Technologies Netherlands. The scan range was ± 20 mV with respect to the open circuit potential.

3.3 Surface characterization

SEM was used to examine the surface morphology of the coated samples after the immersion in 3.5% NaCl solution. SEM images of the samples immersed in 3.5% NaCl for seven days, washed with deionized water and then dried were obtained using a Gemini LEO 1550, 3 kV operating voltage, Zeiss, Germany.

Macroscopic images were taken using a Digital Optical metallographic microscope VHX-100K, KEYENCE, Japan, to investigate the types of corrosion produced on the substrate surfaces after seven days of immersion in 3.5% NaCl solution.

4. Results and discussion

4.1. SEM micrographs

SEM micrograph of the as-polished magnesium samples after immersion in 3.5% NaCl solution revealed presence of many surface defects which stimulated pitting and crevice corrosion on Mg AZ31 HP–O alloys (Fig. 1). After vanadia treatment, a thin vanadium oxide layer was formed (Fig. 1). The coating thickness is ranging from 1.5–2.5 μ m for the coating prepared by immersion in 50 g/l vanadia solution at neutral pH. The vanadia coated samples at 50 g/l showed a self-healing ability to block the localized and pitted area due to corrosion attack.

Increasing the solution pH of 50 g/l vanadia coatings from 7 to 9 affects negatively the corrosion protection performance. A network like micro-cracked surface coatings was observed at pH 9 (Fig. 1). These cracks may be attributed to hydrogen being released from the chemical reaction during the coating formation at alkaline pH or as a result of dehydration of the surface layer after high alkaline pH coating.

4.2. Optical microscopic examination and visual inspection

Visual inspection of the uncoated magnesium samples showed severe pitting as well as crevice corrosion after seven days of immersion in 3.5% NaCl solution. A marked decrease in the number and the size of pits was observed for the samples coated with vanadia. Download English Version:

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