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Short Communication

Investigation of multi-coating process treated magnesium alloy as bipolar plate in polymer electrolyte membrane fuel cell



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ABSTRACT

Magnesium alloy bipolar plates offer great advantage in weight reduction of polymer electrolyte membrane fuel cell. An attempt to improve the corrosion resistance and surface conductivity of magnesium alloy has been made by a medium electroless plating Ni coating prior to PVD deposition of carbon film, envisaging the application of these coating/substrate systems as bipolar plate in PEMFC. The results show that Ni + C coated magnesium alloy GW83 has the best corrosion resistance of $8.65 \mu\text{A cm}^{-2}$ and $0.80 \mu\text{A cm}^{-2}$ in the simulated cathodic and anodic environments. The release of metallic ions is also greatly inhibited by Ni + C coating. The interfacial contact resistance (ICR) of the magnesium alloy GW83 is decreased from $196.5 \text{ m}\Omega\text{-cm}^2$ to $2.97 \text{ m}\Omega\text{-cm}^2$ by Ni + C coating at the compaction force of $150 \text{ N}\cdot\text{cm}^{-2}$. After potentiostatic tests, Ni + C coated sample has the lowest ICR of $15.6 \text{ m}\Omega\text{-cm}^2$ and $52.6 \text{ m}\Omega\text{-cm}^2$ in the anode and cathode environment, respectively.

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Introduction

Polymer electrolyte membrane fuel cell (PEMFC) has received considerable attention as an alternative power device for various industrial applications due to its excellent energy

conversion efficiency, high power density, low operating temperature and low emission [1–4]. The bipolar plate is a key multifunctional component in PEMFC, which separates individual cells, distributes reactive gases, collects the electrons, and removes the heat and exhaust water from the fuel cell [5–7].

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As per material point of view, bipolar plate material should have a balance of the electrical and thermal conductivity, corrosion resistance and gas impermeability, mechanical performance [8–10]. Metallic bipolar plates are of increasing interest due to their low cost, ease of manufacture, high power density and good mechanical properties [2,11]. However, as most of the metals are prone to be oxidized, the high surface electrical resistance caused by passive film and insufficient corrosion resistance in PEMFC working environment remain key challenges for metallic bipolar plate [12,13]. Hence, in order to achieve desired electrical and corrosion resistant properties, further investigation on metallic bipolar plate becomes an important area of research.

Since there are hundreds of bipolar plates in each PEMFC stack, the great potential in weight reduction of magnesium alloy has obvious advantages in transportation applications [14–16]. However, little researcher has suggested magnesium alloy as bipolar plate materials mainly because they are the least noble metal and highly susceptible to corrosion [17]. Therefore, it is essential to use surface engineering to improve the corrosion resistance of magnesium in PEMFC working environments.

Physical vapor deposition (PVD) is a versatile method to improve surface performance of materials as it can deposit various coating materials without thermodynamic limitations pertaining to solubility and phase diagrams [18]. Hollstein et al. [19] deposited TiN coating on AZ31 magnesium alloys by PVD and reduced the current density from 1700 A cm^{-2} to 300 A cm^{-2} . Altun et al. [20] coated multilayered AlN and AlN + TiN on AZ91 magnesium alloy using magnetron sputtering and demonstrated improved corrosion resistance. However, pinholes and cracks are inevitable when preparing PVD coatings [21,22] and the coatings are prone to local corrosion especially after long time immersion in corrosive solution [23,24]. In addition, magnesium alloys are usually considered as “difficult to plate metal” due to the thick oxide layer, inhomogeneous microstructure and active surface [14]. In order to overcome abovementioned challenges, multi-coating process treatment has been applied. Wiliam Grips et al. [25] investigated the electrochemical behavior of TiN, CrN, TiAlN and multilayer TiAlN/CrN coatings with $5 \mu\text{m}$ electroless nickel interlayer in 3.5% NaCl solution. The results indicated that the corrosion resistance was improved by at least an order of magnitude by adding of electroless nickel interlayer.

In this study, we fabricated carbon film with nickel interlayer (Ni + C coating) via multi-coating process including an electroless nickel deposition and subsequent PVD carbon film deposition, to improve the corrosion resistance and surface conductivity of magnesium alloy in PEMFC solution. The feasibility of multi-coating process treated magnesium alloy as bipolar plate in simulated PEMFC working environments is evaluated and discussed from the aspects of corrosion resistance and surface conductivity.

Experimental

Magnesium alloy GW83 with chemical composition (wt.%) of Gd: 8%, Y: 3.2%, Zr: 0.5% and Mg: balance, was chosen as substrate material. The samples were cut into $10 \text{ mm} \times 10 \text{ mm}$

pieces with 4 mm in thickness. All the samples were polished up to No. 2000 SiC waterproof abrasive paper, cleaned with acetone and distilled water in an ultrasonic bath, and dried before subjecting to the following treatments.

The nickel electroless plating includes the pretreatment and the electroless plating. In the pretreatment, solution with 50 g/L NaOH and 15 g/L NaPO_3 was used to alkaline wash at temperature of 55–95 °C. After alkaline wash, acid wash with solution including 70 g/L hydrochloric acid was conducted at room temperature. Following the acid wash, the solution with 70 g/L NaOH and 50 g/L SDS was used to alkaline wash at temperature of 55–95 °C. In the last step of pretreatment, the solution with the concentration of 70 g/L hydrochloric acid was used to acid wash. In the process of electroless plating, the plating solution was composed of 15 g/L $\text{NiCO}_3 \cdot 3\text{Ni}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$, 20 g/L $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 30 ml/L HF, 30 ml/L lactic acid, 2 mg/L CdSO_4 , 4 mg/L KIO_3 . The temperature was kept between 80 and 90 °C.

After nickel electroless plating, the samples were put into a Teer UDP 650 CFUBMSIP system to deposit carbon film. The coating system consists of two targets of 99.99% pure graphite and two targets of 99.99% Cr. High purity argon (99.99%) were used as the sputtering gases. The chamber was evacuated to a base pressure below 5.0×10^{-5} Torr using a turbo molecular pump and a rotary pump. Before the deposition process, the substrates were sputtered by Ar ions to clean the chamber and the samples at bias voltage of -500 V . During deposition, the bias voltage was kept at -60 V . A Cr transition layer was first deposited with chromium target current of 5 A for 15 min. Then, a thin intermediate CCr_x layer was deposited as an interfacial layer by reducing the current supplied to the Cr targets and increasing simultaneously the current supplied to the carbon targets from 0.2 A to 5 A. Finally, the carbon film was deposited using two carbon targets synchronously. The graphite target working current was 5 A and coating time was 2 h.

The surface morphology of coated and uncoated samples before and after the potentiostatic test was examined by field-emission scanning electron microscopy (FE-SEM) of HITACHI S-4800. The cross section of coated samples was also characterized by SEM and EDS to identify the cross section chemical distribution of Ni + C coating.

The electrochemical behavior of the uncoated and coated magnesium alloy GW83 was measured in simulated PEMFC solution containing 0.001 M sulfuric +0.1 ppm NaF (pH = 3). Three-electrode system composed of a platinum sheet as counter electrode, mercury sulfate electrode (MSE) as the reference electrode, and sample as working electrode was used. Reference electrode (MSE) was separated from the solution by a Luggin capillary to avoid anion contamination. The potentiodynamic and potentiostatic tests were conducted on Zahner Zennium electrochemical workstation. All the electrochemical tests were conducted at 70 °C to simulate the PEMFC condition. The solution was purged with either air (to simulate cathode environment) or hydrogen gas (to simulate anode environment) prior to and during the electrochemical test. The open circuit potential versus time was recorded for 1 h to ensure electrochemical stability before the electrochemical test. Potentiodynamic polarization was performed with a potential scanning rate of 1 mV s^{-1} . The potentiostatic test was conducted for 8 h at an applied potential of 0.23 V vs MSE (equally to 0.6 V vs SCE) and

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