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The effects of ionomer content on PEM water electrolyser membrane electrode assembly performance

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ABSTRACT

In this study, the effects of Nafion[®] ionomer content in membrane electrode assemblies (MEAs) of polymer electrolyte membrane (PEM) water electrolyser were discussed. The MEAs were prepared with a catalyst coated membrane (CCM) method. The catalysts inks with Nafion ionomer could form uniform coatings deposited on the membrane surfaces. SEM and area EDX mapping demonstrated that anode catalyst coating was uniformly distributed, with a microporous structure. The contents of Nafion ionomer were optimized to 25% for the anode and 20% for cathode. A current density of 1 A cm⁻² was achieved at terminal voltage 1.586 V at 80 °C in a PEMWE single cell, with Nafion 117, Pt/C as cathode, and Ru_{0.7}Ir_{0.3}O₂ as anode.

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1. Introduction

Water electrolysis is considered a significant technique in the solutions of both energy and environmental issues, because it is a clean method of hydrogen production [1] and an efficient and convenient way of storing energy from renewable sources such as solar, wind and hydroelectric power [2]. Recently, polymer electrolyte membrane (PEM) water electrolyzers, also called solid polymer electrolyte (SPE) electrolyzers, have become of increasing interest. PEM water electrolyzers exhibit several advantages compared with conventional alkaline water electrolyzers, including high hydrogen purity, greater safety, better reliability, lower energy consumption, and possibility of high current density [3–5]. Although the first SPE electrolyser was developed in 1960s by General Electrics Ltd for space applications, PEM water electrolyzers are scarcely applied in large scale hydrogen production yet [6]. One factor

for this lack of adoption is the use of noble metal catalysts in the electrodes, e.g. Ir, Ru, Pt, Pd. Especially, for the anode of a PEMWE, where oxygen evolution reaction leads to the greatest polarization loss, only stabilized catalysts based upon ruthenium or iridium are the best choices [7].

Most PEM water electrolyzers employ perfluorosulfonic acid membranes, e.g. Nafion[®] membranes, which is another reason of their high cost. Indeed, utilization of cheaper catalysts or alternative polymer electrolyte membranes may effectively reduce the cost of PEM water electrolyzers. However, at least now, there are few options for the researchers developing PEM water electrolyzers [1–4]. Therefore, studies on PEM water electrolyser membrane electrode assemblies (MEA), where the electrochemical reactions occur, are of reasonable significance. Optimization of the MEAs could improve the performances and thus reduce the cost of PEM water electrolyzers currently employing Nafion membranes

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and noble metal catalysts. Generally speaking, there are two MEA fabrication methods, which are the catalysts coated on gas diffusion layer (CCG) method and the catalyst coated on membrane (CCM) method. So far as PEM water electrolyzers are concerned, MEAs prepared with CCM method exhibited better performance than those prepared with CCG method [5,6,8–10]. CCM method could possibly enhance the interfacial structure between the membrane and catalysts layer, leading to greater catalyst utilization and smaller MEA resistance [14]. Besides, the catalyst layer coated on membrane would exhibit good mechanical stability and hydrophilicity [12–14], which are important factors of water electrolyzers since a large amount of gas bubbles evolve everywhere in the catalyst layer. In recent years, several PEM water electrolyzers prepared with CCM method have demonstrated high performances [5,6,10–14]. In all of these CCM fabrications, Nafion ionomers were applied in the catalyst layers. The Nafion ionomer is a multifunctional component of the catalyst layer. The Nafion ionomer acts (1) as a conductor extending proton conduction from the bulk of PEM membrane to the surface of catalysts, (2) as a binder providing a three dimensional structure of the catalyst layer, with abundant gas channels and certain mechanical stability, and (3) as a hydrophilic agent retaining moisture inside the catalyst layer. If the amount of Nafion ionomer is low, protons may not access every part of the catalyst layer, leading to insufficient catalyst utilization. However, increasing the amount of ionomer may increase blockage of gas transport channels in catalyst layer. Besides, when oxides catalysts based on RuO_2 and IrO_2 are employed in the anode, electron conduction in the catalyst layer might be reduced once the ionomer exceeds a certain amount. Therefore, there is normally an optimum ionomer content in the catalyst layers while other fabrication parameters (such as catalyst loading, catalyst size, solvent of catalyst ink, etc.) are constant. However, there is little information reported on the optimization of Nafion ionomer content for MEAs. In the present paper, MEAs of PEM water electrolyzers were prepared with a CCM method using various Nafion ionomer contents. The effects of Nafion ionomer content were discussed, with regard to anode performances and cathode performances, respectively.

2. Experimental method

2.1. MEA preparation

Catalyst inks for spraying were prepared by mixing catalysts and Nafion ionomer (5%, Aldrich) with different weight ratios in a solvent of deionized water and isopropanol (volume ratio 1:9). The mixture was finely dispersed in an ultrasonic bath for 30 min. For the MEA, $\text{Ru}_{0.7}\text{Ir}_{0.3}\text{O}_2$ (8–15 nm powders, synthesized in our lab) and Pt/C (20%, Alfa Aesar) were used as catalysts for oxygen evolution reaction (OER) and hydrogen evolution reaction (HER), respectively. $\text{Ru}_{0.7}\text{Ir}_{0.3}\text{O}_2$ was prepared with the Adams fusion method, as described in reference [14]. A 10 mL aqueous solution containing 0.0007 mol RuCl_3 (Alfa Aesar) and 0.0003 mol $\text{H}_2\text{IrCl}_6 \cdot x\text{H}_2\text{O}$ (Sigma Aldrich) were mixed with 10 g NaNO_3 . This NaNO_3 -solution slurry was completely

dried at 100 °C and consequently heat-treated for an hour at 400–500 °C. The result mixture of fusion was rinsed with abundant deionized water with a centrifuge. Nafion ionomer content of both electrodes was controlled to 0%, 5%, 10%, 15%, 20%, 25%, 30%, 35% and 40%, where the percentages regard to weight ratios of Nafion ionomer in the total content of catalyst and ionomer. The inks were sprayed onto smooth PTFE sheets under N_2 gas with 1 bar pressure. Then the catalyst coatings on PTFE sheets were hot-pressed onto Nafion 117[®] membranes. Prior to hot pressing, the Nafion 117 membrane was treated with 3% H_2O_2 and 0.5 mol dm^{-3} (M) H_2SO_4 and then rinsed with abundant deionized water before use. The catalyst loadings of anode and cathode catalyst layers were 1.5 mg cm^{-2} $\text{Ru}_{0.7}\text{Ir}_{0.3}\text{O}_2$ and 0.5 mg cm^{-2} Pt/C. After the PTFE sheets on both sides were removed, the catalysts coated membrane was sandwiched between two carbon papers (Toray, TGP-H-060), which were used as gas diffusion layers (GDL). For the anode, a protecting microporous layer (MPL) composed of 1 mg cm^{-2} $\text{Ru}_{0.7}\text{Ir}_{0.3}\text{O}_2$ and PTFE (60% dispersion in water, Aldrich) was deposited on the GDL before MEA preparation. The MEA was prepared by hot pressing the CCM and GDLs at 130 °C for 3 min at a pressure of 100 MPa.

2.2. Characterizations

The morphology of catalyst coating on membrane was examined by scanning electron microscopy (SEM, JEOL JSM-5300 Low Vacuum) and energy dispersive X-ray analysis (EDX, Rontec Edwin system with 25 KeV beam energy) attached to the above SEM.

2.3. Measurements

A single cell rig was designed and fabricated for testing the PEMWE performance. As shown in Fig. 1, flow fields of water were machined in two blocks made of PTFE. MEAs were placed between the two blocks. A DC power supply (Thurlby Thandar Instruments PL330) was employed to provide the terminal voltage. Deionised water, which has been pre-heated to 60 °C, was pumped into the cell with a pressure of 0.1 Bar and a flow rate of 5 $\text{cm}^3 \text{min}^{-1}$. The single cell was maintained at 80 °C for current and voltage recording.

Electrochemical experiments were carried out with an Epsilon potentiostat (BASi, Epsilon_EC_Ver 1.60.70). The electrochemical cell used a 0.5 M H_2SO_4 solution prepared with deionized water, a platinum foil counter electrode and a saturated calomel reference electrode (SCE). The working electrodes were prepared by pipetting the inks for MEAs onto a polished glassy-carbon electrode (geometric area is 0.07 cm^2). For the OER, the scan rates (v) of cyclic voltammetry (CV) were 1, 2, 3, 5, 7, 10, 15, 20, 30, 40, 50, 60, 70, 80, 100, 150, 200, 250, 300, 500 mV s^{-1} . The voltammetric charge (q^*) for $\text{Ru}_{0.7}\text{Ir}_{0.3}\text{O}_2$ electrodes was determined by integrating the area of the cyclic voltammogram between –42 and 1158 mV referred to SCE. The total and outer voltammetric charges (q_T and q_O) were obtained from fitting of the linear regions in the q^{*-1} vs. $v^{0.5}$ plots and the q^* vs. $v^{-0.5}$ plots, respectively. For the Pt/C electrode, the apparent electrochemical surface area (ESA) of Pt was determined by calculating the amount of

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