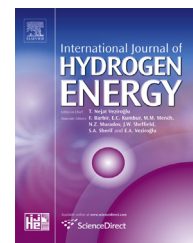


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Membrane electrode assemblies with low noble metal loadings for hydrogen production from solid polymer electrolyte water electrolysis

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ABSTRACT

High performance membrane electrode assemblies (MEAs) with low noble metal loadings (NMLs) were developed for solid polymer electrolyte (SPE) water electrolysis. The electrochemical and physical characterization of the MEAs was performed by I – V curves, electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM). Even though the total NML was lowered to 0.38 mg cm^{-2} , it still reached a high performance of 1.633 V at 2 A cm^{-2} and $80 \text{ }^\circ\text{C}$, with IrO_2 as anode catalyst. The influences of the ionomer content in the anode catalyst layer (CL) and the cell temperature were investigated with the purpose of optimizing the performance. SEM and EIS measurements revealed that the MEA with low NML has very thin porous cathode and anode CLs that get intimate contact with the electrolyte membrane, which makes a reduced mass transport limitation and lower ohmic resistance of the MEA. A short-term water electrolysis operation at 1 A cm^{-2} showed that the MEA has good stability: the cell voltage maintained at $\sim 1.60 \text{ V}$ without distinct degradation after 122 h operation at $80 \text{ }^\circ\text{C}$ and atmospheric pressure.

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1. Introduction

Solid polymer electrolyte water electrolysis (SPEWE) is a pleasing way for pure or ‘green’ hydrogen production at low temperature without fossil fuel consumption and emission of greenhouse gases such as CO_x , SO_x and NO_x and any toxic particulates. Therefore, SPEWE is currently believed as a favorable technique for extensive hydrogen production in the future [1–3]. In recent years, although the researchers have speeded up the development of SPEWE, the systems are still too costly to replace traditional water–alkaline electrolyzers [1,3].

In conventional SPEWE technology, Ir (or IrO_2) and Pt are commonly used noble metal catalysts respectively for the

anode oxygen evolution reaction (OER) and the cathode hydrogen evolution reaction (HER) [3–10]. According to Millet et al. [3], these noble metal loadings (NMLs) require a significant reduction from a few mg cm^{-2} (current state-of-the-art) down to ca. 0.1 mg cm^{-2} for the whole cost reduction. Therefore, many of the recent studies have been focusing on developing highly active anode and cathode catalysts for SPEWE [10–20] with reduced noble metals content and overall cost. For example, oxides such as Ir–Ru [13,14], Ir–Sn [15–17], Ir–Ta [14,18], Ir–Ru–Sn [19] and Ir–Ru–Ta [20], etc. were developed as oxygen evolution electrocatalysts. Some of these catalysts showed much better performance for water electrolysis than the commonly used IrO_2 catalyst. However, it is still difficult to decrease the content of these noble metals to

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an acceptable level due to the unavailability of carbon support suitable for these catalysts [3,21]. On the other hand, some studies have intended to develop low-cost SPEs in place of the expensive Nafion[®] membrane [22–27]. Although significant performance values have been obtained in some studies, the practical use of these composite membranes under industrial electrolysis conditions has not been sufficiently demonstrated. For example, for non-homogeneous membrane, such as a Nafion[®] and PTFE reinforcement membrane, delamination of the MEA in ‘harsh’ environments such as hot water in a working electrolyzer should be considered since the thermal expansion and swelling coefficients of two polymers are different.

Like in low temperature hydrogen fuel cells, the MEA is the key part of an SPEWE system. The fabrication methods for MEAs with low NML (usually, Pt) have been studied extensively in the fuel cell technology to reduce the noble metal use and, accordingly, cost [28–30]. However, there has been limited work reported on developing MEAs with low NMLs for SPE water electrolysis, even though these two fields are closely related. One of the main reasons is the difficulty in fabricating thin (a few microns thick) and uniform anode catalyst layer (CL) due to the unavailability of supports suitable for the anode catalysts [3,21].

In our previous work, a novel catalyst coated membrane (CCM) method, termed catalyst sprayed membrane under illumination (CSMUI) [31], was developed for MEA preparation for SPEWE. The MEAs prepared by this method exhibited high performances for water electrolysis. In this study, we investigated the feasibility of using CSMUI method to prepare low NML MEAs for SPEWE. A high performance MEA with low NML was obtained using CSMUI method via further optimization of the anode CL structure. The effects of the NML and cell temperature on the cell performance were investigated. Polarization and durability tests showed that the MEA with low NML exhibited good performance and stability for SPE water electrolysis.

2. Experimental methods

2.1. Preparation of MEAs

The catalyst inks were prepared by mixing the catalysts powder into a blend of 5 wt.% Nafion[®] ionomer solution (DuPont, USA) and isopropanol. The catalyst used for the cathode and the anode were Hispec 4100 Pt/C (20 wt.% Pt, Alfa Aesar, Johnson Matthey) and IrO₂ (99.9%, Alfa Aesar, Johnson Matthey), respectively. Before being used, the dispersion mixture was sonicated in a 40 kHz ultrasonic bath for 40 min. The polymer electrolyte membrane used in this study is Nafion[®] 212 (DuPont, USA). The membranes were pre-treated at 80 °C in the solutions of 5 wt.% H₂O₂, ultrapure water, 0.5 M H₂SO₄ and ultrapure water for 60 min, sequentially.

The CCMs were obtained by spraying the catalyst inks onto the both side of the pretreated membranes with a spray gun (nozzle caliber: 0.2 mm, atomization style). A more detailed account of the preparation of the CCM has been given previously [31]. The Nafion[®] content varied from 0 wt.% to 30 wt.% in the anodes to be optimized, while it was always 30 wt.% in the cathodes for this study. The NMLs at the cathode and

anode were determined by two ways. Firstly, the catalyst quantities for the cathode and anode inks were weighed accurately. Normally, 10% more catalyst than the calculated amount was used considering the loss during the fabrication process. Secondly, the uncoated membrane, the membrane coated with cathode CL, and the membrane coated with both cathode and anode CLs were weighted, whereby the individual NMLs for both the cathode and the anode can be calculated separately, to make sure that the NMLs conformed to the intended MEA design. Unless otherwise specified, the IrO₂ loading and Pt loading for the cathode and the anode were 0.04 and 0.4 mg cm⁻² respectively, much lower than for conventional SPEWE CCMs, amongst the lowest loadings for SPEWE that the authors have found in the literature [3,20,32].

Porous titanium (Ti) fiber (Bekenit, SaitamaKen, Japan; thickness 0.3 mm, 60% porosity) was used as the anode gas diffusion layer (GDL)/current collector. The GDL for the cathode was prepared with same procedure described in our previously work [31]. The active area of the prepared MEAs was 4 cm².

2.2. Evaluation of MEA performance in water electrolysis

An SPE water electrolysis cell was used to evaluate the performance of the as-prepared MEAs, and details of the cell can be found elsewhere [31]. The water electrolysis performance of the SPE electrolyzer was tested at 80 °C and atmospheric pressure. Preheated deionized water (18.3 MΩ cm), with a flow rate of 50 ml min⁻¹, was circulated and supplied to the anode compartment by a peristaltic pump. The water temperature was kept at 5 °C higher than the cell temperature. Total cell polarization curves were recorded galvanostatically between 1 mA cm⁻² and 2 A cm⁻² using a Neware battery testing system (Neware Technology Ltd, China).

2.3. SEM and electrochemical measurements of MEAs

An ultra-high resolution field-emission SEM (Nova[™] NanoSEM 230, FEI, USA) was used to observe the cross-sections and surfaces of the MEAs.

Electrochemical impedance spectroscopy (EIS) was performed by an Autolab PGSTAT 30 Potentiostat/Galvanostat (Metrohm) equipped with a 10 A booster and a frequency response analyzer (FRA). The impedance data were generated and simulated using the Autolab Nova software. During EIS tests, the cathode was served as both the reference electrode (RE) and the counter electrode (CE) since the polarization of HER is negligible compared to that for OER at anode during water electrolysis operation. The impedance spectra were recorded at a cell potential of +1.5 V in the frequency range of 0.1–10,000 Hz with sinusoidal amplitude of 5 mV.

3. Results and discussion

3.1. Water electrolysis performance of the MEAs with different NMLs

Through gradually decreasing the IrO₂ loading on the anode and Pt loading on the cathode, a series of MEAs with different

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