



# Investigations on degradation of the long-term proton exchange membrane water electrolysis stack



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## HIGHLIGHTS

- Degradation of electrolysis stack after running 7800 h is investigated.
- Cations occupied ion exchange sites of membrane and the catalyst layers.
- Cations impurities cause the increase in ohmic resistance and charge transfer resistance.
- The hydrogen crossover rate of the MEA slightly increased after 7800 h operation.
- Cell performance is almost completely recovered after dilute sulfur acid treatment.

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## ABSTRACT

A 9-cell proton exchange membrane (PEM) water electrolysis stack is developed and tested for 7800 h. The average degradation rate of  $35.5 \mu\text{V h}^{-1}$  per cell is measured. The 4th MEA of the stack is offline investigated and characterized. The electrochemical impedance spectroscopy (EIS) shows that the charge transfer resistance and ionic resistance of the cell both increase. The linear sweep scan (LSV) shows the hydrogen crossover rate of the membrane has slight increase. The electron probe X-ray microanalyze (EPMA) illustrates further that Ca, Cu and Fe elements distribute in the membrane and catalyst layers of the catalyst-coated membranes (CCMs). The cations occupy the ion exchange sites of the Nafion polymer electrolyte in the catalyst layers and membrane, which results in the increase in the anode and the cathode overpotentials. The metallic impurities originate mainly from the feed water and the components of the electrolysis unit. Fortunately, the degradation was reversible and can be almost recovered to the initial performance by using 0.5 M  $\text{H}_2\text{SO}_4$ . This indicates the performance degradation of the stack running 7800 h is mainly caused by a recoverable contamination.

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## 1. Introduction

The extensive consumption of fossil fuels over the last century and the associated perspectives of short-term supply shortages are calling for new, clean and cheap technologies. Hydrogen may play an important role as an energy carrier of the future [1]. Proton exchange membrane (PEM) water electrolysis is potentially interesting for the decentralized production of hydrogen from renewable energy sources [2,3], which offers a convenient and efficient way to produce hydrogen.

Compared to the conventional alkaline process, PEM water electrolysis offers a number of advantages for the production of electrolytic grade hydrogen, such as higher efficiency and reliability, higher gas purity (above 99.99%), and the possibility of producing compressed gases (up to 150 bar and more) for direct pressurized storage without any external mechanical compression [4,5].

Most research papers published in the open literatures on the subject focused on corrosion-resistive current collector [6–8], novel anode or cathode catalysts [9–13]. Although the long-term stability studies for the water electrolysis stack are of crucial importance from the view point of the industrial application, few publications are available concerning the degradation of PEM electrolysis stack [14,15].

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Millet et al. [14] fabricated a GenHy1000 stack and measured its performances. Although the performances almost maintained stable during the first 800 h of operation, they observed performance degradation over longer periods of time. The contamination of metallic cations in the feed water and the degradation of the MEAs were the main causes of performance degradation. Selamet et al. [15] developed and optimized a 10-cell stack, the initial efficiency and performance of the stack was tested. But only a single cell was tested for 2000 h continuously and an degradation rate of  $1.5 \mu\text{V h}^{-1}$  was measured. Wei G Q et al. [16] investigated the stability of a MEA with  $4 \text{ cm}^2$  active area in a single electrolyzer for 208 h, they indicated the short period performance decline of the MEA was mainly caused by a recoverable contamination.

In this research, the water electrolysis stack was fabricated and the long-term durability was tested. The performance degradation was investigated after 7800 h operation and the contamination sources was also identified by using different characterizations methods. Recovery of the electrolysis performance was obtained by means of dilute sulfur acid treatment.

## 2. Experimental

### 2.1. Preparation of MEAs

Membrane electrode assemblies (MEAs) were prepared by using the catalyst-coated membrane (CCM) technique, as follows. Pt/C (40 wt%, Johnson matthey, HiSPECTM 4000) was used as hydrogen evolution reaction (HER) electrocatalyst for the cathode, and Iridium black (Johnson matthey, HiSPECTM 160000) was used as oxygen evolution reaction (OER) electrocatalyst for the anode. To prepare a catalyst layer, a homogeneous ink composed of electrocatalyst, a Nafion solution (5 wt%, E.I. DuPont de Nemours and Company) and isopropanol was sprayed directly onto the each side of a Nafion 115 membrane (DuPont, thickness:  $127 \mu\text{m}$ , conductivity:  $0.10 \text{ S cm}^{-1}$  minimum). The electrocatalysts loading were  $1.5 \text{ mg cm}^{-2}$  Ir black and  $0.4 \text{ mg cm}^{-2}$  Pt, respectively. The Nafion loading in the catalyst layer was approximately  $0.6 \text{ mg cm}^{-2}$  for the hydrogen side and the oxygen side. To form a

MEA for water electrolysis, the prepared CCM was hot-pressed with a carbon paper (Toray, TGP-H-060) together at  $140 \text{ }^\circ\text{C}$  under a pressure of 1 MPa for 1.0 min. The effective area of the MEA was about  $160 \text{ cm}^2$ .

### 2.2. Water electrolysis stack

A 9-cells stack (hydrogen production capacity up to  $0.3 \text{ Nm}^3 \text{ H}_2 \text{ h}^{-1}$ ) with  $160 \text{ cm}^2$  active area per cell was designed and constructed, which was series connected in a filter-press configuration and used to investigate the durability. In the stack, porous titanium disks (Porosity: 40%, provided by Xi'an Baode Powder Metallurgy Co., Ltd., China) electroplated Pt (Suzhou Borui Industrial Material Science & Technology Co., Ltd., China) were used as the anodic current collector, a carbon paper (Toray, TGP-H-060) was used as the cathode current collector and 40 mesh titanium grids electroplated Pt (Suzhou Borui Industrial Material Science & Technology Co., Ltd., China) were used as the spacers. Silicon rubber gaskets (thickness: 0.6 mm) were used as the cell sealants. Both the endplates and the bipolar plates were made of titanium alloy (TC4, thickness: 20 mm, Baoti Group Co., Ltd., China) and titanium foils (TA2, thickness: 0.2 mm, Baoti Group Co., Ltd., China), respectively.

### 2.3. Evaluation of water electrolysis stack

A self-made test station was employed to implement the long-term test of the water electrolysis stack, as given in Fig. 1. During the operation of water electrolysis, deionized water (Milli-Q,  $18.2 \text{ M}\Omega \text{ cm}$  resistivity) was pumped into the anode side of the stack from the bottom of water tank and flowed back to the top of water tank. The tank was also used as the separator of water and oxygen. The total water flow was  $1.8 \text{ L min}^{-1}$  and the average flow rate was  $200 \text{ ml min}^{-1}$  per cell. The DC power source (KIKUSUI PAT60-133T) provided an voltage on the electrolysis stack. Both  $\text{H}_2$  and  $\text{O}_2$  produced were evacuated. The water electro-dragged was separated in the cathode separator and reflowed to the bottom of the anode separator. A desalination cartridge was used before the water was fed to the electrolysis stack. The feed water was heated

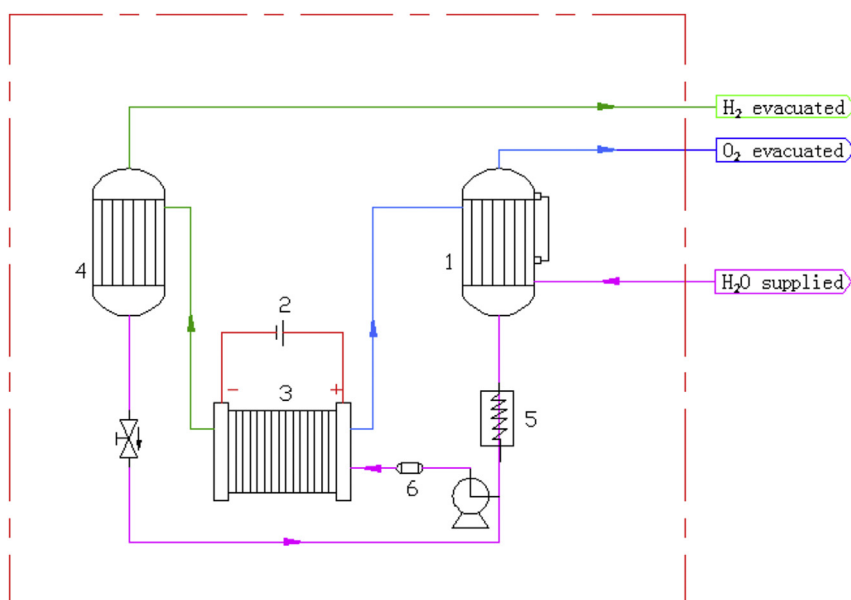


Fig. 1. Schematic sketch of test station for water electrolysis. 1. water tank; 2. DC power source; 3. electrolysis stack; 4. cathode separator; 5. heat exchanger; 6. desalination cartridge.

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