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Analysis of carbon-supported platinum through potential cycling and potential-static holding

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ABSTRACT

The stability of platinum and carbon support in catalyst-coated membrane (CCM) was investigated by a potential cycling between 0.7 and 0.9 V and a potential-static holding at 1.2 V, 1.3 V and 1.5 V in single cells. Clear cell performance deterioration can be observed by polarization curves during accelerated stress tests, along with electrochemical surface area (ESA) loss of Pt catalysts by cyclic voltammogram (CV). The X-ray diffraction (XRD) results of CCM before and after tests show that a distinct Pt agglomeration occurred from approximate 3 nm–8 nm in diameter, which is in accord with the observation of Pt/C by transmission electron microscopy (TEM). It is also interesting to note that, redeposited Pt particles in the membrane could be as large as hundreds of nanometers from TEM images of CCM microtomy. X-ray photoelectron spectroscopy (XPS) of carbon 1S indicates that the corrosion of carbon support is highly dependent on the holding potential, and enormous surface groups, such as carboxyl, lactones and ether were generated after tests. Meanwhile, a severer ESA loss of Pt after carbon corrosion under high potential holdings happens than that of potential cycling. The results indicate that both Pt and carbon support in the catalyst are important to maintain a long-term stable operation for fuel cells.
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Introduction

Proton exchange membrane fuel cell (PEMFC), a device which can convert chemical energy directly into electrical energy, is thought to be the best solution to transportation in the near future due to its high-efficiency, quick start-up and nearly zero-emission [1,2]. Nowadays, the durability of PEMFCs sets a formidable barrier to its commercialization [3].

Studies have shown several influencing factors on PEMFC durability, including water and thermal managements [4], PEM thinning [5,6], catalyst layer (CL) degradation [7,8], and

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gas diffusion layer (GDL) aging [9]. Among these, carbon-support platinum degradation is one of the most critical factors, where the reaction happens. Catalyst layer degradation can be classified into two aspects: platinum catalyst degradation and carbon support corrosion. It has been reported that the Pt catalyst experienced a certain loss of electrochemical surface area (ESA) resulted from Pt loss and agglomeration [10], and Pt particle size growth [11]. In addition, carbon support that anchors the platinum crystallites and provides electrical connectivity to the gas diffusion media is also subject to degradation [12–15]. Significant oxidation of carbon support is thought to deteriorate the performance of a PEMFC [14,16], due to the detachment of Pt particles from carbon and/or Pt agglomeration [17]. Ferreira et al. [18] discussed the instability of Pt/C electrocatalysts in PEMFC, confirming that coarsening of platinum particles occurred via Ostwald ripening and migration of soluble platinum species. Tommy et al. [19] examined effects of crossover hydrogen on Pt dissolution and agglomeration, showing that Pt growth and agglomeration were getting severer with less crossover hydrogen. Besides, Kangasniemi et al. [14] investigated carbon corrosion in PEMFC, demonstrating that oxides on carbon surface weakened the component hydrophobicity, which was an important factor in determining long-term PEM operation.

Potential cycling and potential-static holding is usually used to evaluate the stability of carbon-support platinum. Potential cycling around 0.8 V will cause platinum dissolving [20–22]. Above 0.9 V (RHE), electrochemical oxidation of carbon to CO_2 and CO could occur [23,24]. Patterson et al. used potential cycling method to investigate the platinum dissolution and the loss of electrochemical surface area, then correlating it to PEMFC performance degradation [25]. Kiho et al. [26] have used potential-static holding to analyze the electrooxidation of carbon supports under phosphoric acid fuel cells conditions. Therefore, combination of potential cycling and potential-static holding tests can provide a time-effective tool for evaluating the stability of carbon-support platinum in PEMFC. However, most of papers concerning Pt aggregation and carbon corrosion were only dealing with Pt/C in simulated half-cell environment instead of a single cell of PEMFC. Furthermore, the degradation mechanism of carbon-support platinum in PEMFC is still ambiguous to date.

In this investigation, in-house fabricated catalyst-coated membranes (CCMs) composed of composite polytetrafluoroethylene (PTFE) reinforced membranes and Pt/C were adopted to manufacture MEAs, tested with potential cycling varied from 0.7 V to 0.9 V and potential-static holding at 1.2 V, 1.3 V, 1.5 V to evaluate the stability of both Pt and its carbon support. In-situ CV tests, XRD, TEM, and X-ray photoelectron spectroscopy (XPS) characterizations were employed to study variation of both Pt catalyst and carbon support. Microtomy technique was introduced to observe the morphology of redeposited Pt in the membrane.

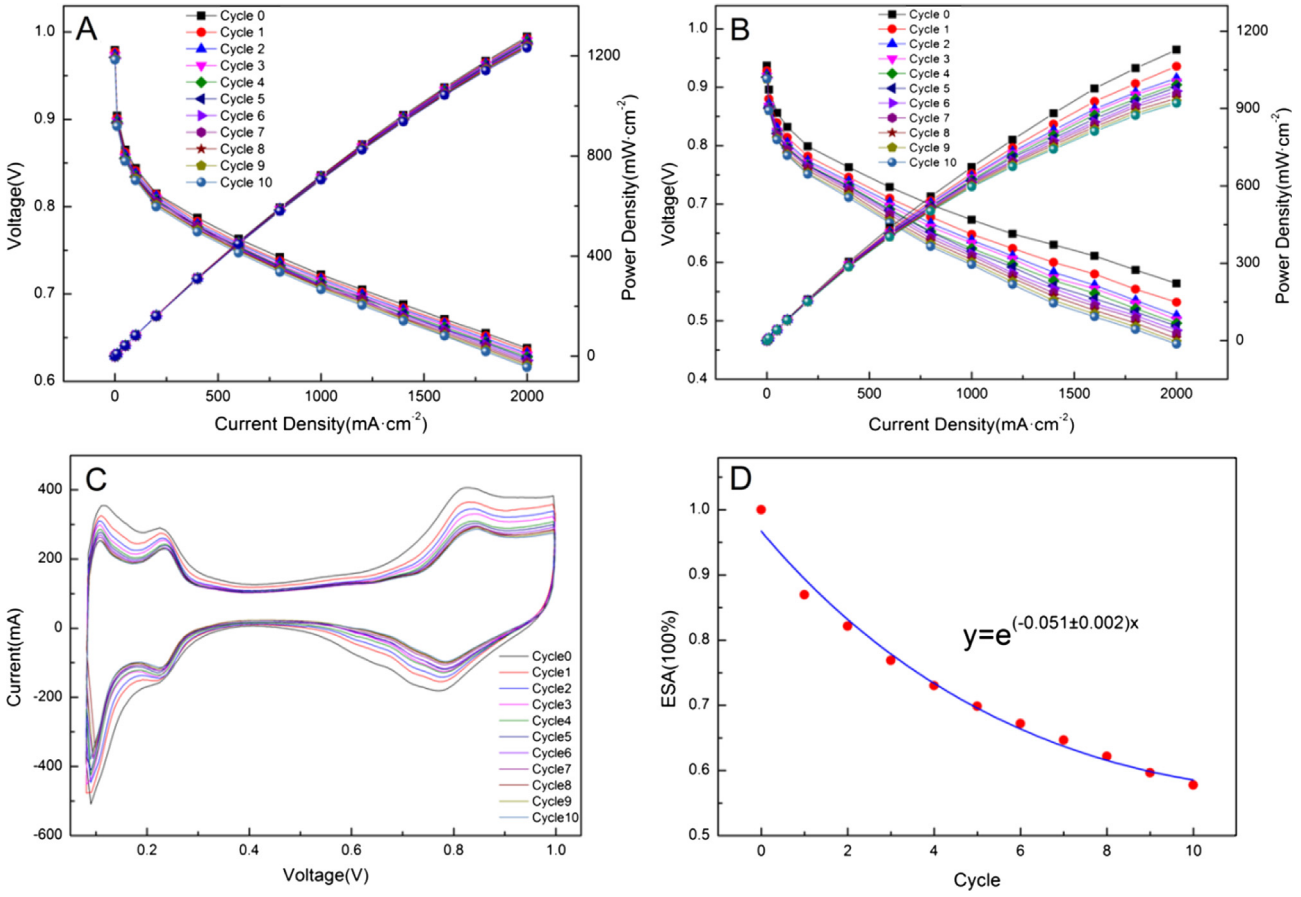

Fig. 1 – In-situ tests during potential cycling. (A) polarization curves under H_2/O_2 atmosphere; (B) polarization curves under H_2/Air atmosphere; (C) cyclic voltammogram curves; (D) variation of ESA.

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