Supplementary Material (ESI) for Chemical Communications

High Pt utilization PEMFC electrode obtained by alternative ion-exchange/electrodeposition

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Experimental Section

(1) **Electrochemical performance test**: Linearsweep voltammetry (LSV) and cyclic voltammetry (CV) were used to characterize the electrochemical performance of the catalyzed electrode prepared by the AIEE method. All electrochemical experiments were performed in a standard three-electrode cell at room temperature. The cell consists of a working electrode prepared by AIEE methods, a Pt wire counter electrode, and a saturated potassium chloride silver chloride electrode (SSCE) (0.20 V vs SHE), which served as a reference electrode. All potentials are quoted with respect to the SSCE reference electrode (0.20 V vs SHE), unless otherwise stated. Electrochemical experiments were performed with Autolab potentiostat/galvanostat PGSTAT 30 with GPES softwar (ECO. CHEMIE. BV)

(2) **CO stripping CV:** The 0.5 M H_2SO_4 electrolyte was first purged with high purity nitrogen. CO adsorption on the electrode catalyst was conducted by bubbling CO through the electrolyte for 15 min, followed by purging with nitrogen for 20 min to remove residual CO in the solution. The CO stripping CV curve and blank CV curve can be obtained from two consecutive scan cycles in the potential between -0.2 and 1.0 V at a sweep rate of 50 mV s^{-1}.

(3) The field emission scanning electron microscope (FESEM) (FEI Nova 400, FEI, USA) and field emission Transmission electron microscope (FETEM) (Tecnai GF2, FEI, USA) were utilized to observe the particle size and surface morphologies of the electrodes.

(4) **Pt Content Test:** Spectrophotometry with a color developing agent of $SnCl_2$ was used to determine the Pt content in a catalyzed electrode.[1] First, the catalyzed electrode after performance tests was calcinated in air at a temperature of 800-900℃ to remove the carbon carrier and polymer; Then the residue was dissolved by aqua regia to form a Pt-ion-containing solution in which 8 mL of 37% HCl, 10 mL of 20% NH_4Cl, and 5 mL of 20% $SnCl_2$ hydrochloric acid solution (the volume ratio of HCl to water is 1:1) were added. Finally, a spectrophotometer (TU-1900/TU-1901, Beijing Puxi, China) with a double beam of lights served to determine the Pt content by detecting the absorbency of the platinum-containing solution.

(5) **Fabrication of Nafion-bonded commercial 40%Pt/C catalyst electrode:** The Nafion-bonded commercial 40%Pt/C catalyst (Jonhson-Matthey In. UK) electrode was prepared as described previously.[2] In short, the Nafion-bonded electrode was composed of a gas diffusion layer and a catalyst layer. The gas diffusion layer was prepared on wet-proofed carbon paper. The carbon powder (Vulcan XC-72, Cabot Corp.), 30 wt % PTFE, and ethanol were ultrasonically mixed with a ratio 3:1 of carbon to solid PTFE content. The viscous mixture was coated onto carbon paper (Tony Co, Jap.) wet-proofed by PTFE and then heated at 340℃ for 30 min. A

suspension consisting of catalysts 40 wt % Pt/C, 5 wt % Nafion solution (DuPont), and anhydrous alcohol was first ultrasonically mixed for about 15 min. The ratio of Pt/C loading to solid Nafion was maintained at 3:1. The suspension was pipetted onto the gas diffusion layer and finally heated at 145℃.

(6) **MEA fabrication and unit cell operation:** The MEA with the AIEE anode (0.014 mg Pt cm^{-2}) and the Nafion-bonded Pt/C cathode (0.3 mg Pt cm^{-2}) was fabricated by hot pressing a sandwich of a AIEE anode, a Nafion 112 membrane (DuPont), and an Nafion-bonded Pt/C cathode in series at 135℃ and 5 MPa for 2 min. The Nafion 112 membrane (Du Pont) was pretreated with 3vol % H_2O_2 and 0.5 M H_2SO_4 for 1 h to remove impurities. The membrane was then washed several times with hot ultrapure water.

The test of a single cell was conducted on the Fuel Cell Test Station (Fuel Cell Technologies, Inc.). Pure hydrogen and oxygen were fed as the fuel and the oxidant, respectively. The Electrochemical polarization behavior were obtained 70℃ and pressures of 0.2MPa on both sides. The gas flow rates of Pure hydrogen and oxygen were fed at rate of 200 ml min^{-1}. The gas humidification temperatures were 5℃ lower than the cell temperature for hydrogen and oxygen, respectively.

Reference

1 S. X. Cai, C. Huang. Noble metal analysis, first edn., Metallurgical Industry Press, Beijing, 1984, pp. 199.

2 Z. D. Wei, S. G. Chen, Y. Liu, C. X. Sun, Z. G. Shao, P. K. Shen, *J. Phys. Chem. C*, 111, 2007, 15456.