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Pt-nanoflower as high performance electrocatalyst for fuel cell vehicle

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ABSTRACT

The development of low platinum catalysts for automotive fuel cells is a way to solve platinum resource constraints. We successfully prepared Pt-nanoflower supported on carbon black in aqueous solution with the aid of Cetyltrimethyl Ammonium Bromide (CTAB). Those nanoflowers showed uniform particle size distribution with average particle size of 27.0 nm. XRD indicated lattice compression for Pt from as-synthesized Pt/C. 71.6% of Pt existed in the form of elementary substance based on XPS. All those assembly nanostructure contributed to remarkable area-specific and mass-specific activities in oxygen reduction reaction.

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Introduction

The development of new energy vehicles is an important way to ease the fossil energy crisis, fuel cell vehicle is an important one of new energy vehicles. Proton exchange membrane fuel cell (PEMFC) is one of the most promising cells used for automobile power supply owing to its high efficiency in energy conversion, low emission of pollutants, low operating temperature and speed in start-up [1–3]. However, the poor stability and high cost in electrochemical environment hinder the practical application of PEMFCs on large scale. As one of the most important components of PEMFC, catalyst is the key to complete the electrode reaction and the high cost and

scarcity of noble metal catalysts have precluded the commercialization of PEMFC [4–7]. In order to reduce/replace noble metal (e.g. Pt)–based catalysts, much research work has focused on the effort to improve the activity and durability of the catalysts, to decrease cost of the catalysts, to optimize the fabrication of catalysts, to develop novel nonprecious metal catalysts and metal-free heteroatom-doped carbons as efficient catalysts for oxygen reduction reaction (ORR) in PEMFC [8–10]. Ghasemi et al. [11] have synthesized carbon nanotube polypyrrole (CNT/PPy) nanocomposites in situ via chemical oxidation and found that the nanocomposite structure of this catalyst made it a suitable candidate for ORR. Qi et al. [12] have developed a facile chemical route to the synthesis of Rh–Pd alloy nanodendrites with dense and straight arms and found

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that this Rh–Pd alloy nanodendrite exhibited extremely higher durability than commercial Pt/C for ORR owing to the incorporation of Rh with high resistance against base and acid etching. Zhang et al. [13] have synthesized three-dimensional porous graphitic carbon networks codoped with nitrogen and phosphorus and found that the resultant metal-free carbon catalysts exhibited remarkably high bifunctional electrocatalytic activities with an excellent durability for both hydrogen evolution and oxygen reduction reactions. However, platinum is remaining the most promising single-metal oxygen reduction reaction catalyst and widely used nowadays and development of low platinum catalyst is the main way to reduce the amount of platinum [14–16]. Besides, the fabrication of platinum nanoparticles with a narrow shape distribution is attractive in developing highly active platinum catalysts in the development of PEMFCs [17,18]. Up to now, nanostructured platinum with various morphologies has been extensively exploited to improve the electrocatalytic performance and long-time stability [19–21]. Wang et al. [22] have demonstrated a general synthetic approach to monodisperse platinum nanoparticles with sizes tunable from 3 nm to 7 nm and controlled polyhedral, truncated cubic, or cubic shapes. Lim et al. [23] have prepared Pd–Pt bimetallic nanodendrites and found that the Pd–Pt nanodendrites exhibited a higher surface area and much enhanced activity for both the oxygen reduction and formic acid oxidation reactions as compared to the foam-like Pt aggregates. Ruan et al. [24] have developed an ultrathin Pt nanowire network structure with a high density of twin planes as an efficient electrocatalyst, which exhibits a higher electrochemical surface area and much improved activity in ORR compared with the commercial Pt/C electrocatalysts.

In this paper, we successfully prepared Pt-nanoflower supported on carbon black in aqueous solution with the aid of Cetyltrimethyl Ammonium Bromide (CTAB) and its physical and electrochemical performances were investigated by means of various characterization.

Experimental

Sample preparation

The Pt nanoparticles were prepared and supported on Vulcan XC-72 as follows. The mixture of 60.0 mg L-ascorbic acid (AA, 99.7 wt%, Shanghai Chemical Reagents Limited by Share Ltd.), 450.0 mg cetyltrimethyl ammonium bromide (CTAB, 98.0 wt%, Tianjin Boya Chemical Limited by Share Ltd.), 60 mL deionized water and 1300 μ L potassium tetrachloroplatinate (II) (K_2PtCl_4 , Shenyang Research Institute of Nonferrous Metal) aqueous solution (50.0 mM) were transferred into an oil bath of 100 °C and remained at 100 °C under protection of N_2 for 5.0 h. The Pt nanoparticles mentioned above formed gradually. Then, the solution containing Pt nanoparticles was allowed to be cooled to room temperature, and 50 mg Vulcan XC-72 dispersed in 5 mL deionized water was added into the solution above. After being stirred for 24 h, the resulted solution was separated by centrifuge. The Pt/C particles was washed with water-ethanol mixture (3:1 in volume ratio) six times. Finally, the obtained Pt/C nanoparticles were dried under vacuum for 12 h.

Physical and electrochemical characterizations

High-resolution transmission electron microscopy (HRTEM) images were taken using a JEOL JEM-2100F microscope operated at 200 kV and equipped with an Oxford X-Max detector. X-ray powder diffraction (XRD) analysis was carried out with a PANalytical X'Pert Pro Super diffractometer using Cu K α radiation ($\lambda = 0.154178$ nm). X-ray photoelectron spectra (XPS) measurement was conducted using Thermo Multi-Lab 2000 spectrometer with Al K α X-ray (1486.6 eV) as the light source.

The electrochemical performance studies were conducted at CHI730 electrochemical working station with a three-electrode system using Pt foil and saturated calomel electrode (SCE) as the counter electrode and reference electrode, respectively. All electrode potentials were given versus reversible hydrogen electrode (RHE). Prior to half-cell test, the electrocatalyst was cycled between 0.05 and 1.20 V for 20 cycles in 0.1 M $HClO_4$ aqueous solution saturated with N_2 at a scan rate of 100 $mV s^{-1}$ to clean the surface of the electrocatalyst. The half-cell accelerated degradation test (ADT) was conducted in N_2 -purged 0.1 M $HClO_4$ aqueous solution by linear potential cycling between 0.6 and 1.2 V for 1500 cycles at a scan rate of 50 $mV s^{-1}$. Moreover, the CV and polarization curves were recorded before and ADT. The cyclic voltammetry curves were recorded in N_2 -purged 0.1 M $HClO_4$ aqueous solution at a scan rate of 50 $mV s^{-1}$. The oxygen reduction reaction (ORR) polarization curves were recorded positively at a sweep rate of 10 $mV s^{-1}$ in O_2 -purged 0.1 M $HClO_4$ aqueous solution at 1600 rpm.

Results and discussion

The morphology of Pt-nanoflower supported on carbon black was investigated as shown in Fig. 1. From Fig. 1, we can see that the Pt nanoparticles highly dispersed on Vulcan XC-72 and existed mainly in the form of nanoflowers, which demonstrated that Pt-nanoflower supported on carbon black was successfully fabricated in aqueous solution with the aid of Cetyltrimethyl Ammonium Bromide (CTAB). Moreover, there was no obvious phenomenon of agglomeration between nanoflowers. Besides, the size distribution histogram and statistical results of Pt-nanoflower were obtained and given in Fig. 2 by analyzing more than 200 nanoparticles. As shown, the average size was used to describe the central tendency and the standard deviation was calculated to describe the particle-size distribution in different samples. According to Fig. 2, the size distribution of Pt-nanoflower was narrow and the particle size was uniform with the average particle size of 27.0 nm. The crystalline structure of Pt-nanoflower/C was investigated by XRD and shown in Fig. 3. As indicated, the diffraction peaks of the Pt from Pt-nanoflower/C shifted towards higher degree and became weaker compared with that of bulk Pt from the PDF card (JCPDF#04-0802). This phenomenon suggested that the crystal structure of Pt from Pt-nanoflower/C experienced lattice compression [25].

The composition and chemical states on the surface of Pt-nanoflower/C were determined by XPS analysis in Fig. 4. These spectra were calibrated against the major carbon peak at the

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