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Nanostructure catalysts prepared by multi-sputtering deposition process for enhanced methanol electrooxidation reaction

Kyung-Won Park[a,*], Young-Woo Lee[a], Yung-Eun Sung[b,**]

[a] Department of Chemical Engineering, Soongsil University, Seoul 156743, Republic of Korea
[b] World Class University (WCU) Program of Chemical Convergence for Energy & Environment (C_2E_2), School of Chemical and Biological Engineering, College of Engineering, Seoul National University (SNU), Seoul 151742, Republic of Korea

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ABSTRACT

We report size and/or composition-controlled nanostructure catalysts for direct methanol fuel cells fabricated by means of multi-sputtering deposition process. The size-controlled Pt nanophases ranging from 0.62 ± 0.14 nm to 3.07 ± 0.34 nm in average size with narrow size distribution are fabricated as a function of sputtering power ratio of targets. The composition-controlled PtRu alloy nanophases deposited at different RF power ratio of Pt and Ru targets with the same power of WO_3 target exhibit varied atomic percentages of PtRu, i.e. (79.8:21.2), (62.2:37.8) and (37.6:62.4). Furthermore, the PtRu nanophases deposited at a constant RF power ratio of Pt and Ru targets with different power of WO_3 target show controlled average sizes such as 3.62 ± 0.15 nm, 1.86 ± 0.23 nm, and 1.20 ± 0.22 nm with the same compositions. The specific maximum power density of the nanostructure catalyst ($\sim 100\ W\ g^{-1}$) is superior to that of the conventional nanostructure catalyst ($\sim 37\ W\ g^{-1}$), representing that the nanostructure catalyst has an excellent electrode structure for methanol electrooxidation as compared to the conventional catalyst.

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1. Introduction

Electronic, chemical, magnetic, and optical properties of nanostructured materials are extremely different from those of bulk materials depending on size, shape, and composition [1–4]. In particular, since catalytic reaction process depends on the size of nanoparticles, synthesis of well size-controlled nanoparticles is crucial for effective catalysis study and catalysts for use in fuel cells [5–7]. Many studies on colloidal bulk particles have focused on the control of particle sizes and have been related to catalytic activity as a function of catalyst size [8,9].

Recently, the miniaturization of polymer electrolyte membrane fuel cells as a power source for small digital devices to microelectromechanical systems has been the subject of intense study [10–12]. The power sources for these applications should be small-sized for on chip or integration as well as have sufficiently available capability to operate the intended devices. Accordingly, electrodes for such miniaturized fuel cells should be considered and fabricated from point of view in both nanostructures for catalysis and thin-film formation for integration with other devices. The electrodes for conventional direct methanol fuel cells (DMFCs) based on bulk properties of catalysts commonly consist of more than two phases such as nanosized noble metals i.e. Pt, PtRu and porous materials i.e. carbon or porous oxides as an assistant or support [13–18].

However, conventional physical deposition methods have difficulty in providing the two-phase electrodes containing nanometallic phases as catalysts and porous oxides as supports, besides, possessing individual properties of each material. The conventional sputtering techniques with one sputtering target of a two phase mixed material or small chips of 2nd phase on the target of 1st phase might not be utilized to form size-controlled thin-film electrodes with various compositions keeping the individual properties of each material. Herein, we suggest multi-sputtering deposition (MSD) method with individual gun of metal and oxide targets in order to fabricate and design nanostructure catalysts containing size-controlled Pt or PtRu nanophases in porous material.

The size distribution of the catalysts was characterized by field-emission transmission electron microscopy (FE-TEM) Energy dispersive X-ray (EDX) analysis of the catalysts was performed on a FE-TEM (Tecnai G2 F30 system). The relative composition of Pt and Ru present in the catalysts was determined by Rutherford backscattering spectrometry (RBS). To evaluate the electrochemical properties of the catalysts, cyclic voltammograms (CVs) and current-potential curves were obtained using a three-electrode electrochemical system. The performance of the unit cell was evaluated using a computer-controlled electronic load.

* Corresponding author. Tel.: +82 2 820 0613; fax: +82 2 812 5378.
** Corresponding author.
E-mail addresses: kwpark@ssu.ac.kr (K.-W. Park), ysung@snu.ac.kr (Y.-E. Sung).

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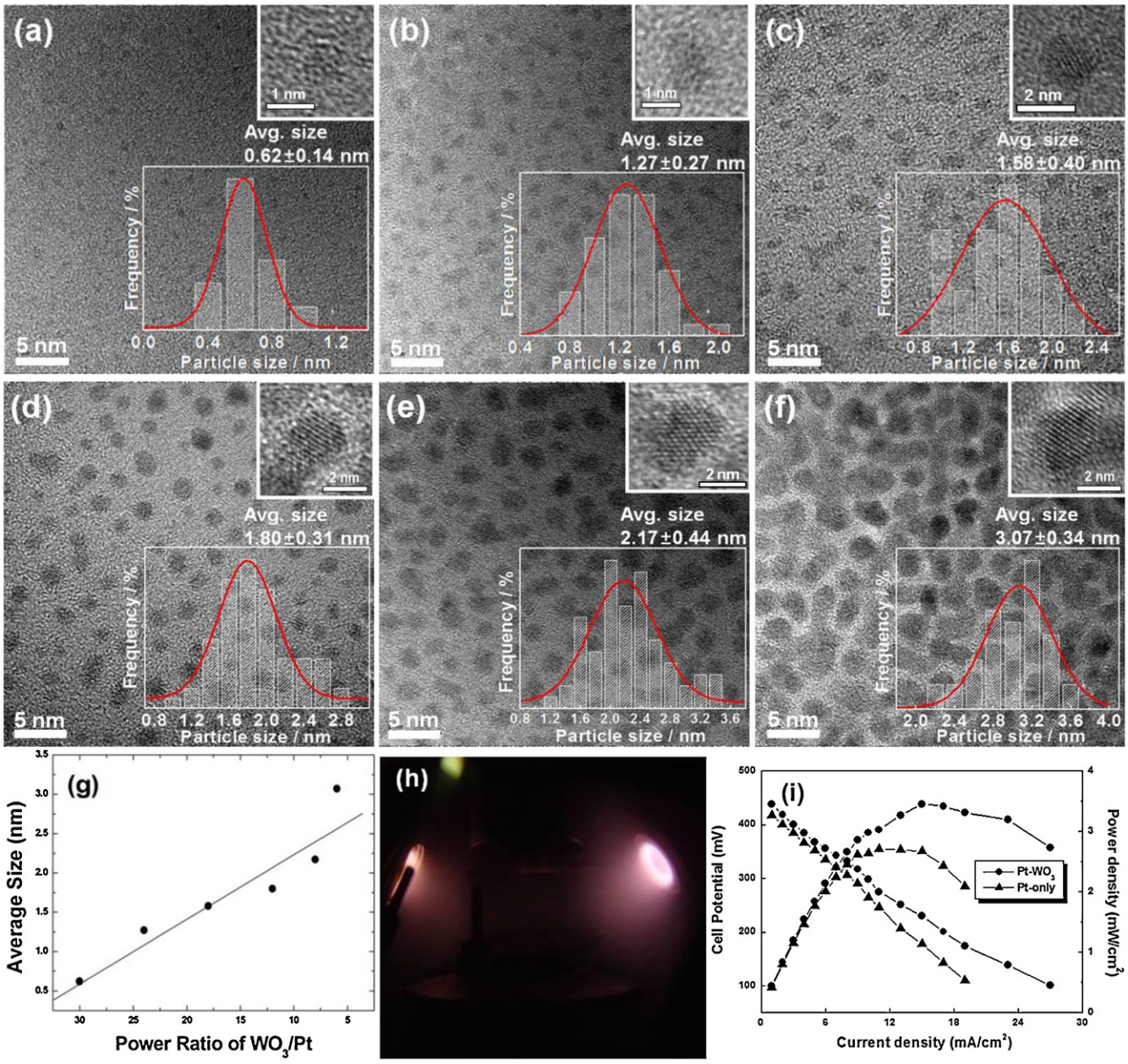

Fig. 1. TEM Images and size distributions of Pt–WO_3 nanostructure catalysts prepared as a function of power ratio (WO_3/Pt) of (a) 30, (b) 24, (c) 18, (d) 12, (e) 8, (f) 6 in the MSD system. (g) A plot of average size of Pt nanophases versus sputtering power ratio of targets. (h) Photo image of actual deposition process in the MSD system to fabricate the nanostructure catalysts. (i) Characteristic curves of DMFCs measured at 25 °C using Pt–WO_3 and Pt-only as an anode and Pt black powders as cathode catalysts.

2. Experimental

2.1. Fabrication of Pt nanostructure catalysts

Pt nanostructure catalysts were grown using radio frequency (RF) magnetron sputtering system with multi-sputtering targets. Indium tin oxides-coated transparent glasses (ITO, Samsung Corning Co., Ltd.) were used as substrate. Pt and WO_3 were used as the target materials. The base pressure was less than 5×10^{-6} Torr and working pressure was 1.1×10^{-2} Torr for all examined. Sputtering was carried out under Ar gas atmosphere at 30 SCCM at room temperature. The samples were sputter-deposited for 2 min at the RF powers of 10 W and 60–300 W of Pt and WO_3 target, respectively.

2.2. Fabrication of PtRu nanostructure catalysts

PtRu nanostructure catalysts were also prepared using RF magnetron sputtering system under an atmosphere of inert Ar gas at room temperature. In order to control PtRu alloy nanophases, the RF power of the Pt, Ru, and WO_3 sputtering targets were individually manipulated. For electrochemical analysis, the ITO-coated transparent glasses and teflonized carbon paper (Toray, TGPH-090) were used as substrates. The different composition of the same-sized PtRu alloy nanophases was modulated by the RF power of metal targets at constant powers of WO_3 target. The size of PtRu alloy nanophases with the same compositions was controlled by the RF power of WO_3 target at constant powers of metal targets.

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