Two-step Microwave Synthesis of Highly Dispersed Ordered Intermetallic PtPb Nanoparticles on Carbon Black

Arockiam John Jeevagan[1], Naoyuki Sawano[1], Govindachetty Saravanan[2], Takao Gunji[1], Shou Usui[1], Taiki Kojima[1], Shingo Kaneko[3], Genki Kobayashi[1], Futoshi Matsumoto[1]

[1]Department of Material and Life Chemistry, Kanagawa University, 3-27-1, Rokkakubashi, Kanagawa, Yokohama 221-8686, Japan
[2]CSIR-National Environmental Engineering Research Institute, Nehru Marg, NAGPUR-440020, India.
[3]Research Institute for Engineering, Kanagawa University, 3-27-1, Rokkakubashi, Kanagawa-ku, Yokohama-shi, Kanagawa 221-8686, Japan

1. Introduction

In the research and development of polymer electrolyte fuel cells (PEFCs), one of the challenges is to design the better alternatives to the state-of-the-art Pt catalyst as anode as well as cathode catalysts in PEFCs which has higher power density at room temperature.[1] The catalytic activities strongly depend on the parameters such as particle size and dispersion of the catalysts because the reactions occur on the catalyst surfaces. The catalytic activity can be improved by converting of nanocrystalline Pt into their alloys or intermetallic compounds using low-temperature nanoparticles (Nps) synthesis protocol. Hence, the objective of the present study is to demonstrate that two-steps microwave method can prepare highly dispersed PtPb ordered intermetallic Nps on carbon black (PtPb Nps/CB) which showed significant catalytic activity towards formic acid (FA) oxidation.

2. Experimental

The two-step procedure for the synthesis of PtPb Nps/CB is as follows. In the first step, H_2PtCl_6 (0.01 mmol) and carbon black (0.040 g) were mixed in 50 mL ethylene Glycol. Potassium hydroxide (0.04 mmol) was added to the mixture.[2] The mixture was then sonicated in a bath-type ultrasonicator until the color turned from yellow to dark brown. The mixture was treated in the flask with a reflux set for 5 min under a microwave (Focused microwave instrument, CEM) power of 100 W. After cooling, the Pt Nps/CB was collected by centrifugation, washed with ethanol, acetone and water and finally dried at 120℃. In the second, synthesized Pt/CB (0.040 g) and $Pb(CH_3COO)_2$ (0.019 g) were mixed in 50 mL ethylene Glycol. The mixture was treated in the flask with a reflux set for 5 min under a microwave power of 100, 200 and 300 W. A reference was prepared based on the Bauer's experiment.[3]

3. Results and Discussion

Figure. 1 shows the TEM images obtained for Pt Nps/CB (Fig. 1a), PtPb ordered intermetallic Nps/CB (Fig. 1b) and PtPb/CB prepared with heat-treatment of Pt/CB (Fig. 1c). Pt Nps/CB and PtPb Nps/CB shows a uniform distribution of dark spots on CB and the average particle size was calculated as 1.1 and 1.9 nm, respectively. This indicates that the PtPb ordered intermetallic compound and the reaction of Pt Nps having the form of hemisphere with the diameter of 1.1 nm with Pb^{2+} to prepare the PtPb ordered intermetallic compound. Figure. 2 shows *p*XRD profiles for PtPb Nps/CB (a-c), Pt/CB (d) and CB (e). All *p*XRD profile in Fig. 2 shows two strong peaks at 25.0 and 43.5° that correspond to the 002 and 101 reflection of carbon (e), respectively. The formation of PtPb ordered intermetallic phase on CB cannot be confirmed with the *p*XRD pattern due to the peaks are unclear around 50-80 °.

The electrocatalytic activity of PtPb ordered intermetallic Nps/CB towards FA would be discussed in the presentation.

4. References

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3. J.C. Bauer, X. Chen, Q. Liu, T.H. Phan, R.E. Schaak, *J. Mater. Chem.*, 18, 275 (2008).

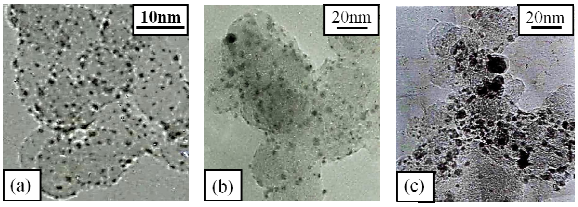

Fig. 1 TEM images of (a) Pt/CB and (b and c) PtPb ordered intermetallic Nps/CB prepared with one- (a) and two- (b) steps microwave methods, respectively. The sample (c) was prepared by heat-treating Pt/CB and Pb^{2+} ions in ethylene glycol at 190℃ for 4 h.

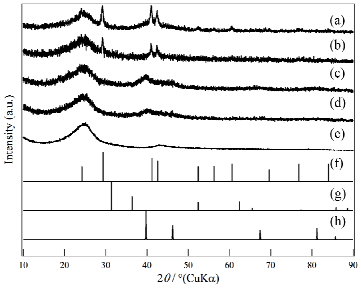

Fig. 2 *p*XRD patterns of PtPb Nps/CB (a(100 W), b (200 W) and c (300 W)). Pt/CB (d) and CB (e). The solid lines (f-h) at the bottom are simulated *p*XRD peaks for ordered intermetallic compound of PtPb(f), Pb(g) and Pt(h).