Synthesis of Nano-size Platinum Particles by Proton Irradiation

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1. Introduction

Recently, the preparation and characterization of nanoparticles have become a noticeable topic because of their peculiar optical, electronic, magnetic and catalytic properties.[1] The peculiar property of nanoparticles is strongly dependent on the shape, size and distribution of particle.[2] Platinum(Pt) nanoparticles are usually used in proton exchange membrane fuel cells (PEMFC) as cathode electrocatalysts for oxygen reduction reactions in relatively low temperature.[3] In general, nanoparticles can be synthesized such as, photochemical, chemical reduction and radiation chemical reduction.[4] However, chemical reduction often can't provide suitable control of particle shape and size.[2]

In this study, we investigated a novel method to obtain of Pt nanoparticle by using proton beam irradiation in Pt aqueous solution. In addition experiments, we studied on the effect of surfactant and beam current as well as beam energy which could control to the size and degree of dispersion of particles in the solution.

2. Experiments

The Pt aqueous solution was irradiated by proton beam at the atmospheric pressure and room temperature to synthesize Pt nanoparticles. Fig. 1 shows the photograph of proton beam irradiation experimental equipments.

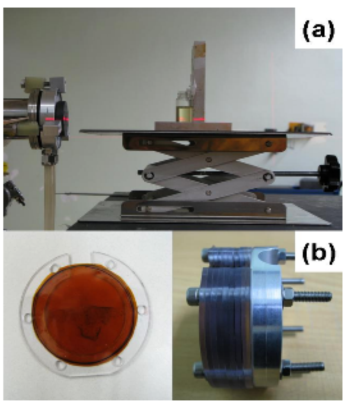

Fig. 1. The photograph of proton beam irradiation experimental equipments. (a) Proton irradiation experimental equipments, (b) Slice sample tool and cell

The experiments were as follows ; (i) The 45MeV Proton beam of 100 nA and 1 μA beam current were irradiated to Pt aqueous solution in order to observe a particle size with relation to beam current. (ii) After proton beam irradiation, the surfactant of 5×10^{-4} mol CTAB was dissolved in reaction solution to prevent particle aggregation. (iii) The other experiment used the slice sample tool in order to observe the size of particle in relation to energy. Primary incident beam lost energy when the beam was passed via cell. Such as, the 1st 44MeV, 2nd cell 38MeV, 3rd cell 34.3MeV etc. The slice sample tool was composed of 8 cells. The cell was made of 2 mm thickness PE disk which has a hole of 50 mm diameter, and 75 μm thickness Kapton film was attached to the both sides of the cell as windows. All experiments were supplied enough irradiation time for completely reaction. Table 1. shows summary on samples preparation condition.

Table. 1. Summary on sample preparation condition.

sample	Pt aqueous solution	Irradiation time	Irradiation condition
1	170 mℓ	1´ 20˝	45MeV/100 nA
2	150 mℓ	25"	45MeV/1 μA
3	Reaction solution + CTAB	5´ 00˝	45MeV/100 nA
4	Slice sample tool	40"	45MeV/100 nA

The particle morphology, size and distribution of Pt nanoparticles were measured by transmission electron microscopy (TEM) using a JEOL-JEM 2200FS operating at 200 kV. Samples were prepared by drying a drop of the reaction solution on a TEM grid. Subsequently, the sample was dry completely at room temperature.

3. Results and Discussion

2-1. Effect of Beam current

Fig. 2 shows two TEM images by irradiation beam current 100 nA, 10 μA, respectively (sample 1, 2 in Table 1). The Pt nanoparticles of average 2.6 nm size were synthesized in sample 1 and were synthesized Pt nanoparticles of average 5.9 nm size in sample 2. The size of Pt nanoparticles increased 2.6 nm to 5.9 nm when the beam current increases 100 nA to 1 μA.

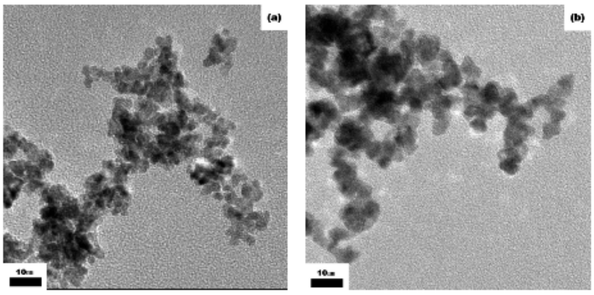

Fig. 2 TEM images of Pt nanoparticles : (a) beam current 100 nA, (b) beam current 1 μA

2-2. Effect of Surfactant

After irradiation proton beam, the surfactant, CTAB was dissolved in the reaction solution. Fig. 3 shows TEM images of sample 3 in table 1. The sample 3 was not aggregated Pt nanoparticles than the other sample. The surfactant might influence to aggregation of Pt nanoparticles, because the surfactant preferentially aggregated with Pt nanoparticles before Pt nanoparticles aggregation.

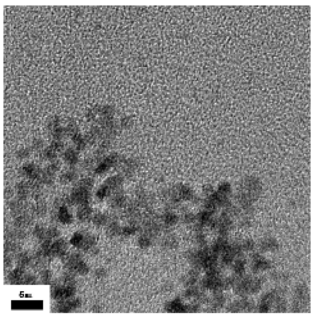

Fig. 3. TEM image of Pt nanoparticles ; surfactant

2-2. Effect of Beam energy

Fig. 4 shows TEM images of Pt nanoparticles that were synthesized by using slice sample tool. The Pt nanoparticles were observed to similar size in all cells. However, Pt nanoparticles of the 5th cell were not aggregated when the reaction solution of all cell kept a few days. The specific energy might influence to aggregation of Pt nanoparticles. Table. 2 shows the energy and particle size in each cell when Pt nanoparticles were synthesized by using slice sample tool.

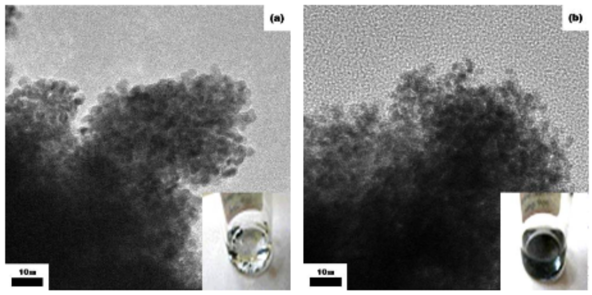

Fig. 3. TEM image and photograph of Pt nanoparticles ; (a) 1^{st} cell, (b) 5^{th} cell

Table. 2. The energy and particle size in all cells

cell	1^{st} cell	2^{nd} cell	3^{rd} cell	4^{th} cell	5^{th} cell	6^{th} cell	7^{th} cell	8^{th} cell
Energy (MeV)	42	38	34.3	30.4	26	20.9	14.4	2.4
Average size(nm)	3.4	3.1	2.9	2.65	2.1	4.1	2.4	2.7

3. Conclusions

The Pt nanoparticles were successfully prepared by using proton beam irradiation in Pt aqueous solution. The size of Pt nanoparticles increased, according to increase of beam current. The surfactant and specific beam energy influenced to aggregation of Pt nanoparticles. In this study, we could confirm the effect of the beam current, energy and surfactant. The beam current could control the size of Pt nanoparticles. The surfactant as well as specific beam energy was effective in prevention of aggregation of Pt nanoparticles. We will study about control of the size and degree of dispersion of Pt nanparticles in order to obtain optimum parameters by proton beam irradiation.

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