MEA preparation of Fe-N/C catalyst synthesized by E-beam irradiation and Effect of acid treatment

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1. Introduction

A polymer electrolyte membrane fuel cells (PEMFC) uses a polymer membrane having a hydrogen ion exchange specification as an electrolyte, and has a lower operating temperature of 60-80℃ than other types of fuel cells, thus maintaining high efficiency and current density [1,2]. In addition, since it can be miniaturized to make a lightweight battery, it can be applied to power sources of transportation and installed power generation.

To increase the reaction rate of the fuel cell and increase the energy conversion efficiency, catalysts are used on the surfaces of the cathode and anode of the fuel cell. Due to its high activity, platinum catalysts are mainly used, but due to the high price due to the scarcity of platinum catalysts, research on platinum alternative hydrogen fuel cell catalysts has been actively conducted [3,4].

Various methods can be applied to the manufacture of catalysts, but it is efficient to mass-produce catalysts through continuous irradiation of electron beams in the manufacture of catalysts for commercialization. An electrode was produced to confirm the performance of the produced catalyst, and the change before and after acid treatment was confirmed by performance measurement to compare the effect of impurities formed during catalyst synthesis on the catalyst.

2. Methods and Results

2.1 Catalysts synthesis with E-beam irradiation and MEA synthesis

The catalyst was synthesized by electron beam irradiation. Iron acetate and Fe-chloride were used as Fe precursors, and 1,10-phenanthroline were used as nitrogen precursors. In the case of the ^{57}Fe precursor of the catalyst, ^{57}Fe metal was dissolved in an HCl solution to prepare Fe-chloride in advance. Carbon black was used as a carbon support for dispersing Fe-N_x moieties. The Fe-N/C catalyst was synthesized by irradiating an electron beam at an intensity of 10 MeV, 80 kGy for 3 minutes, dispersing the solution with mortar and heat-treating the solution with Ar atmosphere at 800℃ for 1 hour. To compare the changes before and after the acid treatment of the catalyst, acid treatment was performed on 1M HNO3 in an environment of 90 degrees for 1 hour.

The membrane electrode assembly(MEA)he catalyst was prepared in the following manner. First, a catalyst ink of anode and anode was made. Fe-N/C and Pt/C catalysts were dispersed solution dispersion of n-propanol and ionomer in DI water, respectively. Thereafter, Cathode ink was bar-coated with membrane and anode ink was coated on PET film with the ultrasonic spray technique. The dried coating film was hot pressed to manufacture an electrode. To compare the electrochemical properties of the catalyst, a unit cell corresponding to only a pole in which an oxygen reduction reaction(ORR) occurs was manufactured. First, the Fe-N/C catalyst was mixed with IPA and Water mixture, and a Nafion solution was added. After that, it was sonicated in a bath-type sonicator, and ink was dropped on a glassy carbon with a diameter of 5 mm and dried.

2.2 Catalysts Characterization

Carbon support dispersion of Fe-N_x was confirmed by using TEM and EDS mapping to confirm the chemical characteristics of the prepared catalyst before acid treatment. In addition, as a result of measuring the contents of iron and nitrogen through ICP and CHNS measurements, it was confirmed that 1.38~1.5 wt.% and 1.18~1.7 at.% of iron and nitrogen were contained, respectively. The chemical characteristics of the catalyst after acid treatment were measured through XRD (X-ray Diffraction) and CHNS, and it may be verified that impurities generated during the synthesis process were removed from the catalyst after acid treatment than before acid treatment (Fig.1) The relative content of nitrogen after acid treatment also increased compared to before acid treatment.

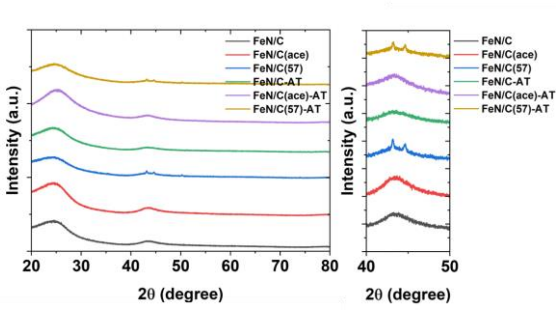

Fig. 1. X-ray diffraction of Fe-N/C and ^{57}Fe-N/C catalysts

2.3 Electrochemical analysis

To compare the performance of the catalysts, Cyclic voltammetry(CV) and Linear sweep voltammetry(LSV) measurements were performed by manufacturing the catalyst as a unit cell, and the performance of the catalyst before and after acid treatment was measured and compared, respectively. The CV is a method of analyzing the characteristics of an electrochemical reaction of an electrode surface or a chemical material occurring near the electrode surface by measuring a current after changing the potential to a working electrode. cv was measured at a scan rate of 20 mV/s at O2-saturated. The LSV is a method of measuring a current-potential curve by changing the potential of a working electrode at a constant speed in a negative or positive direction from an initial potential, and measurement was performed by setting the scan rate to 10 mV/s. As a result of measuring each electrochemical characteristic (Fig.2), Fe-N/C performance was superior than ^{57}Fe-N/C. It was confirmed that ECSA, initiation potential, and half-wave potential after acid treatment decreased compared to before acid treatment.

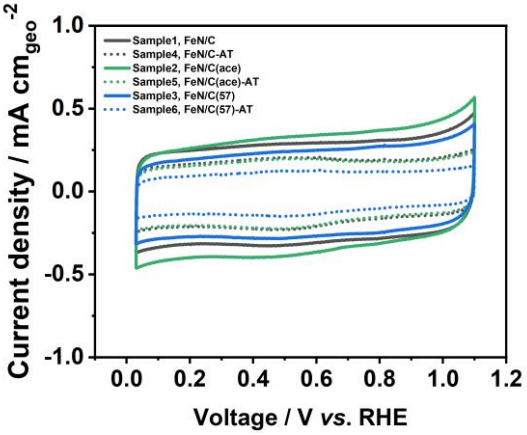

Fig. 2. Cyclic Voltammogram of Fe-N/C and ^{57}Fe-N/C

3. Conclusions

After synthesizing a platinum replacement Fe-N/C catalyst of PEMFC by electron beam irradiation, the characteristics of the catalyst before and after Membrane Electrode Assembly preparation and acid treatment were studied. It was confirmed that the catalyst was successfully synthesized by electron beam irradiation with TEM and XRD, but the formation of some impurities, such as oxides, was also confirmed. A battery was produced using the produced catalyst, and the electrochemical properties of the single cell were measured to compare the catalyst ORR before and after the acid treatment, and the performance was reduced after the acid treatment of the catalyst. This indicates that impurities formed during the catalyst synthesis process may lead to inaccurate results in measuring the performance of the catalyst. It seems that the decrease in catalyst performance after acid treatment can be compensated by increasing the amount of catalyst ink loaded on the battery.

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