

Cross Section Mapping of MEA by SEM, and Top Surface Analysis of Catalyst Powder by LEIS

In this study, we introduce the analysis cases of the following two points as the evaluation item about degradations and characteristics of PEFC (Polymer Electrolyte Fuel Cells).

- Cross section elemental mapping analysis by SEM-EDX (Scanning Electron Microscope – Energy Dispersive X-ray spectroscopy) about the MEA (Membrane Electrode Assembly)
- Elemental analysis of the top surface of the catalyst powder by LEIS (Low Energy Ion Scattering).

Cross section elemental mapping analysis by SEM-EDX

Electrolyte membrane and ionomer : Nafion®

Catalyst powder : Pt/C (anode and cathode)

Coating method of the catalyst ink : Spray method

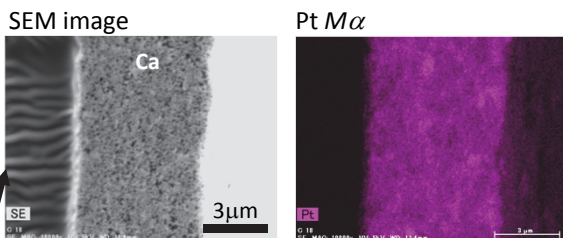
Operating condition : "A. Conditioning operation", "B. Start/Stop operation", "C. Load response operation"

Sawtooth-shaped potential cycle of between 1.0 V and 1.5 V (2 s/cycle)

Square-shaped potential cycle of between 0.6V and 1.0V (6 s/cycle)

● Pt mapping image of the cathode

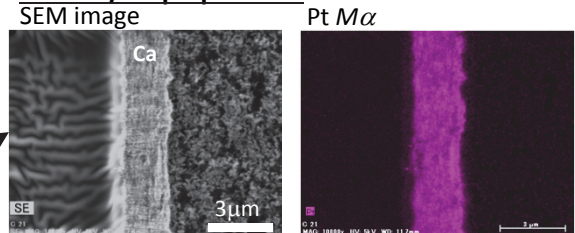
A. Conditioning operation



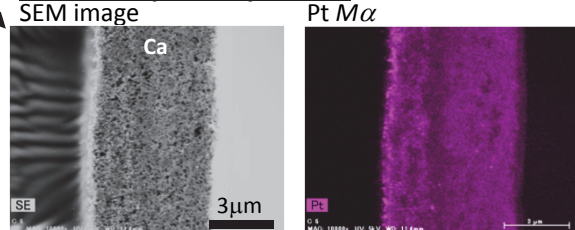
Electrolyte membrane

- For "A. Conditioning operation", the thickness of the Ca catalyst layer is about 6 μm. Pt distributes in the Ca catalyst layer uniformly.
- For "B. Start/Stop operation", the Ca catalyst layer becomes markedly thinner.
- For "C. Load response operation", At the Ca catalyst layer near the electrolyte membrane, Pt are aggregated.

B. Start/Stop operation

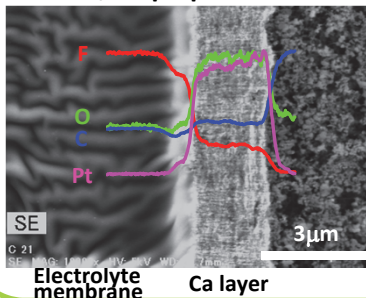


C. Load response operation

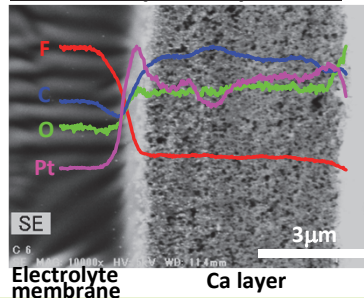


● Line profile of the cathode

B. Start/Stop operation



C. Load response operation



- For "B. Start/Stop operation", the concentration of F is varied at the interface between the Ca catalyst layer and the electrolyte membrane.

- For "C. Load response operation", the Pt concentration in the Ca catalyst layer is not constant. In addition, the higher Pt concentration region is observed at the interface between the Ca catalyst layer and the electrolyte membrane.

SEM-EDX analysis is the lower damage than EPMA analysis, and it is possible to analyze the morphological observation and the elemental distribution of the principal components in a few μm ~ a few 10 μm field size.

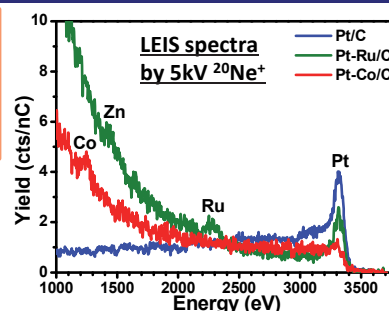
Elemental analysis about the top surface of the catalyst powder by LEIS

LEIS (Low energy ion scattering) :

LEIS spectrum is the energy spectrum of the scattered ions by irradiating the noble gas ion beam of low energy (a few 100 eV ~ a few keV) on the solid surface.

Main features of LEIS

- Qualitative and quantitative determination of the top surface
- Depth analysis up to about 10 nm
- Lower detection limit is a few 100 ppm ~ sub-%



Samples : Catalyst powders for PEFC

Pt/C, Pt-Ru/C, Pt-Co/C

- By the ⁴He⁺ irradiation ion species, it is also possible to obtain the information of the light elements.
- For the core-shell catalyst, it is possible to evaluate the covering state of the shell atoms.