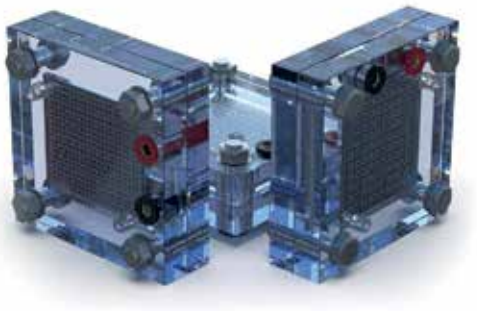
EA8000E

Application Note

HITACHI

EA8000E for the rapid quality control of hydrogen fuel cells



Introduction

In the race to reduce carbon emissions, fuel cells technology is developing rapidly. All types of fuel cells include three basic components: two electrodes (anode and cathode), and an electrolyte sandwiched between them. In the case of polymer electrolyte membrane (PEM) fuel cells, also called proton exchange membrane fuel cells, the electrodes are typically coated with conductive carbon mixed with platinum (Pt) particles. Pt acts as a catalyst for the continuous conversion of fuel (hydrogen) and oxidant (oxygen) into electricity.

In the fabrication process, it is critical to control the Pt coating uniformity on the electrode's surface to ensure efficient chemical reactions in the cell, while minimizing raw material costs and finished product waste.

Additionally to the Pt coating quality, the presence of iron (Fe) particle contaminants has a major impact on fuel cell operation. Iron particles can enter the production process through raw materials and wear and tear of process equipment. When present in the fuel cell, iron can react with hydrogen peroxide (H_2O_2) and form free radicals (Fenton reaction). This reaction can lead to the damage of the cell's ion-exchange membrane and the deterioration of the fuel cell.

Fenton reaction: $Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO\cdot + OH^-$

$H_2O_2 + HO\cdot \rightarrow HO_2\cdot + H_2O$

Conventional X-ray computerized tomography systems require several hours to detect small metal particles in membrane electrode assemblies, and while scanning electron microscopy can detect particles down to 10 µm in size, they only scan the surface of the sample.

Hitachi High-Tech have developed a new particle inspection technology that detects and analyzes metal contaminants down to 30 µm in as little as 15 minutes, greatly increasing testing throughput. The same technology can also determine the Pt distribution on the membrane.

Key Features

- Fast inspection method: typically 15 minutes per sample
- All-in-one: contaminants detection, location and elemental composition, plus Pt coating uniformity determination with one analyzer, for fast process decision-making and consistent product quality
- Analysis of contaminants down to 30 µm in size
- Simple sample preparation: insert the sample in the sample jig, and place on the analysis stage

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Membrane electrode assembly quality control made easy

Hitachi's EA8000E combines X-ray transmission technology, optical miscroscopy and energy-dispersive X-ray fluorescence (EDXRF) spectrometry to locate and characterize metal contaminants within membrane electrode assemblies (MEAs). EDXRF is also used to map the Pt distribution on the membrane.

While other techniques often only identify small particles when they're on the surface of the sample, the EA8000E uses unique, focused X-ray optics to locate and identify the elements in metal particles deeper within the sample.

Sample preparation is fast and easy: simply place the MEA (250 mm x 200 mm maximum size) in the sample jig, and sit the jig on the analysis stage to carry out the measurement. For a comprehensive analysis, the EA8000E measurement stages are:

1. Obtain an optical image of the sample using transmitted X-rays
2. Auto-detect the position of any contaminants using image processing
3. Observe morphology and automatically capture image of contaminated areas
4. Carry out elemental mapping of the contaminated areas and/or the whole membrane by EDXRF
5. Generate report

These steps can be fully automated, freeing the operator to perform other tasks and increasing productivity.

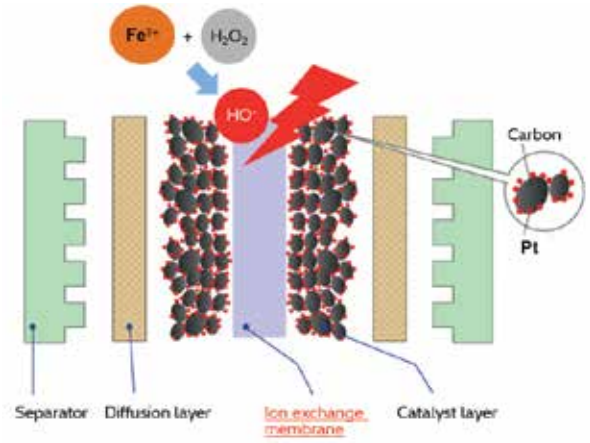

Figure 1: Fenton reaction in the fuel cell structure

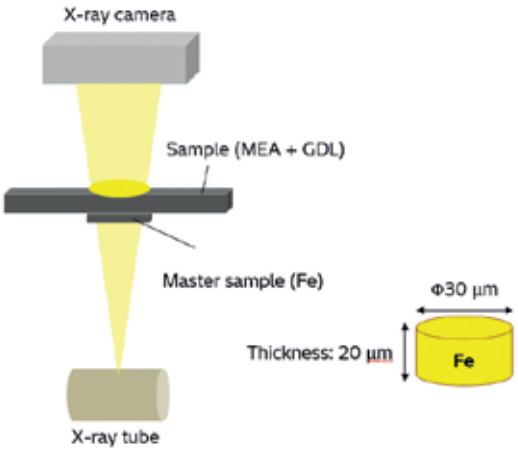

Figure 2: Test setup for iron particle detection

Performance and results

In this application note, the EA8000E was used to determine the number and composition of contaminants in a MEA that also included the gas diffusion layer (GDL). To do so, an iron particle of known size (diameter: 30 µm, thickness: 20 µm) was deposited under the membrane and gas diffusion layer to establish if the EA8000E could detect and identify it through the sample (see test setup in Figure 2).

Firstly, the analyzer detected the presence and the location of the particle under the material - it is clearly identified as a darker spot in the X-ray transmitted image (Figure 3). The EA8000E then automatically performed an EDXRF mapping of the contaminated area, which confirmed the nature of the particle in less than 40 seconds (Figures 4 and 5).

The EA8000E's ability to detect metal contaminants within the sample, not just on the surface, is a clear advantage, as it enables operators to better spot issues before products are moved down the production line and introduced on the market.

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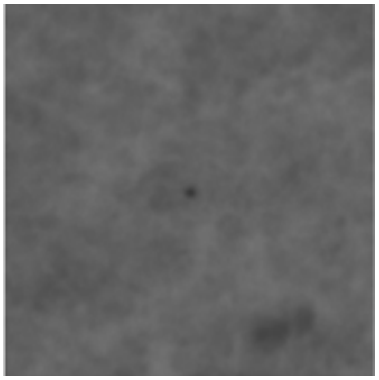

Figure 3: X-ray transmission image

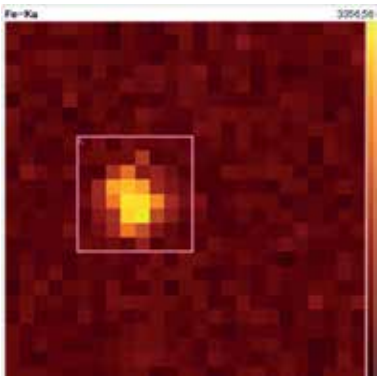

Figure 4: Fe Ka mapping image

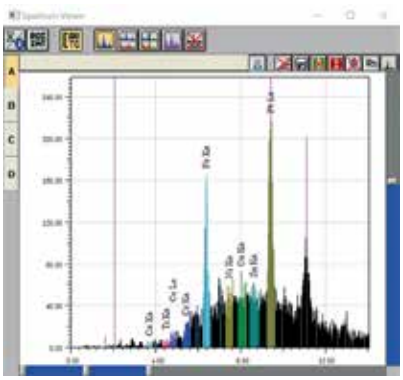

Figure 5: EDXRF spectrum showing Fe peak

To study the Pt coating distribution on the membrane (still including the GDL), an EDXRF mapping of the entire sample (size: 35.9 mm x 35.9 mm) was carried out using a 5 ms/pixel scanning speed. This step took less than 14 minutes to complete. Note that several samples can be placed side by side in the EA8000E sample jig (as long as they fit in a 250 mm x 200 mm area) where they can be measured in one setup. This greatly reduces the overall testing time for multiple samples, while freeing operators for other tasks.

The XRF mapping results showed that Pt was not distributed homogeneously over the sample surface (see Figure 6), and that Pt formed clumps in some areas. This could potentially lead to poor fuel cell quality and premature failure.

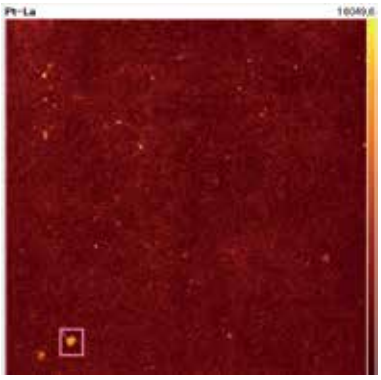

Figure 6: Pt La mapping image

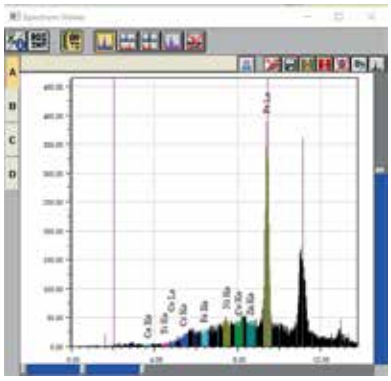

Figure 7: EDXRF spectrum showing Pt peak

Although it was not present in the membrane analyzed in this application note, cerium (Ce) can also be detected and its distribution determined with the EA8000E. Cerium oxide is often used in fuel cell applications to increase the oxidative stability of composite membranes, prevent chemical and mechanical degradation, thus increase fuel cell durability.

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Summary

The advanced technologies in the EA8000E X-ray analyzer were used to detect and characterize metal contaminants present in membrane electrode assemblies used in hydrogen fuel cell production. The analyzer's versatility enables operators to not only detect contaminants, but also confirm the Pt (and other elements such as Ce) distribution on the membrane, to ensure fuel cell quality and durability while minimizing production costs and waste.

A significant advantage of the EA8000E is that it can analyze contaminants beneath the surface, within the bulk of the membrane. This is an important feature to ensure that the MEAs are safe to release into production.

Hitachi High-Tech have developed a range of analytical instruments used for quality control during fuel cell production. For more information on the EA8000E, or to discuss wider testing requirements (for example Pt coating weight analysis), please contact our experts at contact@hitachi-hightech.com.

Technical specifications

- Element range: Mg (12) to U (92)
- Sample form: solids, powders
- Maximum sample size: 250 (W) x 200 (D) x up to 50 (H) mm
- Sample observation: high-resolution lens and CMOS camera
- X-ray fluorescence analyzer: 45 kV max. 900 µA max. X-ray tube with a 30 µm beam size, high-resolution Vortex® silicon-drift detector
- X-ray transmission analyzer: water-cooled X-ray tube, area image sensor

For full specifications, please visit our website or contact us at contact@hitachi-hightech.com.

More information

To find out more visit:
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