

Development of CCM Water Content Distribution Measurement System for Fuel Cell Stack

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ABSTRACT

A system was developed that uses impedance measurement of membrane electrode assemblies to quantify water content distribution in the polymer electrolyte membrane. That distribution influences fuel cell degradation factors including membrane damage in polymer electrolyte membranes and functional deterioration of electrode catalysts.

The impedance measurement sensors in this system are fabricated using flexible printed circuit technology to form thin shunt resistors for current measurement. This allows sensors to be produced in the same form as the membrane electrode assemblies.

With the development of these sensors, it became possible to measure the water content distribution in polymer electrolyte membranes in a fuel cell stack in real time while it is generating electricity. Those measurement results have been put to use in the 2016 model year for control of the water content of electrolyte membranes in actual fuel cell vehicles.

1. Introduction

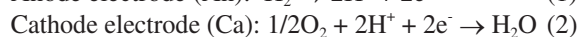
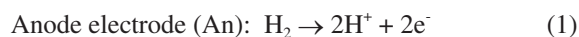
The environmental effects of CO₂, NO_x and other pollutants in automotive exhaust gases and the future depletion of fossil fuels make the development of clean, high-fuel-efficiency vehicles an urgent task. Seeking to realize a sustainable society based on energy circulation, Honda is engaged in research and development efforts in relation to fuel cell vehicles (FCV), which use hydrogen rather than fossil fuels, and fuel cell technologies⁽¹⁾.

Figure 1 shows an image of the structure of a fuel cell and its internal chemical reactions. The fuel cell (simply termed “cell” below) is made up of a membrane electrode

assembly (MEA) and separators that form gas channels. A fuel cell stack (termed “stack” below) is created by layering multiple cells together.

The MEA is made up of a polymer electrolyte membrane (PEM), a catalyst layer (CL), and a gas diffusion layer (GDL). The PEM making up the CL are also called catalyst-coated membranes (CCM).

In the cell, a hydrogen oxidation reaction occurs at the anode electrode as shown by Eq. (1), and an oxygen reduction reaction occurs at the cathode electrode as shown by Eq. (2). These chemical reactions between hydrogen and oxygen produce electricity and water.



In addition to seeking increased compactness and weight reductions, and realizing high-efficiency-generation performance with higher output, it is also necessary for the stacks fitted in FCV to display durability in relation to the fluctuations in current load to which cells used in automotive environments are subjected. To help ensure durability in cells, it is necessary to maintain the surface area of the electrode catalysts, which contributes significantly to the output of the cell, and to maintain the thickness of the PEM, which functions both to separate the oxygen and the hydrogen that serves as fuel, and to electrically insulate the anode and the cathode.

The ability to maintain the surface area of the electrode

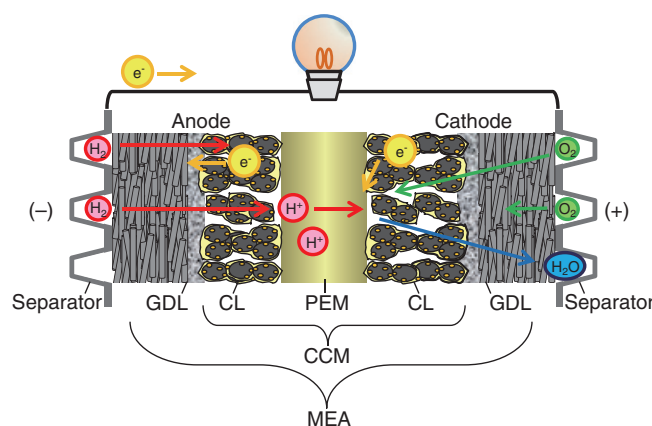


Fig. 1 Schematic image of fuel cell

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catalysts and the thickness of the PEM is closely related to the in-plane water content of the CCM in the cell. Generally, when the water content is high, the electrode catalysts become coagulated due to dissolution, agglomeration, and redeposition, and the decline in the catalyst surface area accelerates. It has also been reported that when the water content is low, impurities and hydrogen peroxide accumulate in the PEM, promoting chemical degradation and reducing the thickness of the PEM⁽²⁾.

In order to increase the durability of automotive fuel cells, it is therefore necessary to identify the in-plane distribution of the water content of the CCM and control it to within the proper range.

2. Purpose of Development

Obtaining a clear grasp of the CCM water content distribution is not only important in terms of enhancing performance and limiting degradation. It also has an important position in stack development because it provides useful information for setting operating conditions as well as for creating and analyzing durability modes.

The development of the stack for the 2016 model FCV therefore sought to realize increased stack durability through the development of multi-segment impedance sensors to identify the in-plane distribution of water content of the CCM in the cells and the calculation of the optimum in-plane water content distribution using simulations.

This paper will describe the development of the sensor that quantifies CCM water content distribution and the construction of a system employing that sensor.

3. Issues in Conventional Water Content Measurement

Methods for measuring CCM water content distribution include nuclear magnetic resonance (NMR) and neutron radiography⁽³⁾⁻⁽⁶⁾. Honda has long used neutron radiography as an analytical method for clarifying the condition of water accumulation in a fuel cell⁽⁷⁾. This measurement method involves irradiating the fuel cell with neutrons from a planar direction. The neutrons are converted into light by a scintillator, and the attenuation of the neutrons by the water is detected by a CCD camera as attenuation of that light. The light is converted into electrical signals and image processing is used to quantify the water content. As shown in Fig. 2, however, this method of measurement using neutron transmission includes the water accumulated in separator channels as well as water in the gas diffusion layer in that measurement. Therefore visual observation of CCM water content distribution was not readily achievable.

The fuel cell stack for the 2016 model is configured for power generating conditions that tend not to result in water accumulation in the separator channels.

There is an issue when the water content and distribution in a CCM with a thickness of several tens of micrometers

are measured using neutron radiography, because the lower water content causes the errors in image processing to increase, thereby reducing accuracy.

Figure 3 shows the results of measurement by neutron radiography during low-load electric generation. The blue portions in Fig. 3(a) indicate the presence of larger amounts of water. Figure 3(b) shows a graph of quantified in-plane water distribution as obtained by calculating the thickness of water from the density of images produced by measurement. The vertical axis of the graph shows the water content as a percentage, where the maximum CCM water content is 100%.

The results from quantification of the water content distribution in the cell by neutron radiography show instances of water content exceeding 100%. This happens because there is water present in the channels and gas diffusion layer, and it indicates that the water content in a CCM with a thickness of several tens of micrometers, which is extremely thin, cannot readily be measured with accuracy. Given this background, there was a call for development of

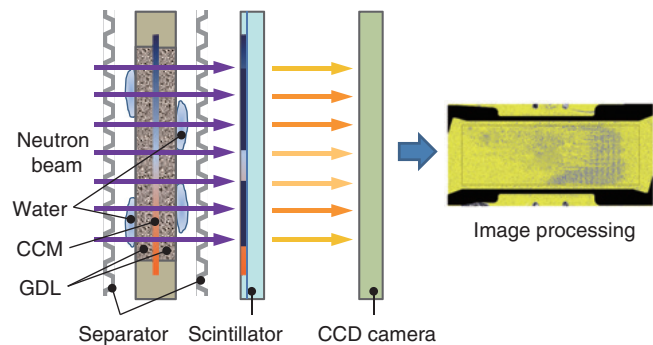


Fig. 2 Schematic image of detected water using Neutron RG

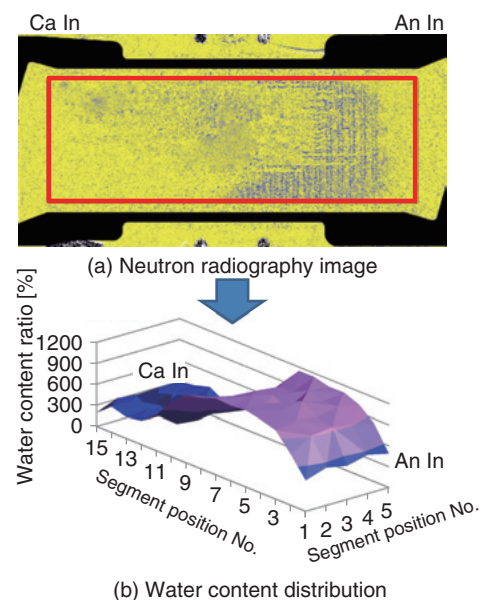


Fig. 3 Neutron Radiography and quantification result

a measurement system capable of accurately measuring the water content of the CCM alone.

4. Selection of Measurement Method

The construction of a system for controlling CCM water content in an actual vehicle, including during vehicle acceleration and deceleration, necessitated a method of instrumentation that would make it possible to obtain measurements without influencing the in-plane temperature distribution, humidity distribution, gas flow distribution, and other such factors during electric generation in a fuel cell system mounted in an actual vehicle. Selection of a measurement method involved examination from six perspectives in addition to the capability to measure CCM water content. The perspectives were production loading; ability to reproduce the humidity distribution, temperature distribution, and other such factors in the electric generation environment in the fuel cell; spatial resolution; time resolution; and capability to perform real-time measurement. The results of examination are shown in Table 1.

Measurement by X-ray and NMR offers high levels of resolution but requires bulky, elaborate equipment, which means that observations cannot readily be made with the system mounted in an actual vehicle. The impedance measurement method, on the other hand, works with a compact measurement system and by incorporating sensors in the system can perform measurement when mounted in an actual vehicle. This means it is capable of performing measurement in an actual electric generation environment.

Based on the results of the above examination, the impedance method was selected for mounting in actual vehicles to make measurements of changes in water content during actual vehicle operation, including acceleration and deceleration, and development carried out.

Table 1 Various measurement methods of water content inside fuel cell

	Impedance	Neutron RG	X-ray RG	X-ray CT	NMR
Indistinguishability for water content of PEM	○	△	△	○	○
Production loading	◎	×	×	×	×
Environment reproducibility	◎	△	×	×	×
Spatial resolution	△	○	◎	◎	○
Time resolution	◎	△	△	×	△
Real time measurement	○	△	△	×	×

◎: Excellent, ○: Good, △: Fair, ×: Poor

5. Development of the In-plane Water Content Distribution Measurement System

5.1. Fuel Cell Stack Construction

A distinctive feature of the fuel cell stack manufactured

by Honda is the cell structure, adopted since the 2009 model FCX Clarity, which provides for cooling alternate cells⁽¹⁾. This reduced by one-half the number of cooling layers previously needed in each fuel cell. The number of separators needed for two MEAs in this structure was reduced from four to three, aiding in making the stack more compact and achieving lighter weight.

The cell structure for cooling alternate cells is in a laminar arrangement with two MEAs and three separators making up a single unit. Figure 4(a) shows a cross-section of the conventional fuel cell structure, and Fig. 4(b) shows the structure with the developed sensors deployed.

5.2. Sensor Development

Figure 5 shows an overview of the sensor portion of the Water Content Distribution Measurement System developed to perform impedance measurement at the MEA surface in the fuel cell. The sensor was named the IDMS, from the initial letters of Impedance Distribution Measurement Sensor.

The sensor is intended for incorporation in a fuel cell stack and was therefore designed with the same shape as the MEA. As a result, it becomes possible to obtain CCM water content measurements without any need to prepare special separators or seals for the purpose because the sensor can be inserted into the fuel cell stack in place of one of the MEAs that make up the stack.

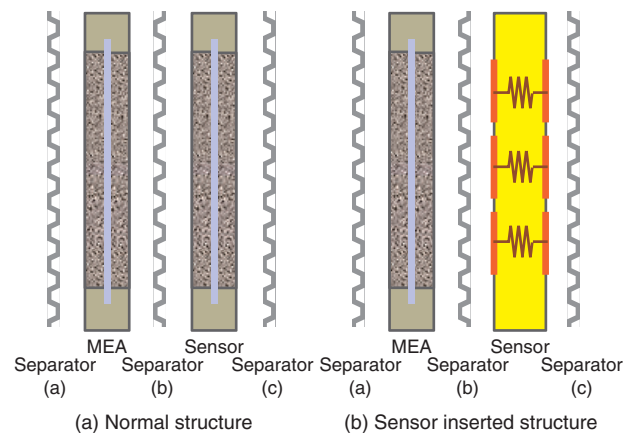


Fig. 4 Cell unit configuration of fuel cell stack

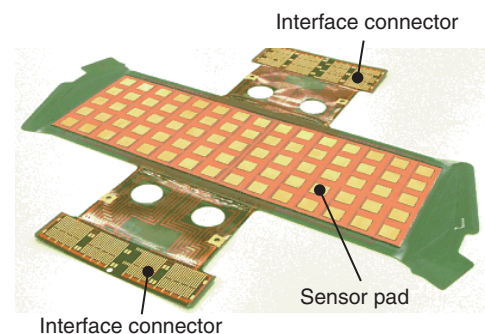


Fig. 5 Impedance distribution measurement sensor

The surface on either side of the sensor has multiple square sensor pads arranged on it. These sensor pads are used to make impedance measurements of the MEA. The sensor pads are designed so that each one handles the measurement of an equal area of the corresponding part of the power generating portion of the MEA that is on the other side of the separator placed between the sensor pad array and the MEA. The area of their contact with separator channels is also equal and their specifications limit measurement error factors resulting from differences in contact resistance.

Figure 6 shows the principle of measurement in this system.

The measurement of water content by means of impedance makes use of the change in ionic conductivity of the PEM due to differences in the water content of the CCM. Measurement superimposes an alternating current over the fuel cell stack as a whole. The membrane resistance is calculated from the current and voltage measurement figures from the sensors, and this is converted into membrane water content.

The superimposed alternating current is detected by shunt resistors inside the sensors. The IR drop that occurs (V_1) when a current flows through a resistor is measured and the current flowing in each sensor pad is derived from Eq. (3), below.

$$i_n = \frac{V_n}{R_n} \quad (3)$$

i_n : Terminal passing current [A]
 V_n : Shunt resistor voltage [V]
 R_n : Shunt resistance [Ω]

The voltage (V_2) is also measured between the sensor pad and separator (a).

The measured current and voltage value data is acquired

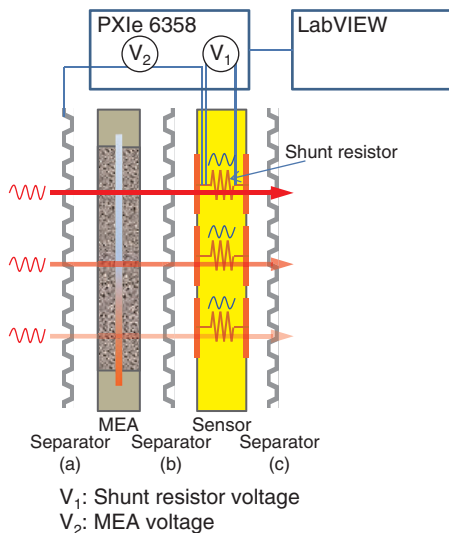


Fig. 6 Principle of CCM resistance measurement

with a PXIe-6358 (manufactured by National Instruments Corporation). Arithmetic processing is carried out by LabVIEW to calculate the impedance.

Impedance conversion is carried out in LabVIEW using Eq. (4), below.

$$Z_n = \frac{\Delta V_n}{\Delta I_n} \quad (4)$$

Z_n : Impedance [Ω]
 ΔV_n : AC voltage modulation [V]
 ΔI_n : AC current modulation [A]

The conversion of impedance measurement values into membrane water content is described in section 5.4.

5.3. Examination of Specifications

During sensor development, the focus was placed on resolution and measurement accuracy of in-plane CCM water content distribution. The design was examined in relation to the following headings.

5.3.1. Number of in-plane segments

The sensors are manufactured using flexible printed circuit (FPC) technology that allows complex wiring to be made thin. The flexible printed circuits have an adhesive layer formed on an insulating film that is from 12 to 50 μm thick. On top of that is affixed a conductive foil that is about 12 to 50 μm thick. Compared to the usual rigid substrate made of glass epoxy, this is thinner, more workable, and can be fabricated in more complex shapes.

Wiring inside the sensors is structured from five layers of wiring sheets that are formed by etching. Connections between layers are made through holes created by laser and plated inside to form conductive through-holes. Electric wiring in this three-dimensional structure is used to connect each sensor pad to an interface connector that connects to the measuring instrument (Fig. 7). Wiring with the same length and same resistance is used for the signal lines from each sensor pad in order to enhance measurement accuracy.

The upper limit and lower limit for the number of sensor pad segments needed in order to measure the water content distribution were examined from the perspectives of the feasibility of manufacturing the sensor and the accuracy of measurements obtained by the sensor.

Figure 8 shows the relationship between the current flowing in each sensor pad and the number of sensor pad segments, on the assumption that current flows equally in-

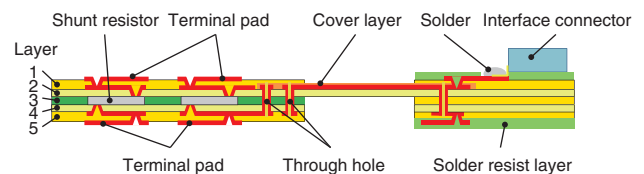


Fig. 7 Schematic image of FPC

plane within the electricity-generating electrodes of the fuel cell stack.

With the same amount of generated electric current in the stack, a larger number of sensor pad segments or partitions means that a smaller current flows through each pad [Fig. 9(a)]. Conversely, when there is a smaller number of sensor pad segments, the current flowing through each one will be larger [Fig. 9(b)].

When the interior wiring layers of the sensor are designed to ordinary parameters for conductor thickness, wiring width, and plating thickness, the upper limit for the current that can flow through each sensor pad is estimated to be about 7 A. Considering the variation in current density distribution when the stack is generating at maximum power, a safety factor of about 20% is added to the current transmitted through each sensor pad. The lower limit for the number of sensor pad segments is then about 60 segments.

The upper limit for the number of sensor pad segments greatly influences the wiring layout of the flexible printed circuit described earlier. Increasing the number of sensor pads complicates the wiring layout leading from the sensor pads at the center portion of the cell surface to the interface connector, so that the number of substrate layers needs to be increased. Considering that sensors are fabricated in the

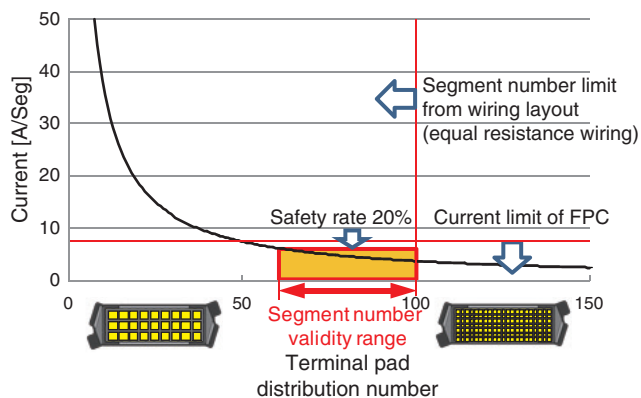


Fig. 8 Through current value of each sensor pad at various distribution numbers

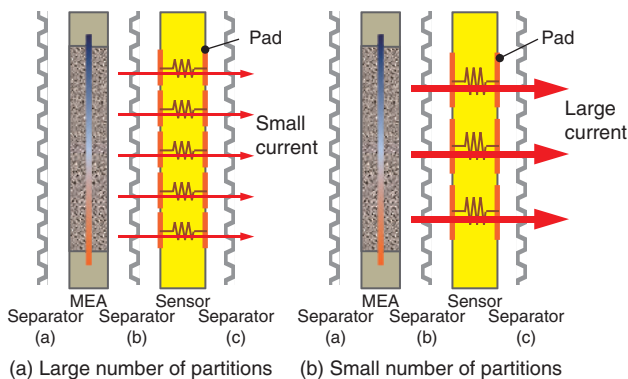


Fig. 9 Image of electric current quantity

shape of the MEA, an increase in the number of substrate layers and subsequent thickness of the sensors makes the mounting layout infeasible. The upper limit on the number of sensor pad segments will therefore generally be about 100 segments.

The results from the above examination of the number of sensor pad segments together with the aspect ratio of the electricity-generating portions of the MEA electrodes led to a sensor pad layout of 75 segments in five longitudinal columns and 15 lateral rows.

5.3.2. Sensor pad pitch

The electric current trapping rate was taken as the index for sensor measurement accuracy. The sensor pad size and shunt resistor magnitude were decided accordingly.

The electric current trapping rate indicates the percentage of an electric current that can be acquired by the corresponding sensor pad located opposite to the terminal outputting the current that will pass through the corresponding portion of the MEA and separator.

A conceptual image of the electric current trapping rate is shown in Figs. 10 and 11.

In a sensor with a high electric current trapping rate, the sensor pad-detected current in the current-impressing part is larger [Fig. 10(a) and Fig. 11(a)]. In a sensor with a lower trapping rate, however, the current values will be higher in parts of the sensor pad other than the current-impressing part, so that the sensor pad-detected current in the current-impressing part will be lower [Fig. 10(b) and Fig. 11(b)].

This indicates that a leak current flowing in a planar direction in a separator, a gas diffusion layer, or other such plane object is lowering the measurement accuracy.

The electric current trapping rate can also vary with the sensor pad arrangement and shunt resistor value. For this development, the pitch between sensor pads and the shunt resistor values were treated as parameters in working from the model in Fig. 12 to calculate the leak current from the contact resistance and specific resistance of each component. The electric current trapping rate of the sensor was then derived.

Figure 13 shows the results from calculation of

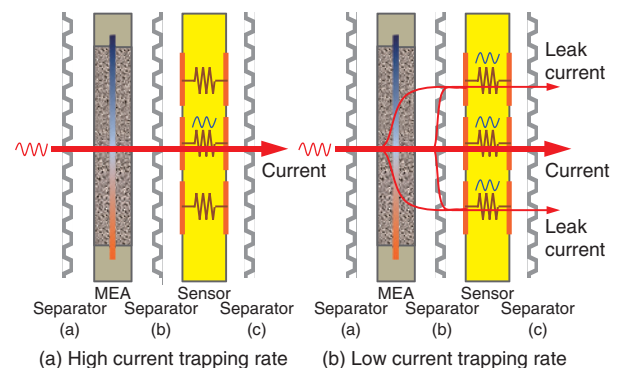
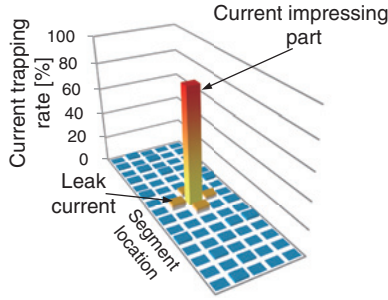
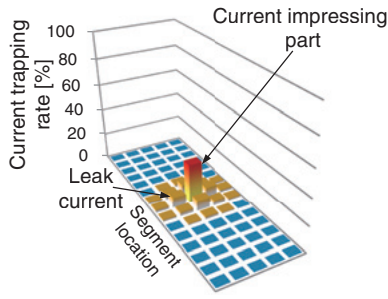


Fig. 10 Image of current trapping rate

variations in electric current trapping rate according to the pitch between sensor pads and the different ratings of shunt resistors used. Increasing the pitch between sensor pads can raise the electric current trapping rate. This happens because of the increase in distance between the separators that carry current to the sensor pad, thus increasing the resistance so that the flow of the leak current is impeded (Figs. 13 and 14). However, increasing the pitch between sensor pads means that the sensor pad size is reduced. This reduces the



(a) High current trapping rate



(b) Low current trapping rate

Fig. 11 Measuring result of current trapping rate (Image)

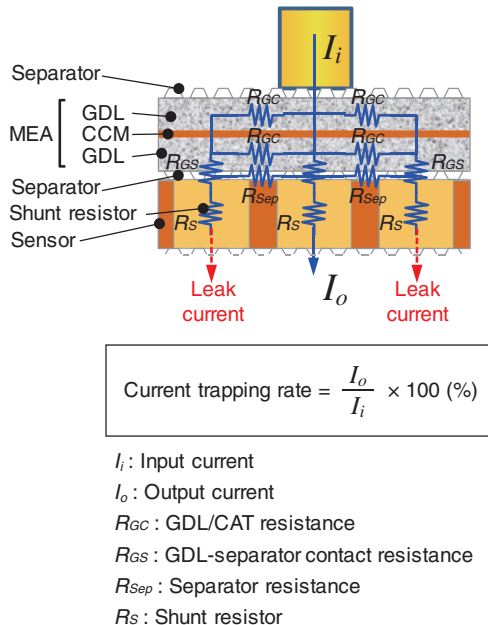


Fig. 12 Model of current trapping rate

contact area with separators, reducing the current path, so that the distribution can no longer be measured accurately.

The total sensor pad area needed to assure a necessary minimum current path was determined from the amount of current flowing when the stack is generating maximum power and from the contact resistance between sensors and separators. The size of each sensor pad was calculated from the necessary minimum total sensor pad area, and pitch between pads was set at 8 mm.

5.3.3. Shunt resistor rating

Manganin (Cu-Mn-Ni alloy) was used for the resistive element in the shunt resistor. This is a material that experiences only slight change in resistance with changes in temperature. The wiring used for current flow in the sensor was completely isolated from the wiring used for voltage measurement, and the specifications were arranged so that the influence of wiring resistance could be excluded from measurement.

Reducing the shunt resistance value can raise the electric current trapping rate. This is because the ratio of the resistance in the stacking direction to the resistance in the separator surface direction becomes smaller, and the flow of

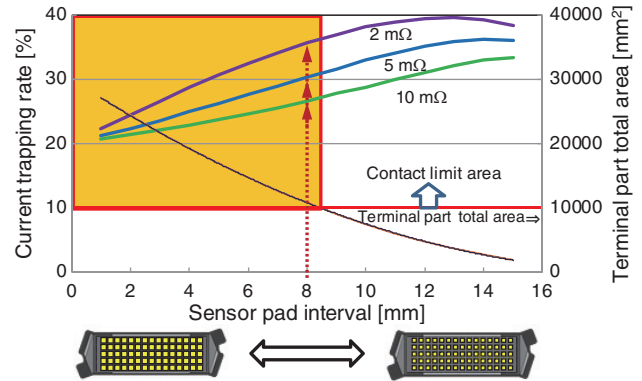


Fig. 13 Electric current trapping rate at various sensor pad intervals (75 segments)

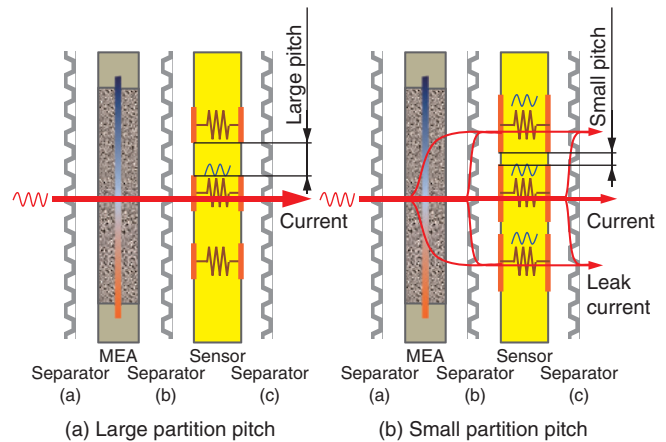


Fig. 14 Image of leak current at various segment pitches

current in the stacking direction is less impeded (Fig. 15).

However, although reducing the resistance value can raise the electric current trapping rate, this also means that requirements for the dimensional accuracy of the shunt resistors become more demanding.

The resistance of $5\text{ m}\Omega$ was adopted. This was a figure that allowed confirmation of the forming accuracy of shunt resistors manufactured by etching while also ultimately achieving the target of 3% or less variation between the shunt resistors arranged in each sensor pad.

Given the above examination of the number of sensor pad segments, the sensor pad arrangement and area, the shunt resistance values, and the flexible printed circuit production technology, it was ultimately decided to manufacture $5\text{ m}\Omega$ shunt resistors to be positioned at intervals of 8 mm.

5.4. Verification of Sensor Function

5.4.1. Evaluation fuel cell

Verification of IDMS function was carried out as shown in Fig. 16(a), by deploying the sensors in a full stack mounted in a 2016 model vehicle. The object of measurement by the sensors was the MEA of the fuel cell at the end of the stack. The sensors were installed in a

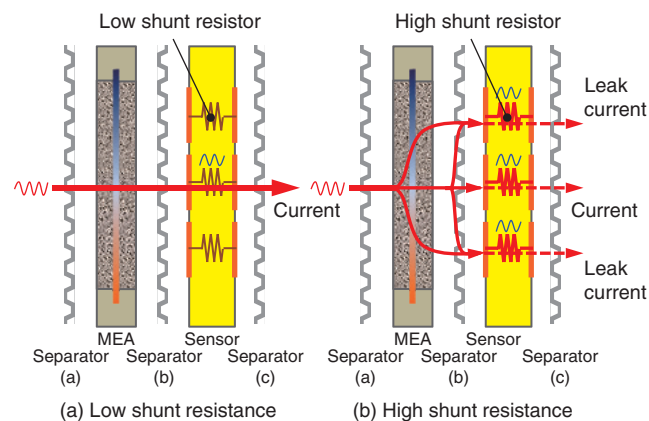


Fig. 15 Image of leak current at different shunt resistance

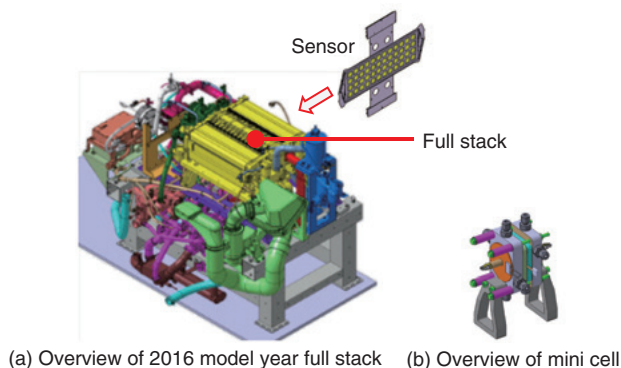


Fig. 16 Schematic image of full stack incorporating IDMS and mini cell for correlation test

position where they were adjacent to the end fuel cell, and measurements were made. The generation equipment used was 150 kW-class generation equipment employed for stand-alone evaluation testing of a full stack.

Sensor measurement accuracy was confirmed using a 2016 model mini-cell [Fig. 16(b)], which was being used to acquire MEA water content characteristics in a humid environment. The mini-cell has an electrode surface area of 12.5 cm^2 and is designed to have uniform in-plane temperature distribution, gas flow distribution, and humidity distribution, with the same channel geometry in the separators as those that make up the 2016 model stack. Cell temperature is controlled by an externally affixed heater. It was assembled to provide the same electrode contact pressure as the 2016 model.

5.4.2. Confirmation of resistance measurement results

In order to confirm the resistance measurement values obtained with the IDMS, resistance values were acquired in a temperature environment of 70°C with the humidity environment varied. The results were compared with the measurement results obtained under the same conditions from the mini-cell.

Variances in measurement accuracy due to differences in sensor electrode position were confirmed by taking the impedance measurement values obtained at 25 locations such as gas ports and the central portion on the IDMS 75-sensor pad array, where impedance measurement values tend to differ even under the same conditions, and comparing them with the impedance measurement values in the mini-cell (electrode surface area of 12.5 cm^2). The same gases as during electric generation were used for input, with hydrogen gas on the anode side and air on the cathode side. The time maintained at each humidity level was set at two hours, a period during which the relative humidity would become stable throughout the entire MEA. The relative humidity was changed in increments of 10% from lower to higher humidity. Figure 17 shows the results for impedance measurement at each humidity level. When IDMS impedance values were compared with the mini-

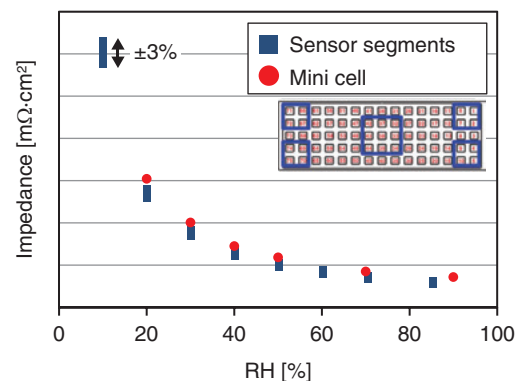


Fig. 17 Impedance CCM measurement value at various RH levels

cell values, it was found that values were uniformly lower by 10% or so across the full range of humidity. This is likely to be because contact resistance within the cell has increased because the mini-cell has fewer cell layers and the fastening load therefore tends to decrease. Differences of approximately $\pm 3\%$ also exist between the segments, and this is thought to occur due to in-plane surface pressure distribution in the full stack as well as in-plane distribution of the humidity environment at the surface.

In order to correct for the influence of these surface pressure and humidity environment distribution factors, the relationship between humidity and impedance was obtained for each segment. This enabled accurate calculation of the water content, which will be described later.

5.4.3. Conversion of measured resistance values to water content

Figure 18 shows the sequence followed in converting the resistance values obtained at each electrode to water content.

First, the distribution of resistance values obtained by impedance measurement is converted to the humidity environment indicated by those resistance values [Fig. 18(a)]. The standard curve obtained from sensor measurements of resistance against changes in humidity was used in making this conversion. Next, the humidity environment was converted into the MEA water content [Fig. 18(b)]. The conversion to water content was carried out using the humidity and MEA water content standard curve.

5.4.4. Measurement of sensor resistance values against changes in humidity

With the IDMS installed on the full stack, the humidity of the input gas was varied and the change in MEA resistance in each segment was measured at the temperature for the electricity generation condition that is to be measured. Figure 19 shows the standard curve obtained (the same 25 segments as in Fig. 17). This standard curve was used, as shown in Fig. 18(a), to convert the resistance value of each segment into the humidity that indicates that resistance value.

5.4.5. Measurement of MEA mass against changing environmental humidity

The CCM water content at the humidity levels described above was measured using a water sorption/desorption analyzer (the IGA-SORP manufactured by Hidden Analytical).

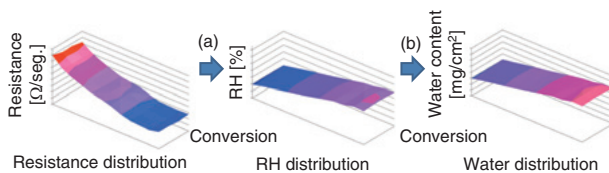


Fig. 18 Conversion of resistance to water distribution

The measurement procedure is shown in Fig. 20.

CCM pieces 20×20 mm in size (4 cm^2) were used as samples. The samples were placed in a stainless steel mesh sample holder [(1) in the figure] and inserted in the IGA-SORP (constant temperature) [(2) in the figure] and dried under a flow of nitrogen gas [(3) in the figure] for two hours. After measuring the mass when the sample was dry, continuous measurements of mass were made under controlled temperature and humidity [(4) in the figure]. The CCM mass obtained after holding for 30 minutes at each humidity condition was used to calculate the water content under each humidity environment. Figure 21 shows a graph of the water content calculated from each humidity level

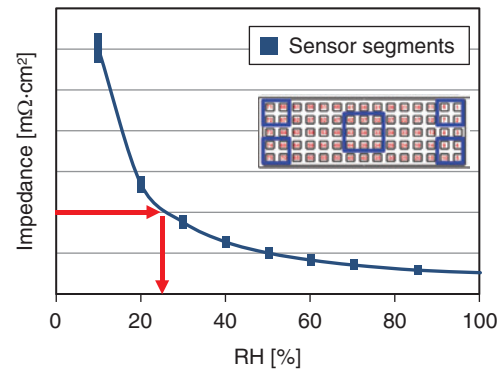


Fig. 19 Impedance CCM measurement value at various RH levels

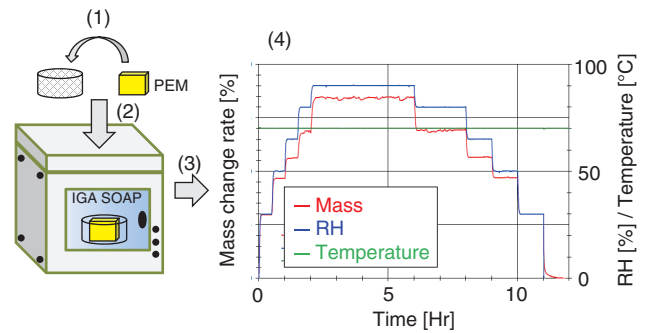


Fig. 20 Test procedure of adsorption and desorption measurement

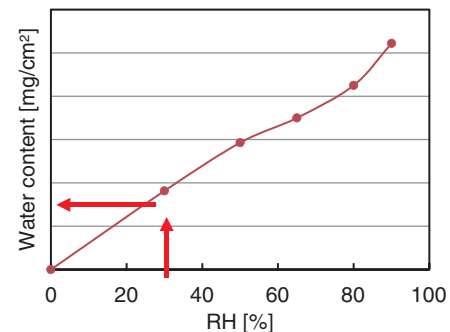


Fig. 21 Water content measurement of CCM at various RH levels

and CCM mass. The graph in Fig. 21 was used to convert the humidity distribution into water content distribution [Fig. 18(b)], yielding the MEA water content distribution during electric generation.

6. Results from Application to Fuel Cell Stacks

6.1. Real-Time Measurement of Water Content

Figure 22 shows the changes in water content and load during electric generation in the stack evaluation operating mode used during development. The sensors are incorporated into the fuel cell stack of a system mounted in an actual vehicle. The CCM water content shows the change over time at the anode inlet side, the central portion, and the cathode inlet side. Under high load, the water content reaches the highest level in the central portion. Meanwhile, it also became clear that the water content on the anode input side shows only slight change.

This measurement system is capable of making impedance measurements in each area at 0.5-second intervals. The time resolution is finer than with neutron radiography, which involves individual 8-second picture acquisition times. As a result, changes in CCM water content during acceleration and deceleration can now be confirmed in detail. The spatial resolution is also sufficient with 75 segments, and the measurements are accurate enough for discussion of upper and lower limits of in-plane water content distribution in the CCM. With development of the IDMS, therefore, CCM water content in an operating fuel cell stack can now be measured in real time and in detail.

6.2. Measurement of Water Content Distribution

The results from IDMS measurement of water content distribution during electric generation under low load [Fig. 23(b)] were compared with those from quantification by

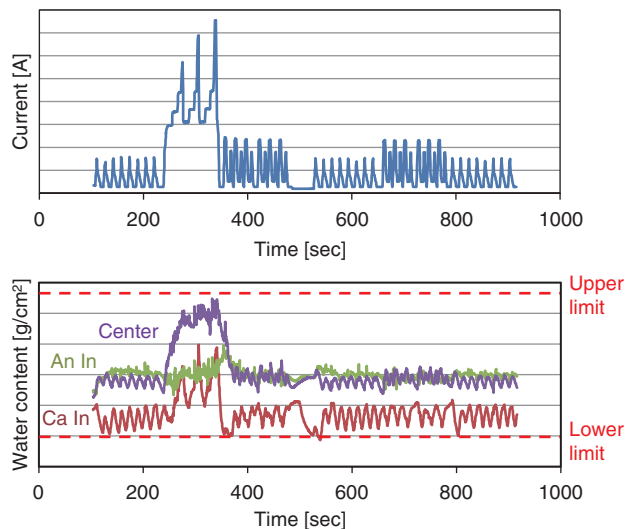
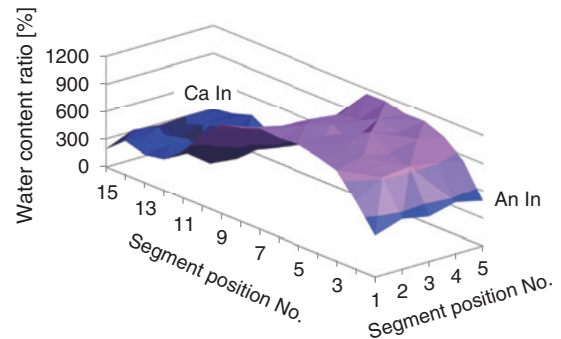


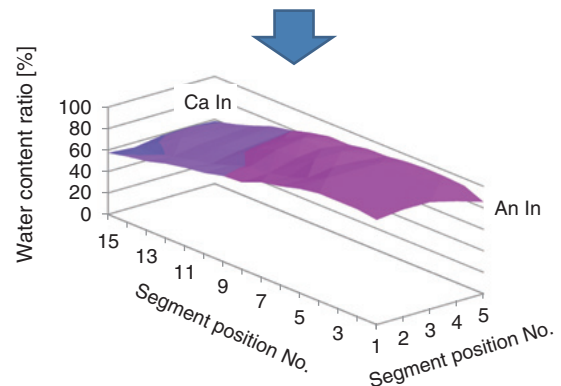
Fig. 22 Change of water content during generation

neutron radiography [Fig. 23(a)]. Neutron radiography was only capable of measurement that included water in the separator channels and gas diffusion layer. With measurement using IDMS, however, the distribution of CCM water content could be measured.

The measurement of CCM water content distribution using IDMS has enabled prediction and actual verification of acceleration modes of mechanical degradation caused by repeated contraction of the CCM when dry and expansion when moist⁽⁸⁾ and enhancement of the predictive accuracy of simulation⁽⁹⁾. Measurement of CCM water content in a system mounted in an actual vehicle also resulted in the creation of water content control during system operation⁽¹⁰⁾, which was applied in actual vehicles.



(a) Quantification result by neutron RG



(b) PEM water content from IDMS sensor

Fig. 23 CCM water content distribution at low generation condition

7. Conclusion

- (1) An impedance measurement sensor with MEA shape enabling incorporation in systems mounted in actual vehicles was developed. The sensor was made capable of measuring CCM water content distribution in real time in an actual vehicle operating environment.
- (2) Examination of the number of sensor pads and their placement interval enabled accurate, high-resolution measurement of CCM water content distribution in a fuel cell stack.

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