

Mechanical Structure and Performance Evaluation of High Differential Pressure Water Electrolysis Cell

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ABSTRACT

High differential pressure water electrolysis is a method that can replace the conventional high-pressure hydrogen production technology that uses normal pressure water electrolysis and a mechanical compressor. The differential pressure method requires a pressure resistant electrolysis cell, and the target hydrogen pressure is to be held by the proton exchange membrane. These issues were addressed by adjusting the strength and surfacial pores of the anode current collector and suppressing proton exchange membrane deformation, enabling the membrane to sustain a differential pressure of 35 MPa. Tests using a piston-type high differential pressure water electrolysis cell clarified that the optimum interfacial pressure between the current collector and the catalyst is 5 MPa. High-pressure hydrogen was successfully produced with a theoretical pressurization energy based on the concentration difference voltage by maintaining a constant interfacial pressure regardless of the hydrogen pressure inside the cell. In addition, it was confirmed that the current efficiency was 97%, the only loss was that due to hydrogen cross-permeation across the membrane, and that the cross-permeated hydrogen had no effect on other functions. Cross-permeation of oxygen was suppressed due to the differential pressure, and the oxygen concentration in the produced hydrogen was the specification value of 5 ppm or less. This means that the obtained hydrogen had a higher purity than that produced by normal pressure water electrolysis.

1. Introduction

Research is being conducted on a Solar Hydrogen Station (SHS) that combines renewable electricity generated by a solar cell with water electrolysis as a way of achieving zero emissions for the fuel production process of fuel cell electric vehicles (FCEV). Proving tests for the initial development-type SHS1 that used water electrolysis and a mechanical compressor showed that the power consumed by the mechanical compressor accounted for approximately 40% of the total of the system auxiliary equipment^{(1), (2)}, and that eliminating the mechanical compressor would effectively further reduce size and increase efficiency for household use. Proton-exchange membrane-type high-pressure water electrolysis is an alternative technology for this, and there are two types: equal pressure and differential pressure. Of these, the equal pressure-type is generally used. Application of differential pressure to the proton exchange membrane can be avoided by making both the cathode and anode high pressure, and housing the water electrolysis cell in a high-pressure vessel. However, this is suited for commercial applications because the size increases. The differential pressure-type was therefore selected as the size can be reduced by making

the water electrolysis cell pressure resistant and making only the cathode high pressure. There are no known cases to date of using the proton exchange membrane to sustain the target pressure difference of 35 MPa. However, the Solar Hydrogen Station SHS2 applies technology that achieves this, and proving tests have been underway since 2010⁽³⁾. The basic configuration of the high differential pressure water electrolysis cell that is the core technology of the SHS2 was investigated, and this paper describes that cell structure and performance evaluation results.

2. Technical Issues for a High Differential Pressure Water Electrolysis Cell

Figure 1 shows a schematic of the high differential pressure water electrolysis cell. The water electrolysis principle is the same as in the case of the atmospheric pressure method, but high-pressure hydrogen is obtained by continuously storing the produced hydrogen in an enclosed space, and only the cathode becomes high pressure.

Equation (1) shows the formula used to obtain the electrolysis voltage E_{WE} required for high differential pressure water electrolysis.

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$$E_{WE} = E_0 + \frac{RT}{2F} \ln \frac{P_1}{P_2} + ir \quad (1)$$

E_0 : Theoretical electrolysis voltage [V]

T : Absolute temperature [K]

R : Gas constant [J/(K·mol)]

P_1 : High-pressure side hydrogen partial pressure [MPa]

P_2 : Low-pressure side hydrogen partial pressure [MPa]

ir : DC resistance voltage [V]

F : Faraday constant [C/mol]

The electrolysis voltage required for high-pressure water electrolysis is obtained by adding the voltage derived from the hydrogen concentration difference to the voltage required for water electrolysis. The Nernst formula shown in the second term on the right side of the formula in Eq. (1) is the voltage from hydrogen concentration difference required by a gas concentration cell⁽⁴⁾. The pressurization energy to 35 MPa derived from this concentration difference is estimated to be approximately 180 Wh per 1 Nm³ of hydrogen at 25°C, and this value does not depend on the cell size or current, that is to say the amount of hydrogen produced. This is a large advantage over compact mechanical compressors for household use.

However, there are three main technological issues that

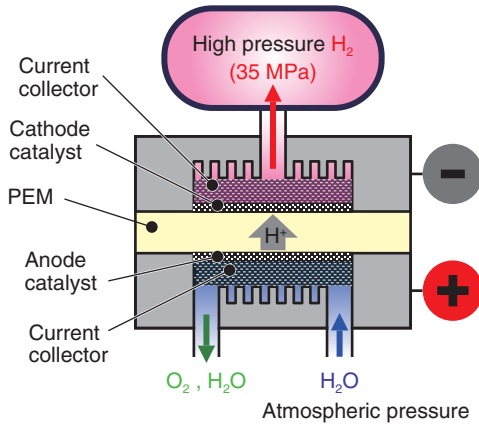


Fig. 1 Schematic of high differential pressure water electrolysis

must be addressed to realize high differential pressure water electrolysis. Figure 2 shows a schematic of these issues and the cell configuration.

The first issue is that the cell must be pressure resistant with respect to the differential pressure between the target produced hydrogen pressure and the anode. This means that the proton exchange membrane (PEM) is used to hold the differential pressure. To achieve this, deformation of the anode current collector should be avoided, and deformation of the proton exchange membrane into the pores present on the current collector surface should also be suppressed. The current collector is porous, and the strength is increased by reducing the surfacial pore and internal pore sizes to approach a bulk body. However, this reduces the fluid permeability required for water electrolysis, so the strength and surfacial pores must be adjusted to simultaneously realize both of these conflicting properties.

The second issue is that unlike water electrolysis, the hydrogen pressure inside the cell generates separation force (hereafter “counter direction load”) between the catalyst and the cathode current collector. Therefore, interfacial pressure should be secured to reduce the contact resistance between the catalyst and the cathode current collector, in accordance with the rise in the hydrogen pressure.

The third issue is an increase in the amount of hydrogen permeation to the anode due to the differential pressure, which is a phenomenon unique to differential pressure-type water electrolysis. Equation (2) shows the theoretical formula of the relationship between the hydrogen pressure and the hydrogen permeation amount⁽⁵⁾.

$$N = \alpha \cdot \left(\frac{P_1 - P_2}{d} \right) \cdot \exp \left(\frac{-E_a}{RT} \right) \quad (2)$$

N : Gas permeation speed per unit area [m/sec]

α : Gas permeation coefficient [m³·m/(m²·sec·MPa)]

d : Membrane thickness [m]

P_1 : High-pressure side pressure [MPa]

P_2 : Low-pressure side pressure [MPa]

T : Absolute temperature [K]

R : Gas constant [J/(K·mol)]

E_a : Permeation activation energy [J/mol]

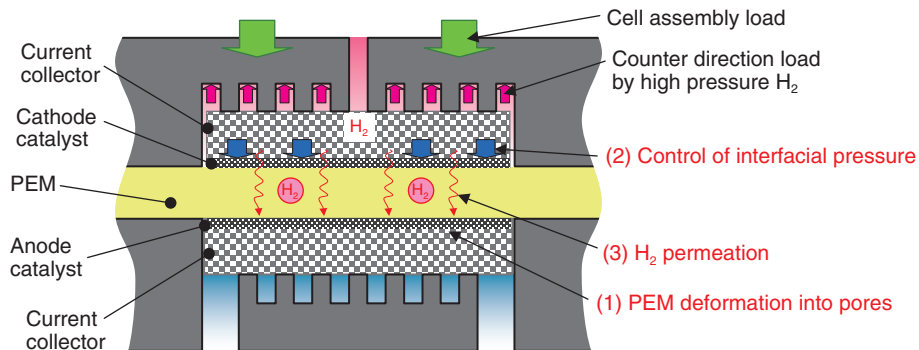


Fig. 2 Three technological issues for high differential pressure water electrolysis cell

According to this formula, the amount of gas cross-permeation across the proton exchange membrane is proportional to the pressure difference, so there is more hydrogen permeation compared to water electrolysis. This permeation reduces the amount of hydrogen that can be obtained relative to the current, and also gives rise to concerns of mixed gas with oxygen. Therefore, high differential pressure water electrolysis must also be investigated from the viewpoint of the gas permeation amount.

This study manufactured a cell that enables investigation of these issues. The goals were to successfully sustain the differential pressure using the proton exchange membrane and to evaluate electrolysis performance under a differential pressure of 35 MPa.

3. Experimental Method

3.1. High Differential Pressure Water Electrolysis Cell Structure

3.1.1. Test high-pressure water electrolysis cell

A piston-type high-pressure water electrolysis cell that enables adjustment of the cell internal pressure and the interfacial pressure was manufactured to investigate the issues described in the previous section. Figure 3 shows the sectional drawing.

The basic configuration is the same as that of a water electrolysis cell, but the high-pressure hydrogen generates additional stress, so a high-pressure vessel design was used for the overall cell. In addition, the cathode applied a piston structure to help ensure contact between the current collector and the catalyst. The piston diameter and the cathode current collector diameter are the same, so the pressure difference between the nitrogen gas introduced to the piston and the produced hydrogen is the interfacial pressure. The interfacial pressure was varied in accordance with the counter direction load by adjusting

this nitrogen pressure.

The anode catalyst used the same Ru-Ir-Fe complex oxide catalyst as that used for water electrolysis. A platinum black catalyst was used as the cathode catalyst. These catalysts were hot-pressed against a fluoride-based solid polymer proton exchange membrane with a thickness of 100 μm . Titanium was used as the cell material as in the case of a water electrolysis cell.

3.1.2. Current collectors

The anode current collector is a key part for sustaining the differential pressure, and spherical titanium powder sintered into a slab shape was used as this material. Figure 4 shows photographs of the external appearance and an enlarged view of the anode current collector. The sintering conditions were adjusted to increase the strength and fineness of this current collector while still retaining the fluid permeability required for water electrolysis.

The cell structure uses the proton exchange membrane to sustain the differential pressure of 35 MPa. Enhancing the surfacial pore fineness of the anode current collector enabled to suppress proton exchange membrane deformation, strengthen the current collector to withstand deformation due to the high-pressure gas load, and support the entire proton exchange membrane. These components

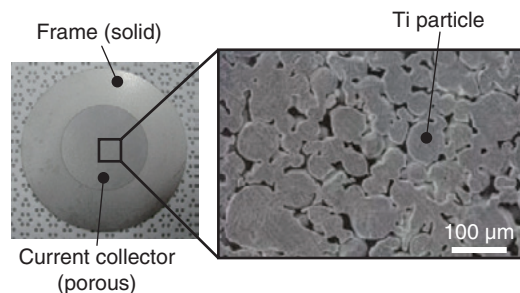


Fig. 4 Current collector

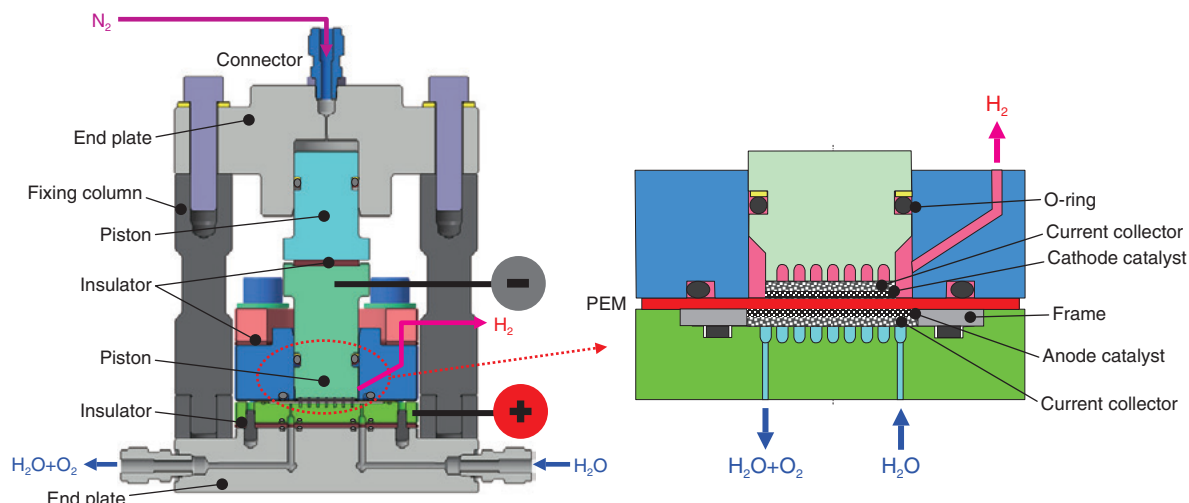


Fig. 3 Structure of experimental electrolysis cell

are stacked together to manifest the differential pressure sustaining function. The same structure was also used for the cathode current collector.

3.2. Confirmation of Differential Pressure Sustaining Function and Test Method for Measuring the Hydrogen Cross-permeation Amount

Figure 5 shows a schematic of the system used for the series of tests in this study.

The proton exchange membrane was soaked in purified water and placed on top of the current collector, then the components were bolted together to prevent gaps from occurring between each component due to the counter direction load of the high-pressure hydrogen. Purified water was circulated and the water temperature was adjusted using a heater. Nitrogen was introduced to the cathode by a compressor, and the internal pressure was regulated by a back-pressure valve. The internal pressure was measured using a pressure transmitter, and the pressure resistance was judged according to whether there was a rapid drop in pressure other than the drop in pressure due to cross-permeation across the membrane.

To measure the amount of hydrogen permeation, hydrogen was introduced by a compressor, the hydrogen that permeated across the membrane was collected by the circulation water, and the amount of hydrogen permeation per unit time was obtained from the product of the nitrogen dilution flow rate measured using a mass flow controller and the hydrogen concentration measured using a gas chromatograph. The permeation area was the anode current collector area.

3.3. Test Method for Evaluation of Electrolysis Performance

First, to determine the appropriate interfacial pressure between the current collector and the catalyst, the nitrogen

pressure was adjusted to vary the interfacial pressure from 0.2 MPa to 40 MPa while performing water electrolysis at atmospheric pressure, and the relationship between the interfacial pressure and the electrolysis voltage was evaluated.

Next, high-pressure water electrolysis was performed at 35 MPa with the appropriate interfacial pressure determined by the above operation, and the relationship between the hydrogen pressure and voltage efficiency was evaluated. The voltage efficiency is obtained by Eq. (3).

$$\eta_V = \frac{E_0}{E_{WE}} \times 100 \quad (3)$$

η_V : Voltage efficiency [%]

E_0 : Theoretical electrolysis voltage = 1.48 [V]

E_{WE} : Electrolysis voltage [V]

The generated hydrogen passes through a back-pressure valve and is then depressurized to atmospheric pressure and separated from the water by a hydrogen separator. The separated hydrogen flow rate was measured by a wet-type integrating flow meter. Equation (4) was used to calculate the current efficiency from the current and the integral value of the obtained hydrogen per unit time. Part of the hydrogen was dehumidified and the concentration of oxygen, which is an impurity, was measured using a gas chromatograph. The generated oxygen was separated using unreacted water and an oxygen separator, diluted by nitrogen, and exhausted.

$$\eta_A = Q \cdot \left(\frac{I_{WE} \cdot 3600}{F} \cdot \frac{1}{2} \right)^{-1} \times 100 \quad (4)$$

η_A : Voltage efficiency [%]

Q : Generated hydrogen amount [mol/hr]

I_{WE} : Electrolysis current [A]

F : Faraday constant [C/mol]

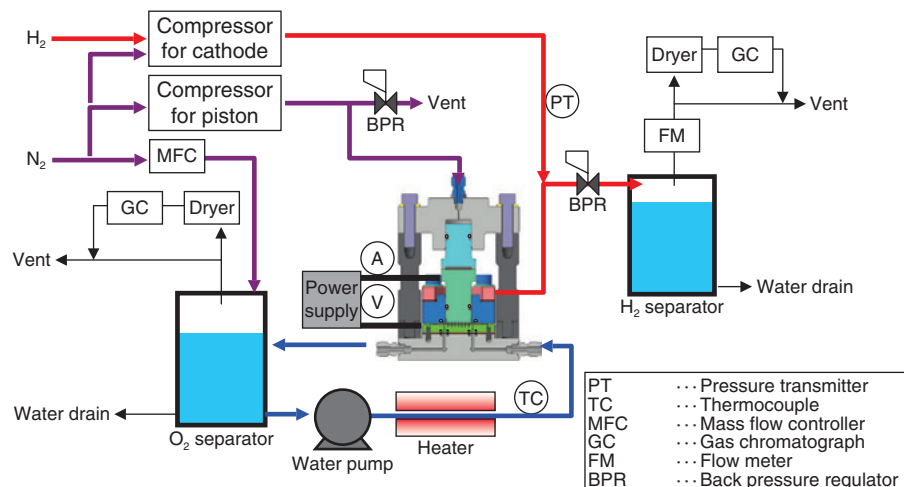


Fig. 5 Evaluation system

4. Results and Discussion

4.1. Test to Confirm Differential Pressure Sustaining Function and Test to Measure the Hydrogen Cross-permeation Amount

4.1.1. Test to confirm differential pressure sustaining function

Tests to confirm that the differential pressure of 35 MPa was sustained were performed before performing actual high-pressure water electrolysis. Figure 6 shows the time series change in the cathode side pressure due to nitrogen pressurization by the compressor. Nitrogen introduction was stopped to check for pressure drops at 1, 5, 10, 15, 25 and 35 MPa. No drop in pressure other than the slight decrease due to cross-permeation across the membrane was observed during this operation, which confirmed that the differential pressure of 35 MPa was sustained by the proton exchange membrane.

Typical proton exchange membranes have a tensile strength of approximately 20 MPa to 30 MPa at room temperature, so they are unable to sustain this differential pressure. It is presumed that in the developed cell structure, reducing the size of the pores in the anode current collector that supports the membrane suppressed proton exchange membrane deformation so that break elongation was not reached, even in the state with pressure of 35 MPa applied.

4.1.2. Test to measure the hydrogen cross-permeation amount

Next, the amount of hydrogen cross-permeation across the membrane during high differential pressure water electrolysis was confirmed. Figure 7 shows the relationship between the hydrogen pressure and the membrane permeation amount, and the estimated current efficiency values calculated from the membrane permeation amount. The membrane permeation amount increases linearly together with the rise in hydrogen pressure, and the trend matched the theoretical formula for the membrane permeation amount given in Eq. (2).

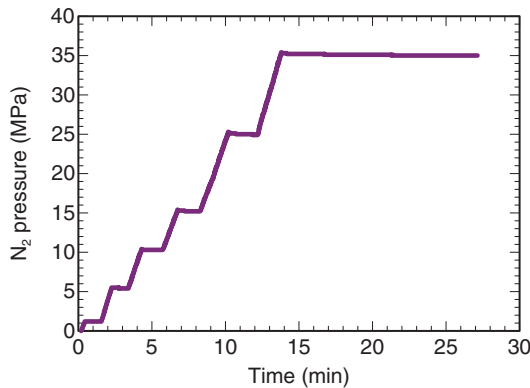


Fig. 6 Pressure sustaining characteristics relative to change in N_2 pressure over time (30°C)

Under conditions of 30°C and 35 MPa, the membrane permeation amount was $0.20 \text{ cm}^3/\text{min}\cdot\text{cm}^2$. This is approximately 2.8% of the amount of hydrogen obtained at a current of $1 \text{ A}/\text{cm}^2$, and becomes the loss due to cross-permeation to the anode, so a current efficiency of approximately 97.2% can be predicted. In addition, this cross-permeated hydrogen forms a mixed gas with a concentration of approximately 5.6% at the anode. This is a combustible concentration, so the reactivity inside the cell was investigated from the two points of the mixed gas state and the presence of an ignition source in order to confirm the effects of this mixed gas on high differential pressure water electrolysis cell function.

As shown in Fig. 5, in order to supply sufficient water to the proton exchange membrane, the high differential pressure water electrolysis cell circulates several times the amount of water used for the reaction. The oxygen and water in this state can be predicted to conform to slag flow in which the gas is separated as bubbles⁽⁶⁾, so even if an ignition source is present the reaction does not continue. This is confirmed by the method reported by Inoue et al. of suppressing combustion propagation by spraying water mist onto hydrogen combustion waves in the presence of inert gas in order to lower the temperature and pressure⁽⁷⁾. In addition, reactivity between the anode catalyst and the hydrogen-oxygen mixed gas was investigated to determine the possibility of the catalyst becoming an ignition source. However, there was no rise in the catalyst surface temperature even at a stoichiometric ratio concentration of 2:1.

The above results indicate that the drop in current efficiency due to the differential pressure is slight, and that water electrolysis cell function is not affected by the generated mixed gas.

4.2. Test to Evaluate Electrolysis Performance

4.2.1. Determination of appropriate interfacial pressure by water electrolysis

First, the water electrolysis voltage was measured while varying the interfacial pressure to determine the target

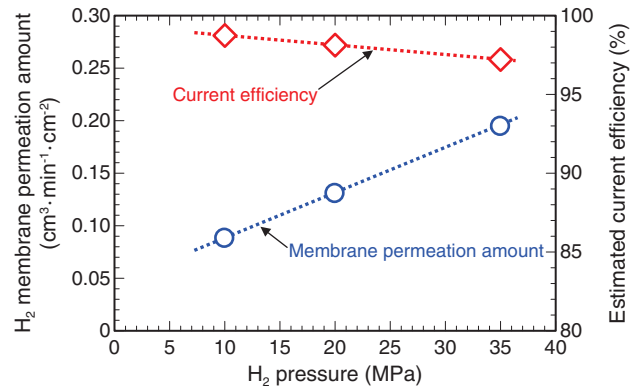


Fig. 7 Influence on H_2 permeation and estimated current efficiency (30°C)

interfacial pressure. Figure 8 shows these results. As the interfacial pressure was increased from around 0, the voltage dropped sharply, exhibited a minimum value in the 3 MPa to 10 MPa range, and then rose gradually towards 40 MPa.

The range with an interfacial pressure of 3 MPa or less is thought to be the range where the contact resistance increases between a current collector that has been smoothed for differential pressure water electrolysis and a catalyst. At high interfacial pressures of 10 MPa or more, other factors that reduce efficiency are present. This behavior is presumed to occur because the interfacial pressure ejects water from the moistened proton exchange membrane, which reduces the proton conductivity and increases the membrane resistance.

Based on these results, 5 MPa was selected as the appropriate interfacial pressure, as this pressure has the least influence on contact resistance and membrane resistance.

4.2.2. Production of high-pressure hydrogen by self-pressurization and relationship between hydrogen pressure and voltage efficiency

Next, actual high-pressure water electrolysis was performed, and the relationship between the hydrogen pressure and the voltage was simulated and evaluated for the two cell types of the pre-set interfacial pressure-type and the constant interfacial pressure-type. Figure 9 shows schematics of each cell structure.

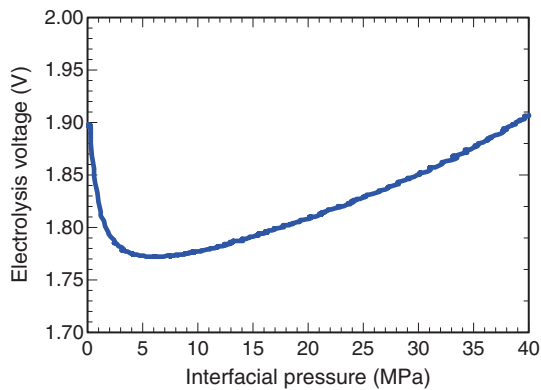


Fig. 8 Influence of interfacial pressure on electrolysis voltage (30°C, 1 A/cm²)

The pre-set interfacial pressure-type is also used for water electrolysis, and has a cell structure that clamps the electrodes from the edges of the cell using bolts or other fasteners. There is no need for a mechanism that applies interfacial pressure inside each cell, so there are the merits of a small number of parts and reduced thickness for stacking. However, when applied to a high differential pressure water electrolysis cell, the interfacial pressure drops as the hydrogen pressure inside the cell increases. Therefore, the sum of both loads of the hydrogen pressure of 35 MPa and the interfacial pressure must be applied during assembly. On the other hand, the constant interfacial pressure-type has a structure that maintains a constant interfacial pressure regardless of the hydrogen pressure, by adding an elastic part (Coned disc spring in Fig. 9) installed inside the cell. Figure 10 shows the results of evaluating the relationship between the voltage and hydrogen pressure while adjusting the piston internal pressure to apply interfacial pressure that simulates each structure.

As the hydrogen pressure changed from atmospheric pressure to 35 MPa, the interfacial pressure for the pre-set interfacial pressure-type changed from 40 MPa to the target 5 MPa. However, the constant interfacial pressure-type

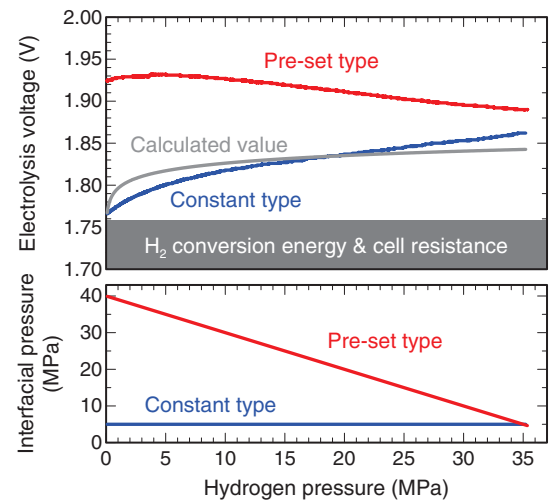


Fig. 10 Comparison of electrolysis voltage and interfacial pressure (30°C, 1 A/cm²)

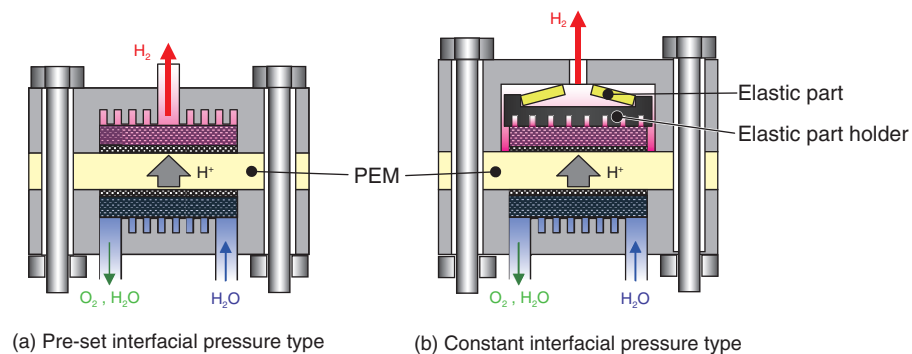


Fig. 9 Comparison of fastening mechanism

maintained a constant interfacial pressure of 5 MPa. During this operation, the pre-set interfacial pressure-type required a voltage of approximately 1.93 V at atmospheric pressure, and this voltage decreased as the pressure increased. The constant interfacial pressure-type required a voltage of approximately 1.76 V at atmospheric pressure, which is lower than that of the pre-set interfacial pressure-type, and this voltage then increased gradually as the pressure increased. The voltage for both types was approximately 1.9 V at 35 MPa, indicating that electrolysis could be performed at the target interfacial pressure.

The pre-set interfacial pressure-type experiences excessive interfacial pressure during pressurization from atmospheric pressure. This means that electrolysis is performed in the state with high membrane resistance, resulting in low voltage efficiency. This matched the tendency measured in the high interfacial pressure test shown in Fig. 8. The constant interfacial pressure-type maintains a constant interfacial pressure, so the effects of contact resistance and membrane resistance are small, and the behavior was close to the calculated concentration difference voltage values.

There is no major difference in voltage efficiency between both structures when steady-state electrolysis is performed at 35 MPa. However, during pressurization, the constant interfacial pressure-type that is not subject to a drop in voltage efficiency was found to be advantageous. In addition, the actual hydrogen pressurization energy was 183 Wh per 1 Nm³ at 30°C, which means that energy consumption could be reduced to approximately 1/4 that of the mechanical compressor used in the SHS1⁽²⁾.

4.2.3. Evaluation of electrolysis performance

Figure 11 shows the current-voltage performance curves (hereafter “IV curve”) from atmospheric pressure to 35 MPa, at 60°C. The IV curves from 10 MPa to 35 MPa trend in parallel to the atmospheric pressure IV curve, and there is almost no change in the resistance component

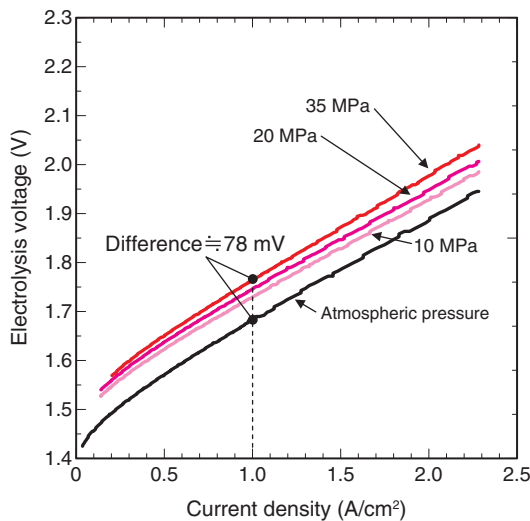


Fig. 11 Effect of H₂ pressure on IV curve (60°C)

due to hydrogen pressurization during high differential pressure water electrolysis. This behavior indicates that the relationship between the hydrogen pressure and voltage for the constant interfacial pressure-type shown in Fig. 10 matches that predicted by the theoretical formula for pressurization energy. The difference between the voltages at atmospheric pressure and 35 MPa was 78 mV at a current density of 1 A/cm², and this value is close to the theoretical concentration difference voltage of 84 mV. In addition, the IV curve trended at a constant slope up to the high current density of 2.3 A/cm², indicating that sufficient water is supplied to the catalyst in the high differential pressure water electrolysis cell.

Figure 12 shows the IV curve for each temperature at 35 MPa. The IV curve slope decreased as the temperature increased from 30°C to 60°C. The voltage efficiency at 1 A/cm² was 77% at 30°C, and increased to 84% at 60°C. This behavior was the same as that for water electrolysis⁽²⁾, indicating that the differential pressure had no effect on the relationship between temperature and membrane resistance.

Figure 13 shows the time series change in current

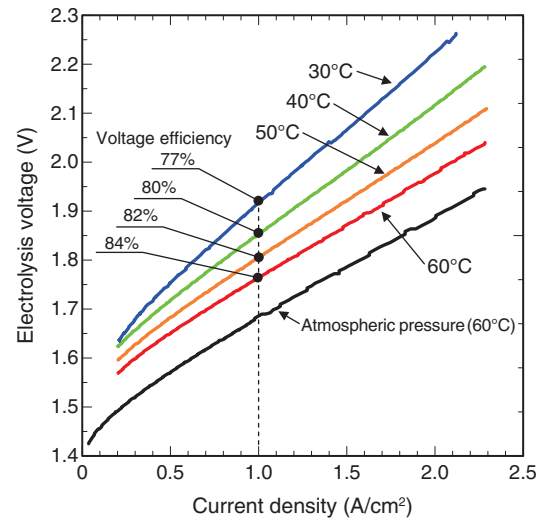


Fig. 12 Effect of temperature on IV curve (35 MPa)

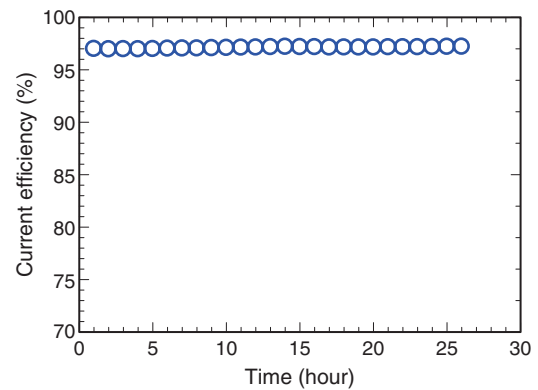


Fig. 13 Duration of current efficiency (integrated values for one hour, at 30°C)

efficiency. Water electrolysis has almost no hydrogen loss, so the current efficiency is approximately 100%. In contrast, the current efficiency at 35 MPa was approximately 97%. This matches the measurement results for the amount of hydrogen cross-permeation across the membrane, and confirmed that there is no loss other than cross-permeation across the membrane. In addition, there was also no drop in current efficiency over time, so it is thought that the proton exchange membrane shape was maintained and there was no deformation at a differential pressure of 35 MPa.

4.2.4. Evaluation of hydrogen quality

Figure 14 shows the pressure dependency of the oxygen concentration in the produced hydrogen. The oxygen concentration was approximately 33 ppm at atmospheric pressure, and decreased rapidly as the pressure increased, reaching approximately 5 ppm at around 1 MPa.

This behavior indicates that movement of oxygen from the anode to the cathode was suppressed due to the pressure difference. According to ISO 14687-2 Hydrogen fuel - Product specification⁽⁸⁾, the oxygen concentration must be 5 ppm or less, and high differential pressure water electrolysis was found to satisfy this concentration condition under steady-state operating conditions. To increase the purity of the produced hydrogen, water electrolysis had to react oxygen in excess of the specified concentration with hydrogen and exhaust it as water. This meant that the system had to be equipped with a reactor, and a process to remove the generated water after the reaction was also necessary. However, these are not needed for high differential pressure water electrolysis.

In addition, the amount of saturated steam in the high-pressure gas is inversely proportional to the pressure, and the moisture concentration that is several ten thousand ppm at atmospheric pressure and 30°C decreases to approximately 135 ppm at 35 MPa. The abovementioned hydrogen fuel product specification also prescribes a water content in the produced hydrogen of 5 ppm or less. However, the dehumidification energy can also be greatly reduced compared to water electrolysis.

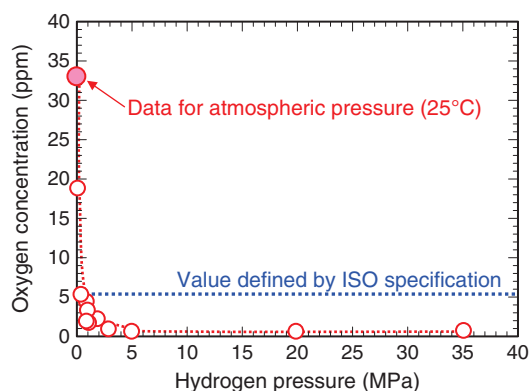


Fig. 14 Oxygen concentration in produced hydrogen (60°C)

These results confirm that a hydrogen refueling system using high differential pressure water electrolysis can simplify the device structure and increase efficiency compared to conventional systems.

5. Conclusion

The basic configuration of a high differential pressure water electrolysis cell was investigated, and tests were performed to confirm the differential pressure sustaining function and to evaluate electrolysis performance. The following conclusions were obtained.

- (1) Differential pressure of 35 MPa was sustained by the proton exchange membrane by reducing the surfacial pore size and using a powder sintered material with increased strength as the anode side current collector. In addition, hydrogen cross-permeating across the membrane due to the differential pressure was not reactive at the anode, so the basic configuration for a high differential pressure water electrolysis cell was established.
- (2) High differential pressure water electrolysis cell structures were investigated, and the constant interfacial pressure-type that maintains a constant interfacial pressure regardless of the hydrogen pressure inside the cell was found to have higher voltage efficiency than the pre-set interfacial pressure-type used by water electrolysis.
- (3) Hydrogen with a pressure of 35 MPa was produced using the theoretical concentration difference voltage. The current efficiency was 97% as a result of hydrogen cross-permeation across the membrane due to the differential pressure, and there was no change in this value over time.
- (4) It was found that when hydrogen with a pressure of 35 MPa is produced by high differential pressure water electrolysis, the oxygen concentration is much less than the ISO specification of 5 ppm, and an oxygen removal process is not necessary. Only dehumidification is required to achieve the specified hydrogen purity.

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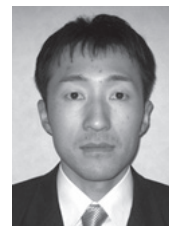
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