EFFECT OF COMPRESSION FORCE IN THE FABRICATION OF MEMBRANE ELECTRODE ASSEMBLY BY DECAL METHOD FOR PROTON EXCHANGE MEMBRANE WATER ELECTROLYZER

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Abstract

Proton exchange membrane water electrolysis (PEMWE) is a promising technique for producing high-purity hydrogen without using fossil fuels. The outstanding advantage of PEMWE is that it is combined with renewable energy sources such as solar, wind, etc. to produce green hydrogen. The most important part in a proton exchange membrane water electrolyzer is the membrane electrode assembly (MEA). The performance of PEMWE is mainly determined by the properties of the MEA. In this paper, the authors studied the preparation of the anode catalyst layer by ink spray technique on a teflon substrate in the fabrication of MEA by decal method. This is the most advanced method that allows the fabrication of MEAs that are both of good quality and easy to perform. The pressing conditions were studied, tested and evaluated, thereby providing the optimal pressing parameters for the fabrication of MEA and then applying MEA to the proton exchange membrane water electrolyzer, running tests at different current densities and achieving the minimum potential value of 1.708 V at a current density of 1 A/cm^2.

Keywords: *Hydrogen; PEMWE; MEA membrane electrode; $IrRuO_2$; electrocatalyst.*

1. Introduction

Currently, hydrogen is acknowledged as a sustainable, eco-friendly, and carbon-neutral energy carrier [1]. Among the several electrolysis technologies for hydrogen production, proton exchange membrane water electrolysis (PEMWE) has garnered significant attention for practical applications due to its advantages, including high hydrogen purity, rapid reaction time, and exceptional stability [2], [3]. The membrane electrode assembly (MEA) is a main part influencing the performance of an electrolyzer. A MEA consists of two layers of electrode catalyst materials, the anode and the cathode, symmetrically positioned across a proton exchange membrane (often a Nafion

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membrane) that serves as an electrolyte medium for the transit of H^+ protons from the anode to the cathode. The efficacy of PEMWE is contingent upon the characteristics of the MEA. The characteristics of the MEA will be influenced by critical factors like catalyst density, ionomer content, and particularly the process of MEA production. Two primary techniques exist for the fabrication of MEA: the catalyst layer fabrication on the diffusion layer (the CCS method) and the catalyst layer fabrication on the proton exchange membrane (the CCM method) [4]. Among them, the CCM method has been developed and popularized recently [6]. In this method, the catalyst material is directly coated on the Nafion membrane. The technique for applying the catalyst layer onto the CCM membrane has been refined and increasingly utilized recently [6]. In this method, the catalyst material is directly placed onto the Nafion membrane in this manner. The catalyst coating approach can be executed through screen printing, spray coating [7], electroplating [8], or sputtering [9] directly onto the membrane. This approach facilitates the production of MEAs with superior quality and high catalyst material consumption efficiency. This procedure necessitates contemporary equipment, primarily utilized in extensive laboratories.

A technique developed from the CCM method is the indirect catalyst coating method using decal (DTM) [6]. In this method, the catalyst material is coated on the decal (usually made of PTFE), then hot pressing with a membrane is performed to transfer the catalyst onto the membrane. MEA is formed by hot pressing again between the catalyst membrane and the two diffusion layers. This method can also produce MEAs with good quality like the CCM method, and at the same time is as easy to perform as the CCS method, making it very suitable for mass production of MEAs.

To date, there have been many studies on the fabrication of MEAs for proton exchange PEMWE. Doan *et al.* used the CCS method with different densities of IrO_2/TiO_2 catalysts and tested in a single PEMWE achieving cell potential of 2 V at a current density of 2.7 A/cm^2 using a Nafion® 115 (N115) membrane and an Ir density of 0.6 mg/cm^2 [10]. Lim et al. prepared MEA by the CCM method with a Nafion® 212 (N212) membrane, IrO_2 catalyst (Ir density: 1 mg/cm^2) and recorded the potential of a single PEMWE achieving 1.8 V at 3 A/cm^2 [11]. In addition, Stahler et al. reported a new method for preparing MEA using Ir density 2.1 mg/cm^2 and Pt density 0.4 mg/cm^2, cell single voltage of PEMWE achieved 2 V at 1 A/cm^2 current density [12]... Regarding the decal process, many studies have been trying to improve the performance of PEMWE, however, some problems still exist. First, the decal method causes the catalyst to transfer from the decal to the film unevenly or incompletely. In addition, it is necessary to find the optimal conditions for hot pressing parameters such as pressure and temperature... Second, in high

current regions, the microstructures of the catalyst layer must be designed to easily remove the product gas. Third, the catalyst ink must be prepared by dispersing an appropriate amount of catalyst and ionomer solution in the solvent mixture, promoting the utilization of the catalyst and proton conduction [14]-[16]. Hot pressing is an important step to bond the components of the MEA membrane electrode. The hot pressing process creates a bond between the gas diffusion layer with the catalyst layer and the Nafion membrane to form the membrane electrode. The contact area between the diffusion layer, catalyst layer and membrane is also known as the three-phase boundary area where electrochemical reactions occur and right on the surface of the catalyst particles. The optimal hot pressing process can improve the properties and structure of the three-phase boundary area. The three main parameters of the hot pressing process, including temperature, pressing pressure and pressing time, need to be optimally controlled during the hot pressing process. Currently, there are not many publications on the influence of hot pressing conditions on the properties of MEA membrane electrodes in PEMWE electrolyzers. Research groups mainly apply experience used for PEM fuel cells to prepare MEA for PEMWE electrolyzers. However, due to the different catalyst materials and membranes, the properties of the MEA will be greatly affected by the manufacturing process, and this is the core technology that needs to be studied.

In this study, we used ultrasonic spraying technique to fabricate the catalyst layer. More specifically, the Siansonic ultrasonic spraying device is equipped with a heating table with a temperature of up to 150°C. The catalyst ink under the effect of ultrasonic waves is sprayed in the form of a fine mist, adheres evenly and dries quickly on the substrate surface, thereby creating a catalyst layer with the desired thickness and structure. The catalyst layer was transferred onto the membrane by decal method and then hot pressed to form MEA. The hot pressing conditions were time and temperature studied and selected from previous studies and kept constant (pressing temperature 130^{o}C, pressing time 180 seconds), only the pressing force value was changed, the range of pressure value was referenced from previous studies of the author group [17]. The MEAs fabricated from different pressing forces were measured for thickness and analyzed for cross-sectional surface by SEM images to evaluate mechanical properties, then mounted on a single PEMWE electrolyzer with an area of 25 cm^2 to evaluate electrical properties, thereby providing the optimal pressing force to fabricate MEA for application to PEMWE proton exchange membrane water electrolyzers.

2. Experiments

2.1. Preparation of catalyst ink

The catalyst ink consists of anode catalyst is $IrRuO_2$ powder with particle size

10-20 nm (99.9%, American Elements-USA); cathode catalyst is Pt/C 20%wt powder with particle size 3-10 nm (FuelcellStore-USA) dispersed in a mixture of iso propanol solvent and 5% nafion solution (FuelcellStore-USA), to form the catalyst ink. To ensure the catalyst particles are evenly dispersed, the catalyst ink is stirred in an ultrasonic vibration device and stirred continuously according to the process: 5 times (10 minutes of ultrasonic vibration + 45 minutes of magnetic stirring) then stirred overnight for 15 hours.

2.2. Preparation of catalyst layer

$IrRuO_2$ anode catalyst ink was coated on the decal sheet (made of PTFE material; 0.3 mm thick, China), 20%wt Pt/C cathode catalyst ink was coated on the carbon paper diffusion sheet (AvCarb 1071 HCB, FuelcellStore-USA) using an ultrasonic spraying system (ULTRASONIC SPRAYING SYSTEM; Model: UC330; ultrasonic source with max. 5.5 W power, ultrasonic power control step (Control Step Size): 0.01 W, spray pressure 0.1 psi, nozzle moving speed 20 mm/s, syringe pump speed 1.5 mL/min, base temperature 50^{o}C), after each spraying, the catalyst layer was left to dry at room temperature, this process was repeated until the metal catalyst density on the decal sheet at the anode was 4 mg/cm^2 and on the carbon paper at the cathode was 2 mg/cm^2. Finally, the catalyst layers were dried at 80^{o}C for 12 hours

2.3. Fabrication of MEA

The procedure for producing membrane electrodes asembly using the decal method is outlined as follows: Commence with the initial hot pressing approach to transfer the catalyst layers from the decal sheet to the N-117 Nafion membrane (thickness 183 µm, Fuelcell store-USA). Subsequently, execute the second hot pressing technique between the membrane with the catalyst layer and the diffusion layer covered with cathode catalyst ink to fabricate the MEA. The summarized procedure is illustrated in Fig. 1.

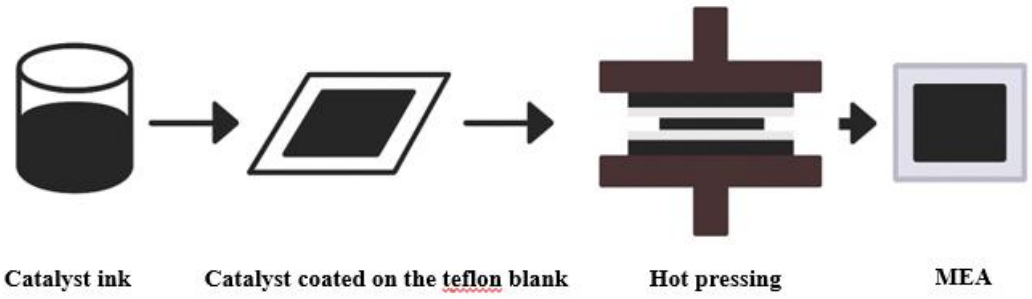

Fig. 1. MEA fabrication process by catalyst coating method on membrane.

This study maintained constant pressing duration and temperature parameters while

varying the pressing force to examine its impact on the properties of membrane electrodes produced via the hot pressing method. MEA electrodes were produced at a consistent pressing temperature of 130°C, a pressing duration of 180 seconds, and with variable pressing force values of 5, 10, 15, 20, 25, 30, and 35 kg/cm².

2.4. Characterization of MEA

The SEM method was employed to capture cross-sectional pictures of MEA membrane electrodes to examine the surface morphology of the catalyst layers and the internal deformation. This technique also facilitates the observation and measurement of the deformation thickness of the Nafion membrane. SEM investigations were conducted using a Jeol JSM 5800 scanning electron microscope. The deformation of the Nafion membrane, as observed in the SEM image post-hot pressing, was assessed using the percentage change in membrane thickness *R*, calculated using the formula:

$$R = \frac{T_0 - T_1}{T_0} \cdot 100\% \tag{1}$$

where T_0 represents the thickness of the Nafion membrane prior to pressing, and T_1 denotes the thickness of the Nafion membrane subsequent to pressing (μm).

The thickness of the MEA is ascertained by the mean of nine measurements taken at various positions on the MEA film utilizing a Mytutoyo thickness gauge. The deformation of MEA post-hot pressing is assessed by the percentage variation in film electrode thickness *H*, calculated as follows:

$$H = \frac{T_0 - T_1}{T_0} \cdot 100\% \tag{2}$$

where T_0 denotes the thickness of the MEA prior to pressing, and T_1 represents the thickness of the MEA subsequent to pressing (μm).

The MEA membrane electrodes (Fig. 2a), post hot pressing, were integrated into a singular PEMWE unit (Fig. 2b) to assess the electrical characteristics. The PEMWE unit was assembled and operated in the laboratory. The electrical characteristics of the individual PEMWE unit were assessed by recording the U-I curve on a 500 W electrolytic evaluation system (Model: WETS SE1K-PE, Scitech Korea, Korea) (Fig. 2c): the voltage of the single PEMWE unit was documented while varying current density values from 0.2 to 2.5 A/cm². The U-I polarization curve was utilized to calculate the slope coefficient a and the voltage at an electrolytic current density of 1 A/cm², which were then compared to determine the best hot pressing conditions for the MEA membrane electrode produced via the decal method.

Fig. 2. MEA membrane electrode (a); PEMWE single electrolyzer (b); 500 W electrolyzer evaluation system-Scitech Korea (c).

3. Results and discussion

3.1. Effect of compressive force value on Nafion membrane deformation in MEA

This study's MEA consists of a gas diffusion layer, a Pt/C cathode catalyst layer, a Nafion 117 membrane, and an $IrRuO_2$ anode catalyst layer. To elucidate the architecture of the MEA, the MEA was sectioned post-hot pressing, and the cross-section was examined using a scanning electron microscope (SEM). The SEM picture (Fig. 3) distinctly reveals the structure of the components of the MEA. At the center lies a Nafion membrane, with anode and cathode catalyst layers symmetrically positioned on either side. Adjacent to the cathode catalyst layer, a gas diffusion layer composed of carbon fibers is present. Consequently, the functionality of the catalyst layer significantly influences the characteristics of the MEA, as the quantity of proton channels enhances the rate of the oxygen generation reaction. This relationship is contingent upon the adhesion of the catalyst layers to the proton exchange membrane, which is substantially impacted by the pressure conditions during the hot pressing process.

Figure 3 presents the SEM image of the MEAs produced under varying press pressure conditions. At a low pressure of 5 kg/cm², the SEM image indicates that the adhesion between the catalyst layers and the Nafion membrane is markedly inadequate. Gaps emerge between the catalyst layers and the Nafion membrane across nearly the whole membrane surface. This demonstrates that the adhesion of the catalyst to the membrane does not occur at low pressure values. As the pressure value rises to approximately 10 to 15 kg/cm², the SEM image indicates an improvement in the bonding quality of the anode catalyst layer to the Nafion membrane. The majority of the anode catalyst layer sticks to the Nafion membrane, potentially enhancing the proton conduction pathways between the membrane and the catalyst layer. Nevertheless, upon examining the cathode catalyst layer, little voids persist between the catalyst layer and the membrane.

The presence of these gaps diminishes the adhesion of the catalyst layer to the membrane, potentially impacting the characteristics of the membrane electrode. Increasing the pressure levels to around 20 to 25 kg/cm² significantly enhances the adhesion of the catalyst layers to the Nafion membrane. The SEM images reveal that the catalyst layers are uniformly adhered throughout the full surface of the Nafion membrane, particularly the anode catalyst layer. This phenomenon can be attributed to the substantial pressure values that uniformly deform the entire membrane surface, facilitating complete contact between the catalyst surface and the membrane. Under thermal circumstances, the Nafion materials in the catalyst layer and on the membrane surface intermingle, creating proton-conducting linkages that enhance the adhesion of both material layers. The strong adhesion of the membrane to the catalyst layer significantly enhances the properties of the MEA membrane electrode. Upon elevating the pressure to around 30 to 35 kg/cm², the SEM image continues to exhibit a commendable degree of adhesion between the catalyst layers and the Nafion membrane. The connection is consistent over the entire membrane surface, with no gaps present between the catalyst layers and the membrane. Nonetheless, upon examination of the anode catalyst layers, fissures are evident in the catalyst layer. Cracks manifest across the entire catalyst layer along the membrane surface, particularly at a pressure value of 35 kg/cm². This may be due to the fact that the compressive force values used in this range were too large, so when hot pressing, the membrane material was deformed to the limit and the excess compressive force affected the catalyst layer. The structure of the anode catalyst layer itself is porous, so when subjected to large compressive forces, the catalyst layer easily cracks. The cracks that appear may reduce the connectivity of the proton exchange networks created by the ion conductors in the catalyst layer and affect the properties of the MEA.

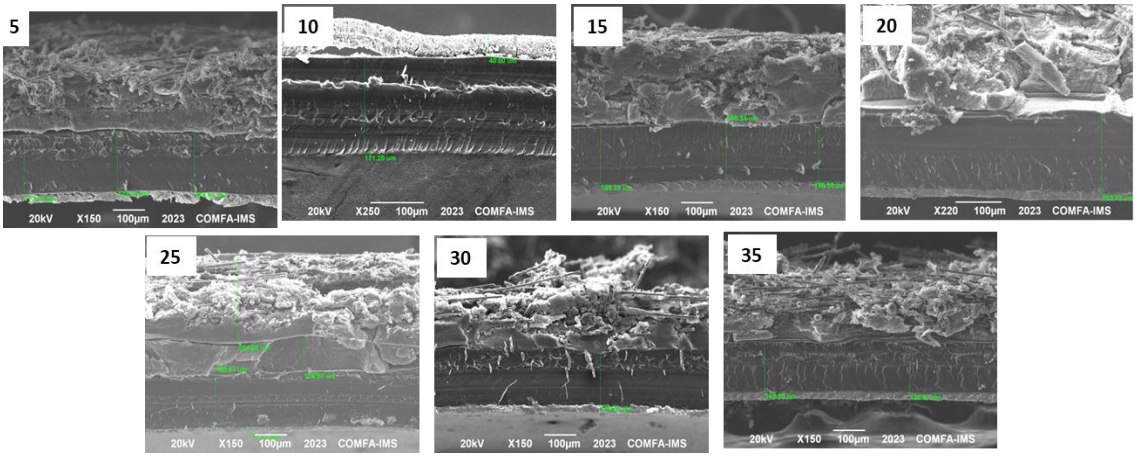

Fig. 3. SEM images of cross-sections of MEA fabricated by hot pressing at different pressing force values.

Tab. 1. Thickness of Nafion films in MEA hot pressed at different pressing force values

Pressing force (kg/cm^2)	5	10	15	20	25	30	35
Nafion film thickness (µm)	173.35	171.20	169.33	162.67	158.67	152.06	145.33
Percentage change in film thickness (%)	5.27	6.45	7.47	11.11	13.30	16.91	20.59

The deformation of the Nafion membrane post-hot pressing can be evaluated by measuring the membrane thickness directly from the SEM images. The commercial Nafion 117 membrane has an unpressed thickness of approximately 183 µm. Tab. 1 presents the membrane thickness values of the MEAs and the percentage change in membrane thickness R at various pressing force values. Generally, as the pressing force increases, the membrane thickness correspondingly diminishes. At modest pressing force values of approximately 5 to 15 kg/cm², the thickness of the Nafion membrane exhibits minimal variation, oscillating between 173.35 and 169.33 µm. As the applied force intensifies, the thickness of the Nafion membrane markedly diminishes. The Nafion membrane thickness is merely 145.33 µm under a maximum pressing power of 35 kg/cm². The percentage change in film thickness R exhibits a trend analogous to that of the film thickness post-pressing. At modest pressing force values of 5 to 15 kg/cm², the variation in film thickness is around 5.27 to 7.47%. Simultaneously, when augmenting the pressing force to approximately 20 to 30 kg/cm², the variation in film thickness ranges from 11.11 to 16.91%. Notably, with a pressing power of 35 kg/cm², the variation in film thickness attains 20.59%. This significant alteration can result in considerable film deformation. The film's structure theoretically comprises a polymer chain interspersed with diminutive water-containing spaces. Protons will be transmitted within the polymer chain and aqueous cavities. Consequently, excessive deformation of the film can result in the rupture of polymer chains, thereby diminishing the rate of proton propagation. The compression of the membrane leads to a decrease in water storage compartments, resulting in membrane desiccation that impairs proton conduction and may ultimately induce membrane fracturing, hence diminishing its longevity.

3.2. Effect of compressive force on MEA properties

Figure 4 illustrates the U-I polarization curves of a single PEMWE employing membrane electrodes produced through hot pressing at various pressing force values. Typically, an increase in current value corresponds to a proportional rise in voltage

values. The connection between voltage U and current I is nearly linear when pressing force values range from 5 to 35 kg/cm²; within this range, the control of electrochemical reactions occurring in the membrane electrodes is characterized as the activation process. The MEA produced under pressing force values of 10-25 kg/cm² exhibit favorable electrical properties, and their structure facilitates efficient mass transfer of reactants and products during electrochemical reactions, thereby not obstructing operational processes. For MEAs manufactured at pressures ranging from 5 kg/cm² to over 25 kg/cm², the polarization curve plots exhibited markedly elevated voltage values. This alteration in voltage is attributable to the increased internal resistance, which can be explained by the poor adhesion of the catalyst layers to the membrane when MEAs are compressed at elevated pressure levels.

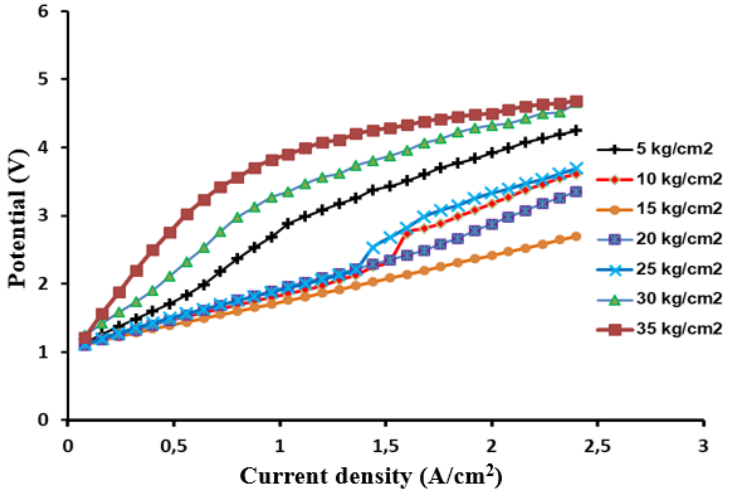

Fig. 4. U-I polarization curves of PEMWE single cell with decal-fabricated MEAs at different pressures.

The slope coefficient a and voltage values at a current density of 1 A/cm² for the electrolyzer with MEAs manufactured at varying pressing durations, were derived from the U-I polarization curves, and the findings are displayed in Tab. 2 and were compared in Fig. 5. The minimal slope coefficients were recorded between 0.372 and 0.375 at pressing force levels of 15 to 20 kg/cm². This may result from the MEAs manufactured at these pressing force levels exhibiting a strong adhesion between the catalyst layers and the membrane, while still allowing adequate deformation of the MEA to facilitate mass transfer processes effectively. At pressing force values below 5 to 10 kg/cm², the slope coefficient a ranged from 4.446 to 0.459. The elevated slope coefficient values may result from the inadequate adhesion of the catalyst layer to the Nafion membrane, as evidenced

by the SEM pictures. Significant compressive force levels between 25 and 40 kg/cm² yield MEAs with the highest slope coefficient a values. The elevation in the slope coefficient 'a' may result from significant deformation of the MEAs, characterized by a decrease in film thickness and the degradation of the catalyst layer SEM image of the MEA at these pressures has many cracks), which impacts the proton transport rate in the MEAs, leading to a decrease in the electrical efficiency of the PEMWE.

Tab. 2. Slope coefficient a value and electrolytic cell voltage at current density 1 A/cm² using MEA at different pressing force values

Pressure (kg/cm²)	5	10	15	20	25	30	35
Slope coefficient *a*	0.446	0.459	0.375	0.372	0.469	0.512	0.571
U (i = 1 A/cm² V)	2.688	1.953	1.708	1.877	2.023	3.268	3.815

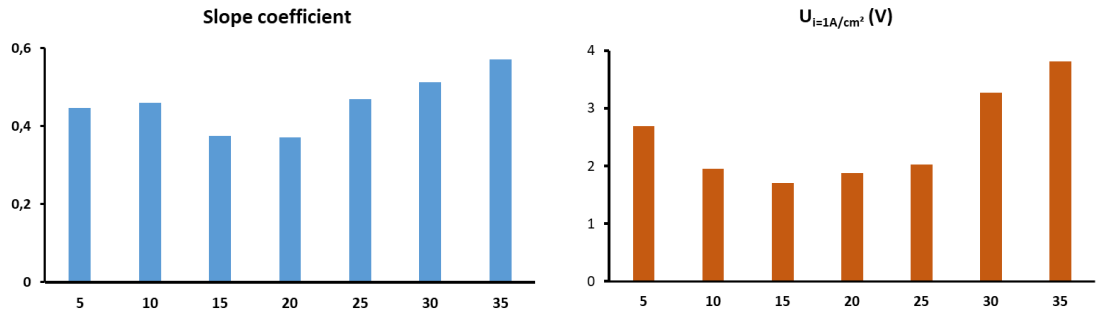

Fig. 5. Comparison of slope coefficient and voltage at current density 1 A/cm² using MEA at different pressing force values.

The electrical characteristics of MEAs produced under varying pressure conditions were compared using the voltage of the single electrolytic cell U (i = 1 A/cm²) at a current density of 1 A/cm² on the U-I polarization curve. Upon comparison of the U (i = 1 A/cm²) values, it is evident that the single PEMWE cell utilizing MEAs produced at pressure levels of 15 to 20 kg/cm² exhibits superior electrical characteristics, with U (i = 1 A/cm²) values ranging from 1.708 V to 1.877 V, which are comparable to contemporary studies on PEMWE cells, such as in the study by Huang and Cieluch used the CCM method to fabricate MEAs and tested on PEMWE single cell to achieve voltage values of 1.9 V [10] and 1.96 [13] at the current density of 1 A/cm².

4. Conclusion

The anode catalyst layer and MEA were successfully manufactured using the decal process. The impact of compressive force values on the characteristics of the MEA was examined and assessed. At low compressive force levels of 5 to 10 kg/cm², the adhesion between the catalyst layers and the Nafion membrane was inadequate; conversely, at high compressive force values ranging from 25 to 40 kg/cm², the MEAs experienced significant deformation and even compromised the integrity of the catalyst layer. The optimal compressive force range of 15 to 20 kg/cm² yielded MEAs with superior characteristics. Under these compressive force circumstances, the adhesion of the catalyst layers to the Nafion membrane was excellent, while also facilitating advantageous mass transfer processes in these MEAs, hence enhancing their characteristics. The MEAs, once integrated into a PEMWE single cell, were evaluated at several current densities, attaining a minimum potential of 1.708 V at a current density of 1 A/cm², which aligns with prior PEMWE research findings. This is a promising result. In the next study, the authors may apply the decal method to the preparation of the cathode catalyst layer to further reduce the contact resistance in the MEA, thereby increasing the efficiency of PEMWE, contributing to accelerating the fabrication of small and medium capacity PEMWEs for applications in teaching and small-scale hydrogen storage production.

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ẢNH HƯỞNG CỦA LỰC ÉP TRONG CHẾ TẠO ĐIỆN CỰC MÀNG BẰNG PHƯƠNG PHÁP DECAL CHO THIẾT BỊ ĐIỆN PHÂN NƯỚC MÀNG TRAO ĐỔI PROTON

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Tóm tắt: Điện phân nước sử dụng màng trao đổi proton PEMWE là một kỹ thuật hứa hẹn khi kết hợp với các nguồn năng lượng tái tạo như mặt trời, gió... để sản xuất hydro xanh có độ tinh khiết cao. Bộ phận quan trọng nhất trong PEMWE là điện cực màng (MEA) nơi xảy ra các phản ứng điện hoá phân tách nước. Hiệu suất của PEMWE được quyết định chủ yếu bởi tính chất của MEA. Trong bài báo này, các tác giả nghiên cứu chuẩn bị lớp xúc tác anốt bằng kỹ thuật phun mực lên đế teflon trong chế tạo MEA bằng phương pháp decal, đây là một phương pháp tiên tiến nhất hiện nay cho phép chế tạo MEA vừa có phẩm chất tốt vừa dễ thực hiện. Ảnh hưởng của điều kiện lực ép đến tính chất của điện cực màng MEA đã được nghiên cứu, thử nghiệm và đánh giá, từ đó đưa ra các thông số lực ép tối ưu để chế tạo MEA. Các kết quả thử nghiệm trong thiết bị điện phân PEMWE đơn cho thấy giá trị điện áp tốt nhất đạt được khoảng 1,708 V tại mật độ dòng điện làm việc 1 A/cm^2.

Từ khóa: *Hydro; PEMWE; điện cực màng MEA; $IrRuO_2$; chất xúc tác điện hóa.*

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