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ORIGINAL PAPER

Influence of hot-pressing temperature on the performance of PEMFC and catalytic activity

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Abstract The influence of hot-pressing temperature on catalytic activity and the performance of proton exchange membrane fuel cells (PEMFCs) was investigated using current–voltage (I–V) polarization, electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV). EIS provided detailed information on the contribution, from high to low frequencies, of internal impedance (R_s), interfacial impedance (R_{if}) and reaction impedance (R_{rxn}). The ohmic resistance of the cell (R_Ω) was estimated from the I–V diagram for comparison with the R_s and R_{if} impedances. The R_{if} is useful for diagnosing catalytic activity and interpreting PEMFC performance. A cell was hot-pressed at 125 °C (near the transition temperature of Nafion), an optimum temperature for lowest ohmic resistance and total impedance in response to a maximal catalyst-specific activity.

Keywords Proton exchange fuel cell · Electrochemical impedance spectroscopy · Interfacial impedance · Catalyst activity · Hot-pressing temperature

1 Introduction

Proton exchange membrane fuel cells (PEMFCs) have received increasing attention because of their high-energy efficiency in stationary, mobile and transportation applications [1–5]. A membrane electrode assembly (MEA) is the key power generating material of PEMFCs. Much effort has been expended in the evaluation of the MEA's diffusion layer, catalyst layer and membrane to improve PEMFC performance. Hot pressing is a simple way to assemble the anode, cathode and membrane for good interfacial contact between the electrodes and membrane. Hot-pressing has become a preferred way to prepare MEAs for PEMFCs. Some hot-pressing parameters (temperature, pressure and time) inevitably influence MEA preparation and the subsequent performance of the fuel cell. However, the effect of the parameters on the catalytic activity of MEAs and on PEMFC performance of has not been studied.

In the literature [6–14], MEA is commonly prepared by hot pressing under the following conditions: a temperature range of 120–195 °C, a pressure range of 2–200 kg cm^{-2} (196–19,614 kPa), and a duration of 50–300 s. It was reported that hot pressing might have led to the dehydration of the Nafion membrane, which inhibited fuel cell start-up and caused irreversible performance loss [15]. Dehydration of Nafion membranes becomes a problem when fuel cells operate at elevated temperatures (>100 °C) [16]. By contrast, in MEA preparation, a high temperature during hot-pressing ensures tight contact between the electrode and

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the membrane. However, theoretical understanding of the electrochemical mechanism is still lacking.

The I–V curve is a popular method for evaluating the performance of fuel cells. This technique has been extensively used to find the optimum parameters for fabricating a PEMFC with high electric power density. A typical I–V curve of fuel cells is divided into three regions (active, ohmic and mass-transfer limiting regions) to describe the polarization [17–20] based on the magnitude of the current density. Although this usefully illustrates estimated fuel cell performance, the I–V curve provides no detailed information on impedances from electrochemical kinetic effects on the MEA components.

Electrochemical impedance spectroscopy (EIS) is a powerful tool to explore the kinetic mechanisms [21]. In the performance of PEMFCs, EIS measures impedance spectra and indicates electrochemical mechanisms. In our previous study [22], we simulated EIS data and on the basis of this simulation, we proposed a set of equivalent circuit elements that were satisfactory for illustrating the electrochemical kinetics of fuel cells and the individual contribution of MEAs. The impedances in the equivalent circuit consist of three groups. Group I (in response to high frequencies) is the impedance at the catalyst–Nafion ionomer interface. Group II (in response to medium frequencies) is the impedance due to electrochemical reactions. Group III (in response to low frequencies) is the impedance from the adsorption/relaxation of CO. It is interesting that the interfacial impedance (Group I) was almost constant, regardless the increase of discharging current in the identical MEA [22].

In this study, we investigate the effects of the hot-pressing temperature on the catalytic activity and the performance of a fuel cell. In addition to the I–V technique, EIS technology is used to investigate the temperature effect and the electrochemical kinetics of a PEMFC at a variety of hot-pressing temperatures are discussed. We use a modified version of our previously-proposed equivalent circuit for investigating the temperature effect. The catalytic activity is evaluated by cyclic voltammetry (CV) for confirmation.

2 Experimental

2.1 Preparation of the electrodes/MEA

The catalyst-coated thin gas diffusion layer, called gas diffusion electrode (GDE, LT-120EW), was supplied by E-tek (E-TEK Division, New Jersey, U.S.A.). An electrocatalyst layer (30 wt% Pt/Vulcan XC-72 carbon black; 112 $m^2\ g^{-1}$ surface area; E-TEK) was applied to carbon cloth at 0.5 mg cm^{-2} Pt loading. The GDE was sprayed with a Nafion solution (containing 5 wt% Nafion ionomers), and dried overnight in an oven at 80 °C to control the uniform loading of Nafion (i.e., 1.0 mg cm^{-2}).

Nafion 112 (DuPont, Delaware, USA) was used as the proton exchange membrane. Prior to its service, each membrane was boiled in 10% hydrogen peroxide for 8 h to remove organic contaminations. It was then boiled in deionized water (DI) for 2 h, and boiled in 1 M sulfuric acid for 8 h to remove metallic impurities and ensure its H-form. The membrane was finally rinsed in boiling DI water for 2 h before use.

To fabricate a cell, one piece of the treated Nafion 112 was placed between two electrodes (anode and cathode were identical, with 0.5 mg cm^{-2} Pt loading and an active area of 10.89 cm^2), and they were placed in a hot press where the pressure was maintained at 50 kgf cm^{-2} and the temperature increased in stages of 75, 100, 125 and 150 °C for 3 min. A half-cell electrode was prepared by the same procedure, but only one electrode was used with the pretreated membrane in the hot press.

2.2 Evaluation of the electrodes/MEA

2.2.1 *Characterization of I–V polarization and estimation of impedances*

Two Teflon gaskets were used to hold the MEA, and they were sealed tightly by screwing on the graphite plates engraved with a serpentine flow channel. Two pieces of gold-plated stainless steel plates were in close contact outside the graphite plates for collecting the electric current. A test station (Beam Inc., Taiwan), consisting of a temperature controller, humidification chamber, mass flow meter, and electronic load (10 V, 60 A), was set up to operate the single cell and make necessary measurements. An I–V diagram was constructed by monitoring the current with stepwise loading of the voltage that decreased by 0.025 V from the open circuit potential in each step and was maintained at that level for 30 s. The measurement was continued until the cell voltage reached 0.25 V.

EIS was carried out under open circuit potential and monitored by a galvanostat/potentiostat (AutoLab PGS TAT30, ECO Chemie BV, Netherlands) equipped with a frequency response analyzer (FRA2 module). A two-electrode system was employed to conduct the EIS so that the original anode (fed with hydrogen gas) also acted as a reference (the dynamic hydrogen electrode (DHE)) that could be connected with the counter electrode to the electrometer. The cathode (fed with air) was connected to the working electrode of the potentiostat. The impedance spectra were measured potentiostatically, through input-perturbed sine waves with an amplitude of 10 mV within a frequency range of 20 kHz–10 mHz. Analysis and curve fitting of the data were done with Z-View software (Scribner Associates, Inc., North Carolina, USA).

While conducting I–V and EIS measurements, the atmospheric pressure was controlled during the operation of the PEMFC: humid hydrogen was fed to the anode (stoichiometry: 1.5) at 75 °C, and humid air was fed to the cathode (stoichiometry: 2.0) at 65 °C. All experimental runs were conducted thermostatically at 60.0 ± 0.1 °C.

2.2.2 *Measurement of catalytic activity using cyclic voltammetry (CV)*

The catalytic activity of the half-cell electrode (with identical anode and cathode) was measured by the potentiostat/galvanostat. Pt-foil (with 99.99% purity) served as the counter-electrode, and a saturated calomel electrode (SCE) coupled with the Luggin-Haber probe served as the reference electrode. During testing, nitrogen gas was continuously purged (with a flow rate of 150 ml min^{-1}) on the backside of the electrode, which was immersed in 0.5 M H_2SO_4 to avoid interference from oxygen reduction. The CV plot scan rate was 20 mV s^{-1} scanning forward from 50 to 1,400 mV (vs. standard hydrogen electrode, SHE) then inverse. Temperature was controlled at 60 ± 0.1 °C.

2.3 Characterization of the Nafion

A differential scanning calorimeter (DSC, Q-10 TA Instruments, Delaware, U.S.A.) was employed to characterize the membrane. Roughly 10 mg of pretreated Nafion was placed in an aluminum pan, purged with nitrogen gas and heated to 20 °C min^{-1}. Indium was used as a calibration standard. The DSC Standard Data Analysis Program (version 4.0, supplied by TA Instruments Thermal Analyst 2000 system) was employed to analyze the DSC traces.

3 Results

3.1 The performance of PEMFC

Figure 1 shows the polarization curves at 60 °C for a PEMFC in which the electrodes were fabricated under different hot-pressing temperatures. Comparing the curves in Fig. 1, we found that the performance of the PEMFC was determined by the hot-pressing temperatures: the performance improved when the hot-pressing temperature was increased from 75 to 125 °C). However, performance worsened when the hot-pressing temperature was increased from 125 to 150 °C. The cell hot-pressed at 125 °C displayed the best performance (revealing the lowest potential drop in all the regions of activation, ohmic and concentration polarizations).

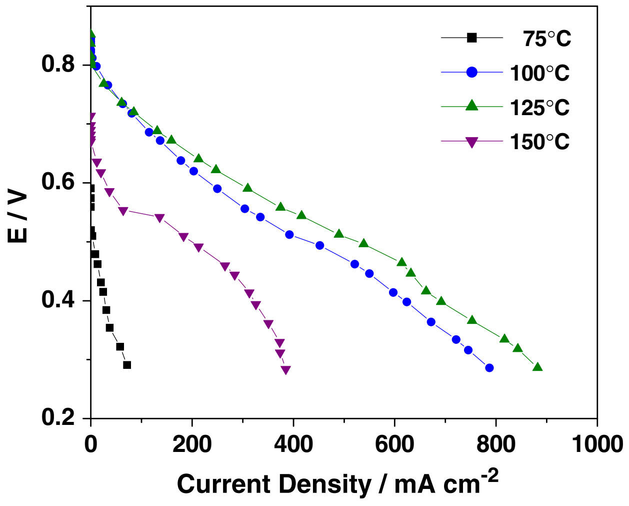

Fig. 1 Polarization curves of PEMFC at different hot-pressing temperatures

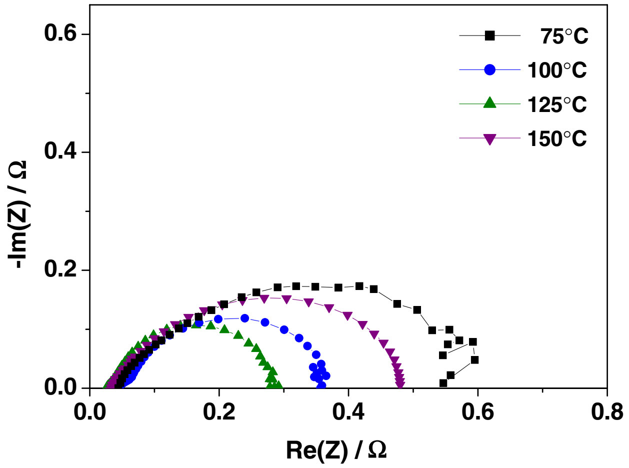

Fig. 2 Nyquist plots of PEMFC at different hot-pressing temperatures at open circuit potential

Figure 2 shows the electrochemical impedance spectroscopy with open circuit potential at 60 °C for a PEMFC fabricated at different hot-pressing temperatures. The Nyquist plot depicts a distorted capacitive semicircle on the upper plane. Point R_s, where the capacitive loop at the highest frequencies intersects with the horizontal axis, indicates the internal impedance of the cell. R_s remained constant (ca. 0.03 ohm), although the MEA was fabricated under various hot-pressing temperatures. The distorted capacitive loop in the positive imaginary plane (in the first quadrant) may be ascribed to the impedance from charge transfer in the electrochemical reaction in the three-phase zone. The diameter of the distorted capacitive loop related to PEMFC performance. The smaller the diameter of the loop, the better is the performance. The loop reduced in size as the temperature was increased from 75 to 125 °C. However, it grew in size when the hot-pressing temperature

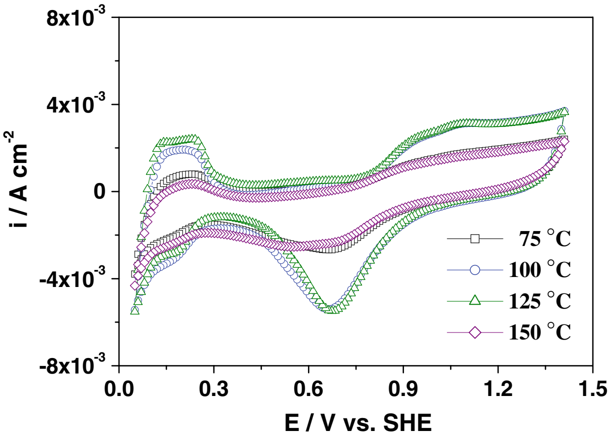

Fig. 3 Cyclic voltammetry for different hot-pressing temperatures by half-cell testing

was increased from 125 to 150 °C. Figure 2 clearly shows that the MEA fabricated at 125 °C had the smallest loop. The cell with the smallest loop performed best and a small loop implies that the impedance is less. On the basis of this fact, we presumed that the MEA fabricated at a hot-pressing temperature of 125 °C has the greatest catalytic activity. The higher catalytic activity may be ascribed to the greater surface area induced by the reactive catalysts.

3.2 Catalytic activity evaluated using half cell testing

The reactive surface area of the electrodes is usually evaluated using cyclic voltammetry. Figure 3 is the cyclic voltammogram for the half-cell electrode fabricated under different hot-pressing temperatures. The activity of the Pt catalyst is estimated by integrating the anodic peak that is responsible for the hydrogen adsorption at potentials ranging from 0.05 to 0.4 V (vs. SHE). The area for the peak, responsible for hydrogen adsorption, increases as hot-pressing temperature increases from 75 to 125 °C, and it decreases with additional increases, from 125 to 150 °C.

4 Discussion

4.1 Effect of hot-pressing temperature evaluated by EIS and I–V curves

In our previous study [22] on the mechanism of direct methanol fuel cells (DMFCs), we proposed a schematic model coupled with one set of equivalent circuits to interpret the possible impedances included in the performance of DMFC (i.e., internal (R_s), interfacial (R_{if}) and electrochemical impedances (R_{rxn})). The fabrication of a DMFC and a PEMFC is quite similar, and their reaction is governed by a similar mechanism, except that different fuels are fed to the anode (methanol for DMFC and hydrogen gas for PEMFC). The equivalent circuit formerly proposed for DMFC would benefit from a better understanding of the PEMFC mechanism, with some modification.

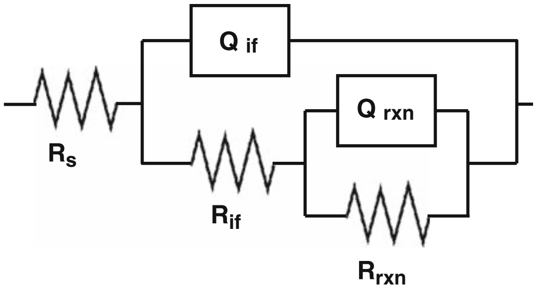

Fig. 4 The equivalent circuit for PEMFC

Figure 4 shows a modified equivalent circuit delineating the PEMFC mechanism. R_s is the internal impedance of the PEMFC. Two R–Q circuits, R_{if}/Q_{if} and R_{rxn}/Q_{rxn}, are responsible for the interfacial and electrochemical reactions, respectively. The constant phase element (CPE, Q) is used in place of the capacitance element (C), unlike the previous model, for a better fit between the experimental data and theoretical calculations, following similar, previous work [23].

The data of different impedances in the simulated equivalent circuit were evaluated and re-plotted against the hot pressing temperature, as shown in Fig. 5. We found that R_{total} (summation of R_s, R_{if} and R_{rxn}) was minimum at 125 °C. The profile of R_{total} seemed to be determined by that of R_{if} and R_{rxn}. The R_s arising from ionic and electronic resistivity in the PEMFC remained constant (0.0025 ohm cm^{-2}) regardless of the hot-pressing temperature.

The interfacial impedance (R_{if}) is thought to closely correlate with the reactive area of reactant-catalyst-

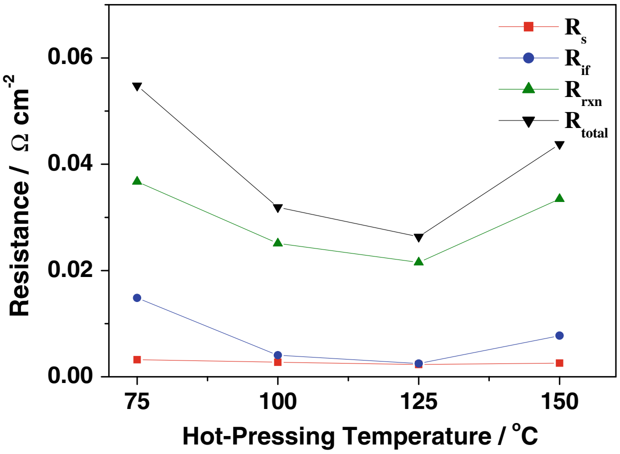

Fig. 5 A plot of simulated impedances from equivalent circuits with various hot-pressing temperatures

electrolyte interfaces, where electrochemical reactions take place. An increase in the reactive area leads to a decrease in the interfacial impedance. The relationship between electric resistance and resistivity is as follows:

$$R = \rho \frac{l}{A} \tag{1}$$

where ρ is the electric resistivity; l, the length; and A, the cross-sectional area of the materials. In Fig. 5, R_{if} was minimum at 125 °C. This implies that the Pt catalyst exposes a maximum reactive area to electrochemical reactions in the three-phase zones when the electrode has been hot-pressed at 125 °C. As a result, the impedance of electrochemical reaction (R_{rxn}) should be similar, and reduce to a minimum. Then, the overall electrochemical impedance can be resolved into R_{if} and R_{rxn}. Once the interfacial area of the catalyst is determined by the hot-pressing temperature, R_{if}, and consequently R_{rxn}, is fixed. In other words, the performance of a fuel cell is determined partially by the catalytic activity, and the catalytic activity is governed by its reactive area, depending on the hot-pressing temperature during cell fabrication. Advantageously, introducing R_{if} helps explain the contribution of the reactive interfacial area in the three-phase zones to the electrochemical reactions.

An attempt was made to understand the effect of hot-pressing temperatures on the electrochemical polarization of the cell, in particular, the reaction mechanism in the region of the three-phase interfaces. As mentioned earlier in Sect. 3.1, the electrochemical polarization of a cell includes three kinds of polarization: activation, ohmic and mass transport. The overall polarization in the single cell can be described in the following mathematic equation [19]:

$$\mathrm{E} = \mathrm{E}_0 - \mathrm{b} \log \mathrm{i} - \mathrm{i}\mathrm{R}_\Omega - \mathrm{m} \exp(\mathrm{ni}) \tag{2}$$

Here, the first term on the right (E_0) is the open circuit potential. The second (b log i) is the Tafel term corresponding to the voltage loss caused by activation; b is the Tafel slope. The third term, iR_Ω, is the linear potential drop term due to ohmic resistance (R_Ω); R_Ω is the resistance between the electrode and electrolyte. The final term, m exp(ni), is responsible for the potential loss due to concentration polarization; m and n are parameters having units of potential and the reciprocal of current density, respectively. Kim et al. [18] discussed the parameters m and n and suggested that they are functions of current density. In this study, we concentrated our attention on the linear region caused by ohmic polarization. The experimental conditions were delicately controlled to avoid interference arising from activation and concentration polarizations. By feeding the cells with sufficient concentrations of the reactants, we could ignore the contribution of concentration polarization. On the other hand, it is known that the activation process of a fuel cell is predominantly controlled by the kinetics of oxygen reduction reaction (ORR) on the cathode. The kinetics in turn depends on the characterization of the Pt catalyst. The contribution of activation polarization could be kept unchanged if only one kind of Pt catalyst was invariantly loaded. As previously mentioned, an experimental concern is that the effect of hot-pressing temperature on the polarization would be confined to the linear ohmic region.

The effect of hot-pressing temperature on linear polarization will now be discussed in greater detail. According to Eq. 2, R_Ω data could be evaluated using a linear regression technique to fit the ohmic region on the curves shown in Fig. 1. The slopes on the linear region of the curves are equal to R_Ω. Figure 6 is a plot showing the dependence of R_Ω on the hot-pressing temperature. The magnitude of R_Ω was the least at 125 °C. When a summation of R_s and R_{if} (estimated from Fig. 4) was co-plotted in Fig. 6 to compare with R_Ω, their magnitudes are approximate at constant hot-pressing temperatures. In other words, R_Ω evaluated from the linear region of the I–V curve could be further visualized as a summation of R_s and R_{if} through EIS investigation. Since R_s almost remained constant throughout the range of hot-pressing temperatures, R_{if} may provide a diagnosis of ohmic polarization in PEMFC performance.

Referring to Fig. 5, we find that the temperature-dependency of R_{total} is mainly determined by R_{rxn} and R_{if}. The magnitude of the component impedances decreases in the order $R_{rxn} > R_{if} > R_s$. This is based on the assumption that R_{rxn} plays a major role in the contribution of kinetics. In our previous discussion, we inferred that the interfacial impedance (R_{if}) directly correlates with the area of reactant-

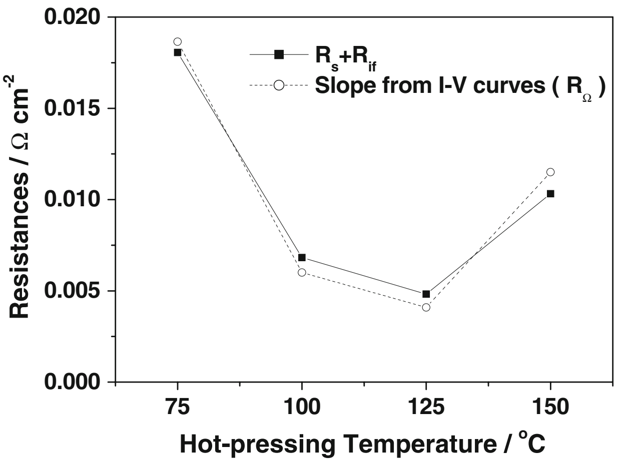

Fig. 6 Partial impedances ($R_s + R_{if}$) and ohmic resistance (R_Ω) of PEMFC estimated from EIS and I–V curve, respectively, as a function of hot-pressing temperature

catalyst-electrolyte interfaces, where electrochemical reactions take place. This implies that the magnitude of R_{rxn} is determined by R_{if}. Therefore, electrochemical kinetics is eventually governed by R_{if} and it provides a diagnosis of the catalytic activity in the performance of PEMFCs.

4.2 The catalytic activity evaluated by cyclic voltammetry

The dependence of catalytic activity on hot-pressing temperatures in the fabrication of half-cell electrodes could be reconfirmed by cyclic voltammetry. The catalytic activity is usually represented in the form of the electrochemical surface area (ECSA). The ECSA of the Pt-catalyst was estimated by integrating the broad peak (in the range of 0.1–0.4 V vs. SHE) responsible for adsorption of hydrogen in the cyclic voltammograms (Fig. 3). The charge area under the H_2 adsorption peak can be integrated by subtracting the nonfaradic charge from the anodic peak to evaluate catalytic activity. It is believed that the charge was 2.20×10^{-4} C cm^{-2}, which was required to adsorb atomic hydrogen on the smooth Pt surface. The ECSA of the catalyst can be calculated with the following equation [24]:

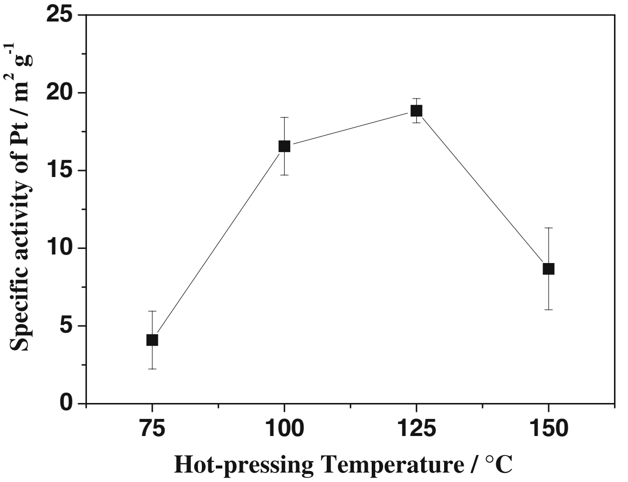

Fig. 7 A plot of the electrochemical surface area (ECSA) of Pt against the hot-pressing temperatures

$$\mathrm{ECSA}\ (\mathrm{m^2 g^{-1}}) = \frac{\text{Charge area}(\mu\mathrm{C\,cm^{-2}})}{10 \times 220(\mu\mathrm{C\,cm^{-2}}) \times \text{Catalyst loading (mg\,cm}^{-2})} \qquad (3)$$

Figure 7 displays the variation of ECSA for the Pt-catalyst with various hot-pressing temperatures. It increases rapidly from 4.09 to a maximum of 18.84 when the temperature is increased from 75 to 125 °C, and then decreases to 8.67 at even higher temperatures (150 °C). It is interesting that the profile of ECSA shown in Fig. 7 is the inverse of the profile of R_{total} (i.e., Fig. 6.). The maximum catalytic activity (the highest ECSA) but the minimum R_{total}, R_{if} and R_{rxn} are obtained at 125 °C. As mentioned earlier, the catalyst with greater interfacial area is more reactive and has less resistance. As a result, the catalyst hot-pressed at 125 °C is presumed to have the greatest interfacial surface and the highest catalytic activity.

4.3 Thermal characterization of the Nafion membrane (Nafion 112)

The purpose of the hot-pressing process is to fabricate a reactive MEA in which the components (the electrode and membrane) are tightly combined. The performance of the MEA is influenced by temperature, pressure, and duration. In this study, all the parameters were fixed within only a short range of temperatures (from 75 to 150 °C). The low variation in temperature caused no notable change in the catalyst and carbon properties. However, the structure and property of the polymer (Nafion 112) changed more. The thermal proprieties of Nafion were investigated using a differential scanning calorimeter (DSC).

Figure 8 shows the DSC curve for Nafion 112 in H-form with nitrogen gas purged. Two thermal relaxation peaks are above room temperature. The first one is at 50–140 °C and

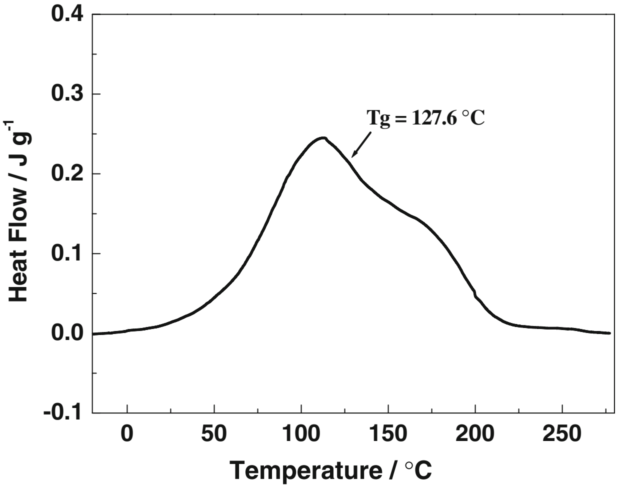

Fig. 8 The DSC data for Nafion membrane (N112)

the second (the weak endothermic peak) at 140 to 200 °C. According to some investigators, the first peak corresponds to water desorption from the ionic cluster and is considered to be the glass transition temperature (Tg) for the polymeric matrix [25, 26]. Through TA Universal Analysis, we found the Tg of Nafion 112 to be 127.6 °C. The second peak corresponds to the melting of crystalline regions. After further increasing the temperature to above 250 °C, the Nafion-H membrane degraded, according to another thermal analysis (Thermo-gravimetric Analyzer, TGA).

We conclude that the optimal hot-pressing temperature is 125 °C. According to DSC analysis, this temperature approaches the Tg of Nafion 112. It is logical to choose a temperature approaching the Tg of the selected membrane for hot-pressing in MEA fabrication. Under temperatures just a little less than the Tg of the membrane, the polymer was soft enough to combine tightly with the catalyst. This behaviour supplied the greatest interfacial area for the electrochemical reaction to proceed with the highest catalytic activity.

5 Conclusions

Hot-pressing plays an important role in the preparation of MEAs for PEMFCs. Temperature selection becomes a significant issue when pressure and duration are fixed. The optimum temperature is 125 °C, at a pressure load of 50 kgf cm^{-2} for 3 min. EIS provided detailed information on the contribution, from high to low frequencies, of internal impedance (R_s), interfacial impedance (R_{if}) and reaction impedance (R_{rxn}). The R_s remained invariant, but the R_{rxn} revealed a trend similar to R_{if}. The magnitude of R_{if} was significantly determined by the interfacial area between the Nafion and the catalyst where the electrochemical reaction occurred. An MEA fabricated at the optimum temperature (125 °C) reaches the minima of R_{if} and R_{rxn}. Minimal interfacial and reaction resistances imply the highest surface area of the catalyst that will result in a maximum of catalytic activity. Ohmic polarization fitted with linear regression on the linear region, yielding a separation of R_{if} from R_s. This separation confirms the major contribution of R_{if}. On the whole, R_{if} provides a useful diagnosis for the catalytic activity and performance of PEMFC. DSC analysis showed that the optimum hot-pressing temperature should be chosen at the temperatures approaching the glass transition temperature (Tg) of the membrane. Under optimal temperature, the catalyst and membrane combine best to provide a maximum electrochemical area at the interfacial interface, resulting in the highest catalytic activity.

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