



Short communication

## Reversible and irreversible degradation in fuel cells during Open Circuit Voltage durability testing

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## ABSTRACT

In this study reversible and irreversible voltage loss in a polymer electrolyte membrane fuel cell undergoing an open circuit voltage (OCV) durability test was studied. OCV durability testing is thought to promote chemical degradation of the electrolyte membrane material via radical attack and degradation of the catalyst layer. The results for degradation under constant relative humidity showed that voltage degradation rates measured in the first 20–50 h after polarization curve measurement consisted of a reversible, or transient, and irreversible component. A steady voltage decay rate became evident after 50 h of operation. Comparison to the voltage decay rates obtained from polarization curves showed that the steady voltage decay rate was representative of irreversible voltage loss due to irreversible changes in materials as shown by crossover and active surface area measurements. This study highlights the necessity of understanding the difference between reversible and irreversible voltage decay rates since the reversible decay rates were found to be much higher than irreversible decay rates.

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### 1. Introduction

Polymer electrolyte membrane (PEM) fuel cells are currently facing many technical challenges to commercialization, with some of the greatest challenges being with respect to reliability and durability issues. The causes of these reliability issues extend from sources such as material properties, defects, assembly issues, operating conditions, and maintenance procedures [1]. The degradation modes can be broadly categorized as being mechanical, chemical, or thermal in nature [1–3]. Ultimately the result of material degradation is an impact on fuel cell performance which is often gauged by metrics such as polarization characteristics or voltage degradation rates.

The measurement of voltage degradation rates provides practical information about the effects of extended fuel cell operation, while also being relatively easy to obtain. Voltage degradation rates have been reported for many durability experiments related to reactant contamination [4], load cycling [5], and freeze/thaw cycling [6,7], as well as general long-term fuel cell experiments [8,9]. Some voltage degradation modeling work has also been done

by Fowler et al. [10] who used a semi-empirical method for predicting degradation rates of fuel cells.

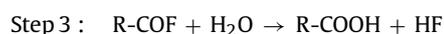
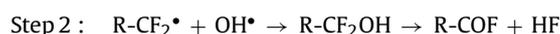
Generally there are two types of voltage degradation: reversible or irreversible, both of which cause similar observable voltage decay. Reversible degradation is often a result of transient processes where the loss in voltage may be reversed by changing the operating conditions (or with the aid of an ‘in situ’ recovery procedure) and consequently the cell performance may return to pre-degraded levels. An example of reversible degradation includes water flooding, which can reduce cell performance, although performance may be recovered by removing the excess water. Irreversible degradation includes irreversible changes to the fuel cell materials such as membrane thinning or loss of catalytic surface area from platinum migration or carbon corrosion. The type of mitigation strategy employed to minimize voltage degradation will be influenced by the cause and mode of degradation, specifically if the degradation is reversible or irreversible. Therefore, it is important to understand when observed voltage decay is due to reversible processes and when it is due to irreversible materials degradation. Accelerated testing can be useful for studying both types of degradation.

One accelerated durability experiment that has been used in the literature is an open circuit voltage (OCV) durability test or OCV ‘hold testing’ [11–14]. During this type of experiment the fuel cell is operated without drawing any load for extended periods of time, i.e. at open circuit voltage conditions. It is generally thought that chemical degradation, caused by reactant crossover, is most severe under

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these conditions since partial pressures of the reactant gasses are at their relative maximum values [15,16]. Recent modeling work has also shown that hydrogen crossover is at a maximum at OCV conditions [17]. It has been proposed that hydrogen or oxygen crossover may lead to the formation of peroxide species which are believed to facilitate the degradation of the electrolyte membrane [2,18–21]. This type of degradation generally leads to failure of the membrane electrode assembly (MEA) by compromising the integrity of the electrolyte membrane. In the case of DuPont™ Nafion® it has been proposed that carboxylic acid end groups left over from the manufacturing process may be susceptible to attack by these radical species [19] generated during fuel cell reactions. The proposed mechanism [19] is as follows:



It has been proposed that the radical species, such as hydroxyl radicals, are formed by the decay of hydrogen peroxide, which is an intermediate of the oxygen reduction reaction (ORR) [20], as well as through the reaction of permeated oxygen with hydrogen at the anode [19].

Some of the main observations in OCV durability testing attributed to chemical degradation are fluoride ion release into the effluent streams, membrane thinning leading to increased hydrogen crossover, open circuit voltage degradation [20–22] and platinum migration [11,23]. Fuel cell open circuit voltage is influenced by crossover (mainly hydrogen), whereby crossover species may create mixed potentials at the electrodes. This can be described by Eq. (1), where the open circuit voltage is made up of the Nernst potential,  $E_{\text{eq}}$ , as well as a term associated with losses due to hydrogen crossover,  $\eta_{\text{ix}}$ . The voltage loss from hydrogen crossover can be modeled using a Tafel-like expression as shown in Eq. (2) [24] where EAS is the electrochemically active surface area per geometric surface area ( $\text{m}_{\text{pt}}^2 \text{cm}_{\text{geo}}^{-2}$ ),  $i_0$  is the exchange current density per  $\text{cm}^2$  of platinum ( $\text{A cm}_{\text{pt}}^{-2}$ ) and  $i_{\text{crossover}}$  is the crossover current considered on a geometric area basis ( $\text{A cm}_{\text{geo}}^{-2}$ ). Inspection of Eqs. (1) and (2) reveals that an increase in hydrogen crossover or a reduction in active surface area should result in higher voltage loss and consequently lower open circuit potential however the current literature has not shown this experimentally

$$E_{\text{OCV}} = E_{\text{eq}} - \eta_{\text{ix}} \quad (1)$$

$$\eta_{\text{ix}} = \frac{RT}{F} \ln \left[ \frac{i_{\text{crossover}}}{\text{EAS} i_0} \right] \quad (2)$$

This paper examines reversible and irreversible voltage degradation in a fuel cell operated under open circuit voltage conditions. Irreversible voltage degradation is identified by comparing voltage degradation trends at OCV with voltage data from polarization curves. Further, irreversible voltage degradation will be modeled using crossover current and active surface area measurements and compared to experimental data which is unique to the literature. This will also allow the reversible, or transient, voltage losses to be identified. Finally, this study will demonstrate the difficulty in evaluating the magnitude of irreversible degradation over short periods of time under the conditions explored.

## 2. Experimental

### 2.1. Fuel cell OCV durability testing

The two fuel cells (Cell 1 and Cell 2) used in this study were water-cooled single cells made by Hydrogenics Corporation, each with a geometric active area of  $80.1 \text{ cm}^2$ . The cells were assembled using Gore™ PRIMEA® Series 55 catalyst-coated membranes (CCM) and proprietary gas diffusion layers (GDL) with microporous layer (MPL). The electrolyte membranes use an ePTFE reinforcement which increases mechanical stability of the membrane. Both cells were tested on a Hydrogenics FCATS™ test station which controlled temperature, humidity, and gas flows. The cells underwent a break-in period of 8 h prior to OCV durability testing. During break-in, the cell voltage was kept at approximately 0.6 V until stable operation was achieved.

Open circuit voltage durability testing was done at a cell temperature of  $90^\circ\text{C}$  and 100% RH. Cell temperature was regulated using a hot water bath and deionised water which flows hot water into the cooling channels in the anode and cathode plates. An external heating tape was also added to maintain cell temperature. Cells were operated vertically with gases and temperature regulating water entering at the top and exiting at the bottom. Temperature was controlled within  $\pm 1^\circ\text{C}$  of the set point.

### 2.2. Polarization curves

Polarization curves were taken at two different conditions. The first was at a cell temperature of  $80^\circ\text{C}$ , 100 kPa gauge, and anode and cathode relative humidities of 65% and 50%, respectively. The first polarization curve will be referred to as the “high pressure high temperature” or HPHT polarization curve. The second polarization curve was taken at a cell temperature of  $65^\circ\text{C}$ , 20 kPa gauge, and anode and cathode relative humidities of 65% and 50%, respectively. The second polarization curve will be referred to as the “low pressure low temperature” or LPLT polarization curve.

### 2.3. Crossover current and cyclic voltammetry

Crossover current (CC) and cyclic voltammetry (CV) measurements were performed using an EG&G Princeton Applied Research potentiostat/galvanostat model 273 and Coreware software. Humidified hydrogen was passed on the anode, and humidified nitrogen was supplied to the cathode. The cyclic voltammetry measurements were conducted with a sweep rate of  $20 \text{ mV s}^{-1}$ ; while the crossover current measurements were conducted with a sweep rate of  $2 \text{ mV s}^{-1}$ .

## 3. Results and discussion

The first fuel cell, Cell 1, underwent open circuit voltage durability testing continuously at 100% RH for a total of 380 h. Cell 2 was operated at 100% RH for its entire 900 h testing period. Fig. 1 depicts OCV curves of both cells over the testing period. Though data was taken every minute, the OCV curves only display data from every 5 h for clarity of presentation. Cell 1 was subjected to multiple breaks in OCV testing for balance of plant maintenance and scheduled stoppages where polarization curves and other electrochemical tests were conducted. Cell 2 was stopped very infrequently for diagnostics and maintenance. From Fig. 1 it can be seen that both OCV durability experiments produced similar a voltage trend between 0 and 380 h of operation.

At the beginning of testing or after an interruption, such as polarization curve measurement, the open circuit voltage beings at some

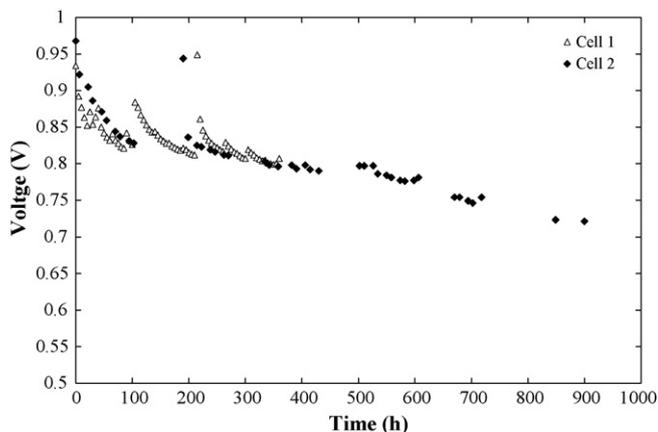


Fig. 1. Open circuit voltage durability data over the testing period.

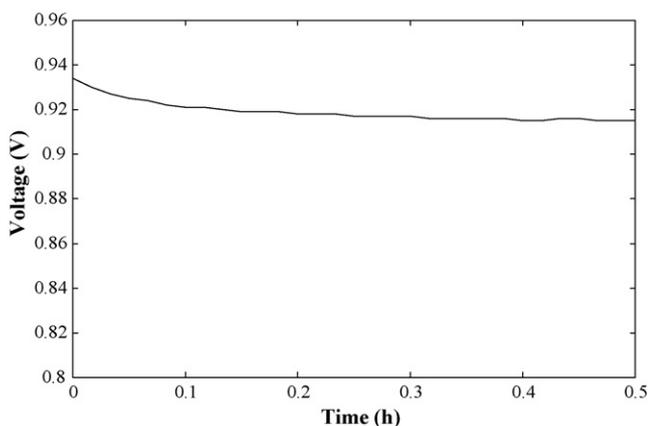


Fig. 2. Stability of OCV on small time scales.

relatively high level. For example at 0 h the potential of both cells range between 0.93 and 0.97 V which is a typical OCV for a fuel cell of this configuration. These initial voltages seem stable for periods on the order of minutes as shown in Fig. 2, varying by less than 2 mV between 15 and 30 min of operation. However, as shown in Fig. 3, when examined on larger time scales the voltage can be observed to be decaying relatively rapidly in an exponential fashion until it

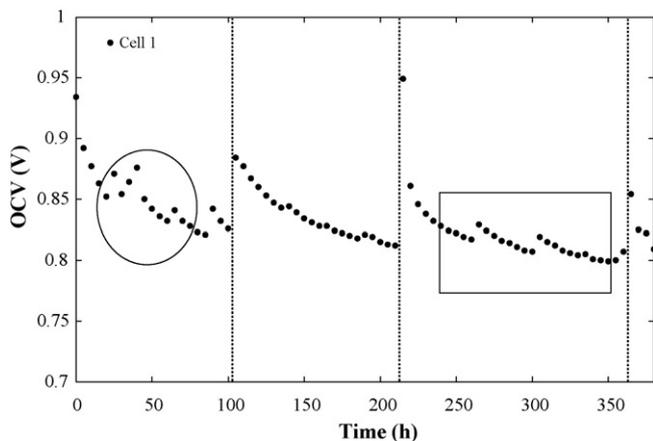


Fig. 3. OCV durability test data 0–380 h, 100% RH operation Cell 1. Dotted lines represent polarization curve measurements, interruptions for maintenance are shown within the circle, and voltage recovery due to temperature excursions are shown within the box.

eventually approaches a more steady decay trend which may take many hours to establish. The steady decay trend was identified with the voltage data from Cell 2 which was not interrupted as frequently as Cell 1 and hence is considered to have a fully stable degradation trend free of transient effects. The exponential decay region will be termed the transient decay period while the latter period will be referred to as the steady decay period. This is an important observation because it demonstrates that error may be introduced to voltage degradation data for short experiments if they fall in the transient period.

Fig. 3 also clearly shows voltage recovery phenomena caused by three types of interruptions: polarization curve measurement, maintenance, and temperature excursions. These periods when polarization curves were measured are marked with dotted lines while interruptions for maintenance can be seen encircled in the data segment from 0 to 100 h as sharp voltage spikes, finally cell temperature excursions can be seen in the data after 100 h as smaller jumps in voltage followed by a relatively smooth decay as marked within the 'square'. In all cases interruptions resulted in a temporary voltage recovery which returned to the overall steady decay trend with time. The recovery phenomena also raise an important issue of what it means to report a voltage degradation rate. Ideally, voltage degradation rates would represent irreversible changes to fuel cell materials however if part of the voltage loss is recoverable or transient then this specific measurement losses some meaning. From Figs. 1 and 3 it would seem reasonable to estimate an irreversible voltage degradation rate from the steady decay period however some justification is needed.

Eq. (2) suggests that irreversible changes to the membrane, leading to increased crossover, as well as the catalyst layer, leading to reduced electrochemically active surface area, will result in an irreversible change in OCV. During the testing of Cell 1 diagnostic tests such as crossover current, electrochemically active surface area, and polarization curves were carried out after 0, 100, 200, and 360 h of operation. CC and EAS results are given in Table 1. Cell 2 was not tested in this way in order to limit the number of interruptions. The results show that over time CC generally increased, which is consistent with the literature cited above, and this is caused by irreversible material degradation processes such as membrane thinning or pinhole formation. The EAS decreased with operational life, which is possibly as a result of irreversible contact loss between the electrolyte membrane and the catalyst layer, carbon corrosion, or from platinum migration as suggested in the literature [11,13,23]. Some platinum migration was observed in the membranes after degradation during this study. The effect of the irreversible decrease in EAS and increase in CC can be seen in the polarization curves as shown in Fig. 4 which show an overall downward translation with testing time.

The rate of irreversible degradation was estimated using three sources: (1) the HTHP polarization curves, (2) the LTLTP polarization curves, and (3) through application of Eqs. (1) and (2) using CC and EAS data. Since the measurement of polarization curves causes temporary voltage recovery and the short measurement times it is believed that the OCV measurements taken for the curves will only be impacted by irreversible changes in the materials and be free of reversible effects. The predicted rate of voltage degradation

Table 1  
Crossover currents and electrochemical surface areas for Cell 1

Time (h)	EAS normalized to 0 h	CC (mA cm <sup>-2</sup> )
0	1.00	2.40
100	0.77	2.31
200	0.86	2.58
361	0.23	3.17

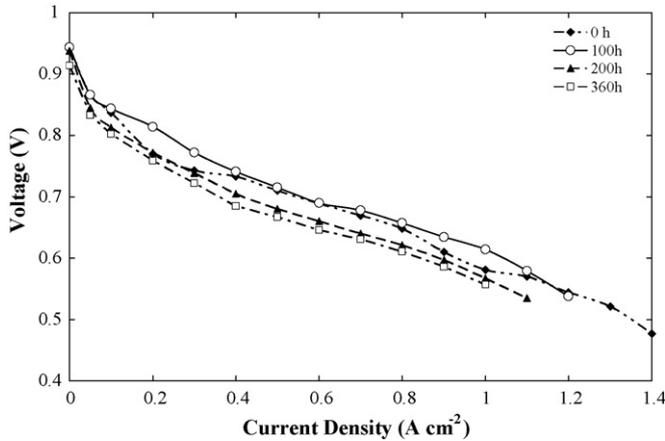


Fig. 4. 'High pressure high temperature' polarization curves for Cell 1.

from CC and EAS data was estimated by rearrangement of Eq. (1) for two data points at times  $t_1$  and  $t_2$ . Thus the amount of voltage degradation,  $\Delta E_{OCV}$ , can be calculated from the following equation.

$$\begin{aligned} \Delta E_{OCV} &= E_{OCV,t_2} - E_{OCV,t_1} \\ &= (E_{eq} - \eta_{ix})_{t_2} - (E_{eq} - \eta_{ix})_{t_1} = \eta_{ix,t_1} - \eta_{ix,t_2} \end{aligned} \quad (3)$$

Substituting Eq. (2) into Eq. (3) gives

$$\Delta E_{OCV} = \frac{RT}{F} \ln \left[ \frac{i_{crossover,t1}}{EAS_{t1} i_0} \right] - \frac{RT}{F} \ln \left[ \frac{i_{crossover,t2}}{EAS_{t2} i_0} \right] \quad (4)$$

Assuming that the exchange current density remains constant and simplifying:

$$\Delta E_{OCV} = \frac{RT}{F} \ln \left[ \frac{i_{crossover,t1}(EAS_{t2})}{(EAS_{t1})i_{crossover,t2}} \right] \quad (5)$$

The estimated voltage degradation rates from polarization curves and from application of Eq. (5) are summarized in Table 2. The average rate of irreversible voltage degradation calculated from OCV points of the polarization curves are  $0.083 \text{ mV h}^{-1}$  for the HTHP and  $0.141 \text{ mV h}^{-1}$  LPLT polarization curves. The estimated rate of voltage decay from the Eq. (5) was an average decay rate of  $0.146 \text{ mV h}^{-1}$ . The estimated irreversible decay rates are similar and within the same order of magnitude. The voltage degradation rates reported for this study are higher than those typically reported in the literature which range from  $0.09$  to  $0.001 \text{ mV h}^{-1}$  for cells degraded by various mechanisms [10]. Cleghorn et al. [8] report a degradation rate between  $0.004$  and  $0.006 \text{ mV h}^{-1}$  at operational conditions of  $800 \text{ mA cm}^{-2}$  and a similar type of membrane used in this study. The data by Paik et al. [14] shows a degradation rate of approximately  $0.016 \text{ mV h}^{-1}$  using a similar membrane and test conditions found in this study (although this number is low due to recovery phenomena in the presented data). The conditions used in this study represent accelerated ageing conditions so a relatively high rate of degradation is expected, and was observed.

The average decay rate during the steady decay portions of the voltage degradation curves for Cell 1, estimated by taking the

Table 2

Comparison of irreversible degradation rates from polarization curves, durability data, and calculated

Open circuit irreversible voltage degradation rate ( $\text{mV h}^{-1}$ )	
High pressure/temperature polarization curve (HPHT)	0.083
Low pressure/temperature polarization curve (LPLT)	0.141
Proposed model	0.146
OCV durability data	0.089

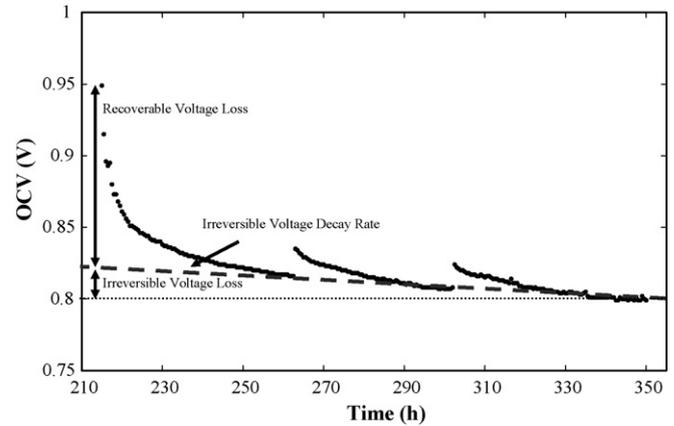


Fig. 5. Reversible and irreversible degradation as well as irreversible degradation rate for Cell 1.

final data points at the end of each segment, was calculated to be  $0.089 \text{ mV h}^{-1}$  which shows good agreement with the polarization and modeled data. In comparison, voltage decay rates calculated over the first 20 h after a recovery phenomenon, during the transient decay period, can be as high as  $5.8 \text{ mV h}^{-1}$ .

These results emphasize that the rate of voltage change during the transient period, which is higher than the degradation rate in the steady decay period, should not be mistaken for voltage degradation caused by irreversible changes in the materials. The cell used in this study was able to recover most of the voltage loss after drawing current confirming that this was this observation was composed of transient losses unrelated to material degradation. Had the transient voltage degradation been permanent it would have been evident in the polarization curves given the magnitude of the transient voltage loss. The identification of the recoverable and irreversible voltage loss as well as the irreversible voltage decay rate is illustrated in Fig. 5.

Though identification of the exact mechanism responsible for reversible voltage degradation is out the scope of this study, there are a number of processes that could be responsible. Recent work with direct methanol fuel cells has shown that platinum oxidation may be a cause of reversible voltage degradation [25]. Inspection of Eq. (2) reveals that a loss of catalytic surface area as a result of a reversible process would increase the overpotential. It was also observed during the study reported here that a short drop in cell temperature, and the effect this would have had on water content, played a significant role in voltage recovery. The cell temperature was controlled at  $90^\circ\text{C}$  by a hot water bath throughout the experiment. When refilling the water bath with fresh water the bath temperature would drop causing a consequential drop in cell temperature of approximately  $3^\circ\text{C}$  lasting between 45 and 60 min, a short time considering the time scale of the entire experiment. However, since inlet gas streams were at 100% RH and  $90^\circ\text{C}$ , a lower cell temperature would conceivably cause water to condense in the cell, specifically in the GDL, catalyst layer, or membrane. Fig. 6 shows the cell temperature during one such incident and the voltage response at the same time.

A lower cell temperature will naturally increase the open circuit voltage as described by the Nernst equation. However, temperature change cannot completely describe all of the voltage recovery experienced by the cell. It is also conceivable that when water condensed in the cell it could have caused the GDL/MPL to temporarily flood, pinholes to fill, or cause membrane swelling to close pinholes. This would increase resistance for hydrogen to be transported from the channel and through the MEA to the cathode resulting in lower overpotentials from crossover, and result in a higher OCV as

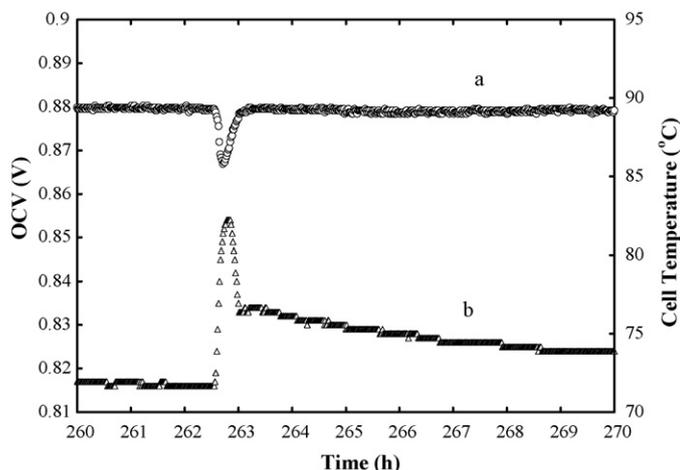


Fig. 6. (a) Cell temperature fluctuation due to waterbath refill and (b) voltage fluctuation for Cell 1 between 260 and 270 h.

indicated by Eq. (2). The return to steady conditions may take some time because the driving force for water removal is not high when using fully saturated gasses. This possibility and the study of reversible process in fuel cells requires further study.

In light of the sometimes large impact of reversible processes on the perceived degradation rate of fuel cell voltage under the stated conditions it seems necessary to include a reversible degradation term in models of OCV by adding an additional term to Eq. (1)

$$E_{OCV} = E_{eq} - E_{Reversible} - \eta_{ix} \quad (6)$$

where  $E_{Reversible}$  is related to the voltage drop caused by reversible processes.

#### 4. Conclusions

In this study the extent of reversible/transient and irreversible voltage degradation was examined for a single cell open circuit voltage durability test. It was observed that during such testing the voltage would initially begin high, over 0.93 V, and then fall with time. Over the first 50 h this initial voltage degradation would be rapid followed by a slower degradation rate after 50 h. These were referred to as the transient and steady decay periods, respectively. To determine the irreversible voltage decay rate, OCV measurements from polarization curves was used. Further, irreversible changes in crossover current and electrochemically active surface area were measured and used to calculate a theoretical irreversible voltage decay rate. The results indicated that irreversible voltage loss ranged between 0.083 and 0.146 mV h<sup>-1</sup>. These compared well with the voltage decay estimated from the steady decay period. This shows that the steady decay period is reflective of the irreversible changes in the materials with time. In contrast, decay rates estimated from the transient period were as high as 5.8 mV h<sup>-1</sup>.

The exact nature of the processes leading to voltage recovery was not studied however it was observed that cell temperature, and the effect it had on MEA water content, played a role. It was postulated that the changes in the state of water caused by temperature changes may be the cause. Future studies will attempt to confirm the specific nature of the physical processes taking place in this transient period. Overall the results show that care must be taken when reporting voltage degradation data to be able to identify how much is reversible or irreversible. In terms of modeling it may be useful to include a reversible degradation term into voltage degradation models.

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