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Development of an Accelerated Stress Test Protocol for Rapid Catalyst Degradation Evaluation in Polymer Electrolyte Membrane Fuel Cell Stacks

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Keywords

PEMFC; Catalyst degradation; Accelerated stress test

Abstract

As polymer electrolyte membrane fuel cell (PEMFC) durability targets continue to increase, rapid and reliable evaluation of stack durability becomes increasingly important. Conventional catalyst accelerated stress test (AST) methods are primarily designed for single-cell evaluation using a potentiostat, limiting their direct application to stacks. In this study, a catalyst AST method applicable to stacks is investigated using load cycling. A three-cell stack is subjected to four durability protocols with different voltage ranges, operating temperatures, and oxidants, and the resulting performance and catalyst degradation are evaluated. Increasing the operating temperature by 10 °C accelerates the performance degradation rate by 36%. The use of oxygen as the oxidant with an increased upper potential further accelerates the degradation rate by 85%. When the temperature increases by 10 °C, oxygen is used as the oxidant, and both the upper

and lower potential limits are extended, the degradation rate increases by more than twofold. These results indicate that Pt oxidation promoted by increased oxidant concentration and upper potential, together with Pt dissolution associated with the applied potential range, plays an important role in accelerating catalyst degradation. The proposed load-based AST provides a practical approach for rapid catalyst durability evaluation at the PEMFC stack level.

1. Introduction

Proton Exchange Membrane Fuel Cell (PEMFC) is recognized as the most suitable energy conversion device for next-generation zero-emission electric vehicles (ZEV). However, to expand the commercialization market of PEMFC, cost reduction and lifespan extension are essential [1, 2]. The Department of Energy (DOE) has set cost targets for PEMFC stacks at \$40/kW for light-duty vehicles (LDVs) by 2025 and \$80/kW for heavy-duty vehicles (HDVs) by 2030, with ultimate goals of \$30/kW and \$60/kW, respectively. Most of the cost reduction is expected to result from mass production-driven cost-down efforts. As production scales up, the cost proportion of components like the gas diffusion layer (GDL) and bipolar plates in the stack decreases. However, the price of Pt, a precious metal used as a catalyst, does not decrease in the same manner, leading to an increase cost proportion.

Therefore, in PEMFCs, there is a trend to reduce the Pt loading to cut costs. For LDVs, the goal is to reduce Pt loading to 0.125 mg/cm², and the cathode loading to 0.1 mg/cm² by 2025. For HDVs, the target is 0.3 mg/cm² by 2025, with an ultimate target of 0.25 mg/cm². In the research and development of PEMFC electrodes, studies into low-Pt and non-Pt are actively underway due to the high cost of Pt. Alloy catalysts containing Pt have been considered as a strategy for reducing Pt usage, and Pt-Co/C catalysts have recently been widely used as catalysts that satisfy both performance and durability requirements [3-7]. However, reducing the catalyst loading results in decreased performance and durability of the catalyst. As a result,

extensive research has been conducted to improve both the performance and durability of catalysts [8-12].

Recently, the durability of PEMFC has been increasingly emphasized, as the required durability improvements have risen with focus on large truck ZEVs. The lifespan target for light-duty vehicles (LDVs) is 8,000 hours by 2025, while the target for heavy-duty vehicles (HDVs) is 25,000 hours by 2030, with an ultimate goal of 30,000 hours. The lifespan target for HDVs is more than four times higher than that for LDVs [13]. Since durability studies inherently require extended testing periods, these increasingly stringent lifetime targets further highlight the need for rapid and reliable durability evaluation methods.

PEMFC MEAs consist primarily of a polymer electrolyte membrane and catalyst layers, and various durability evaluation methods have been developed to investigate the degradation behavior of each component. For example, open-circuit voltage (OCV) holding tests are commonly used to evaluate the chemical durability of the polymer electrolyte membrane, whereas wet/dry cycling tests are employed to assess its mechanical durability [14, 15]. Catalyst degradation is typically evaluated using potential cycling protocols, while potential holding tests are widely used to investigate the durability of carbon support materials. In addition to these component-specific durability tests, drive-cycle-based durability tests have been developed to evaluate PEMFCs under dynamic operating conditions representative of actual vehicle operation [16-22].

Because fuel cell vehicles are subjected to continuously varying power demands during operation, actual driving conditions inherently involve dynamic load changes. Consequently, the degradation of PEMFC stacks under practical operating conditions is not solely determined by membrane degradation but is also strongly associated with catalyst degradation induced by repeated load cycling. In particular, repeated changes in cell voltage and current density during load cycling can accelerate catalyst degradation through processes such as Pt dissolution,

migration, redeposition, and particle growth, ultimately leading to a loss of electrochemically active surface area and cell performance. Therefore, an accelerated stress test (AST) intended to predict the durability of PEMFC stacks under practical operating conditions should reproduce the dominant degradation mechanisms associated with dynamic load cycling.

However, conventional catalyst ASTs have several limitations when applied to PEMFC stacks. Standardized catalyst durability protocols, including those developed by the U.S. Department of Energy (DOE), generally employ potential cycling using a potentiostat and are primarily designed for single-cell [23]. Therefore, their direct application to PEMFC stacks is challenging. Moreover, the durability behavior of a PEMFC stack can differ from that observed in a single cell because stack durability is influenced not only by the MEA materials but also by the stack configuration and system-level operating conditions. Consequently, durability results obtained from single-cell evaluations may not fully represent the degradation behavior of an actual PEMFC stack. Therefore, stack-level durability evaluation is essential for accurately assessing the practical durability of PEMFC systems.

In this study, a stack-level catalyst AST was developed using a load cycling method. A three-cell PEMFC stack was used to apply four different durability test protocols using an electric loader, with variations in the voltage range, operating temperature, and oxidant. The degree of performance degradation acceleration under each protocol was investigated. In addition, cathode catalyst degradation was characterized through electrochemical measurements and post analyses using transmission electron microscopy (TEM) and X-ray diffraction (XRD).

2. Experimental

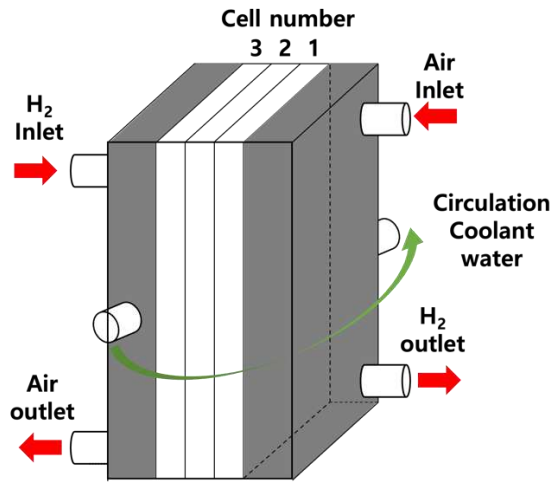


Figure. 1. Schematic diagram of 3 cell short stack of PEMFC

2.1 Stack specification

In this study, a water-cooled stack with an active area of 25 cm², consisting of three cells, was used. Each stack was clamped with carbon-based bipolar plate (five-channel, serpentine), commercial MEA (Vinatech, Korea), and a GDL (Sigracet 39BB, SGL Carbon, Germany). The stack temperature was maintained using a chiller (model name), and the gas flow were controlled by a test station (CNL Energy, Korea).

2.2 Durability test

The stack durability test was conducted by applying load variations to the stack using an electric loader to control voltage changes. Each durability test protocol was set with different voltage ranges and temperature conditions. The specific conditions for each protocol are shown in Table 1. Each protocol was conducted until the DOE catalyst AST target condition (<30 mV loss at 0.8 A/cm²) was reached. Protocols A and B were conducted using air under a relatively low potential range. In contrast, Protocols C and D were accelerated protocols using oxygen, with a high upper potential of 0.95 V applied. Protocol D was the most severe protocol, employing the widest potential range and a high temperature of 90 °C.

Table 1. Durability test condition.

Test protocol	Voltage range (each cell)	Temperature	Cathode gas	Relative humidity
Protocol A	0.6-0.9 V	80 °C	Air	100%
Protocol B	0.6-0.9 V	90 °C	Air	
Protocol C	0.6-0.95 V	80 °C	O ₂	
Protocol D	0.4-0.95 V	90 °C	O ₂	

2.3 Stack characterization

2.3.1 I-V and mass activity

The I-V curve was measured using an electronic load while supplying H₂ to the anode at a stoichiometry of 1.5 and air to the cathode at a stoichiometry of 2.0. For the mass activity measurement, the anode was supplied with H₂ and the cathode with O₂, under a constant back pressure of 50 kPa

2.4 MEA characterization

MEA characterization was performed by disassembling the stack after evaluating the durability of the stack and analyzing the MEA of the cell located in the center.

2.4.1 Cyclic voltammetry (CV)

CV was performed using a potentiostat (SI 1287, Solartron, Germany). Hydrogen and argon were supplied to anode and cathode at a flow rate of 40 ml/min, and 200 ml/min, respectively. CV was conducted for 15 cycles at a scan rate of 30 mV/s in the potential range of 0.05-1.2 V. The electrochemical surface area (ECSA) was calculated using the current density from the 0-0.4 V hydrogen desorption region, with the area above the trend line in the 0.4-0.45 V electric double layer region.

2.4.2 X-ray diffraction (XRD)

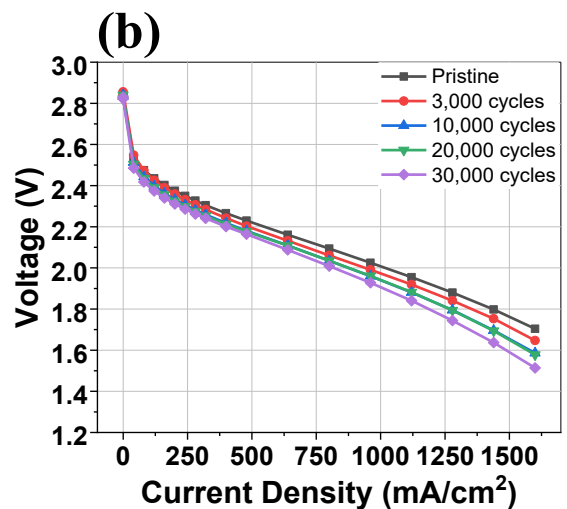
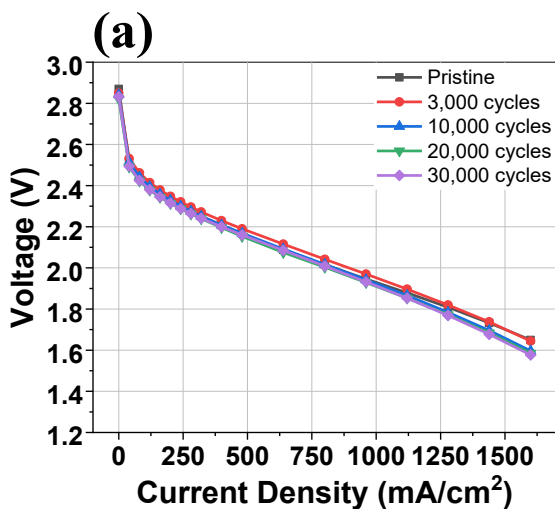
XRD patterns were observed with X-ray diffractometer (D8 discover, Bruker) employing CuK α radiation ($\lambda = 1.542 \text{ \AA}$) as the X-ray source. The diffraction pattern was collected between $2\theta = 10\text{--}90^\circ$, with a scanning speed of $3.0^\circ/\text{min}$.

2.4.3 Transmission electron microscope (TEM)

The cathode catalyst layer (CCL) of MEAs were scraped with forceps after soaked in ethanol, and subsequently sonicated in an alcohol solution. The CCL was observed using TEM (JEOL, JEM-F200). The TEM was operated at an accelerating voltage of 200kV. Observations were also conducted at room temperature.

3. Results and Discussion

Durability tests were conducted by applying each protocol to the stack. Each protocol was conducted for 30,000 cycles, and the durability test was terminated when the end of life (EOL) criterion was reached. Based on the DOE catalyst degradation cycle evaluation criteria, I–V curves were obtained at regular intervals during the durability test, and the test was terminated when the voltage at 0.8 A/cm^2 decreased by more than 90 mV (equivalent to an average voltage loss of 30 mV per cell).



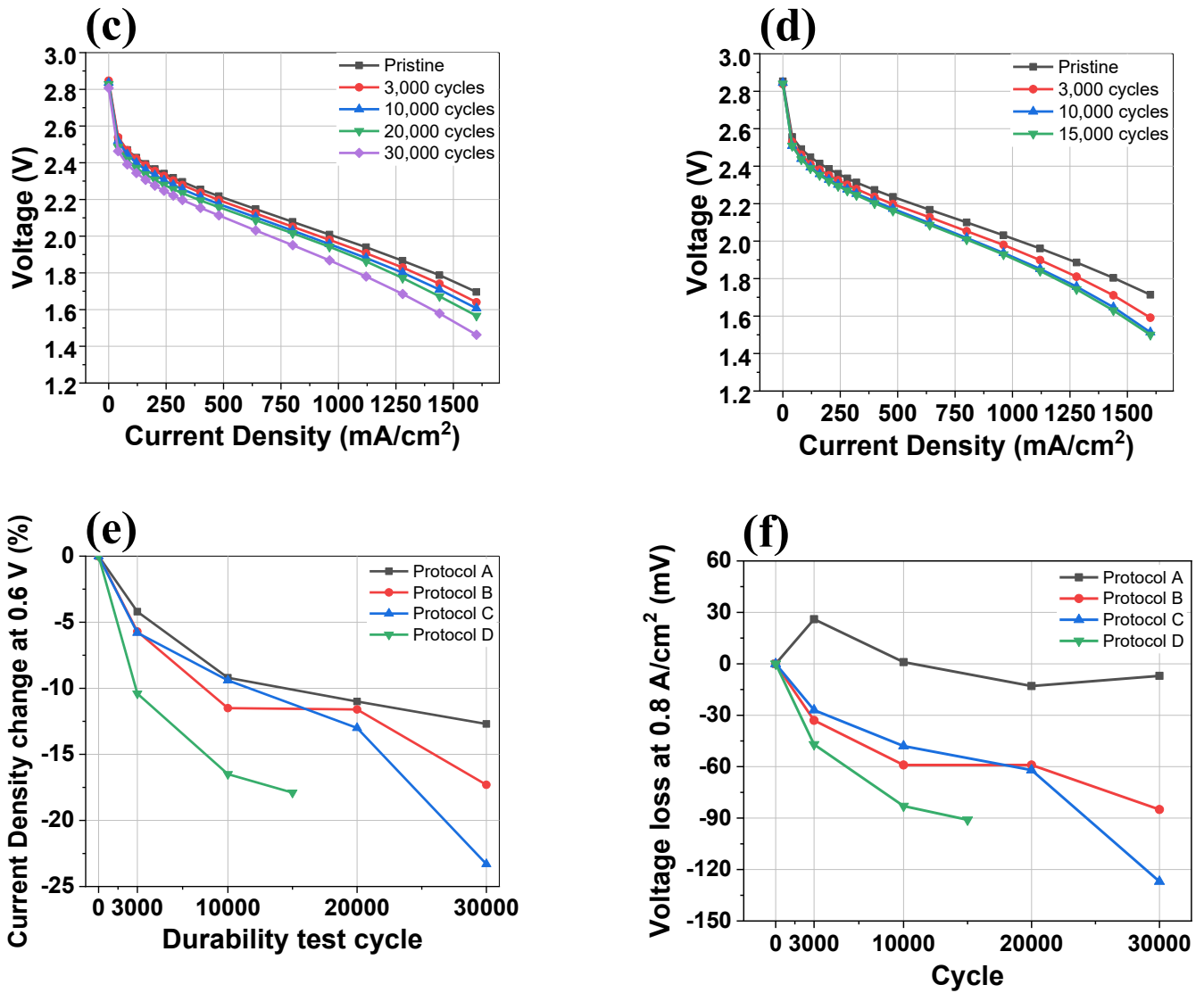


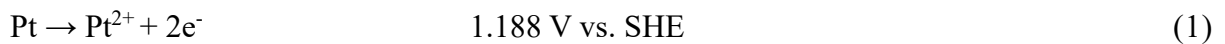
Figure 2. Changes of I-V during AST of catalyst by (a) Protocol A (b) Protocol B (c) Protocol C (d) Protocol D (e) compare of Current Density (f) voltage loss at 0.8 A cm⁻²

3.1.1 Comparison of stack performance changes during the AST process

Figure 2 shows the I-V curve changes of the stack during catalyst durability evaluation. Protocols A, B, and C were conducted for up to 30,000 cycles, among which Protocol C reached the end-of-test criterion. Protocol D reached the end-of-test criterion after 15,000 cycles, and the durability test was therefore terminated. The performance decrease at 1.8 V was 12.7% for Protocol A, 17.3% for Protocol B, and 23.5% for Protocol C. Under the accelerated Protocol D, performance decreased by 17.9% after only 15,000 cycles.

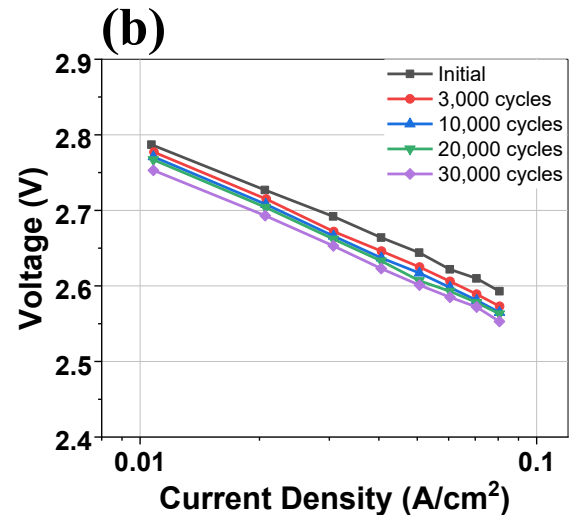
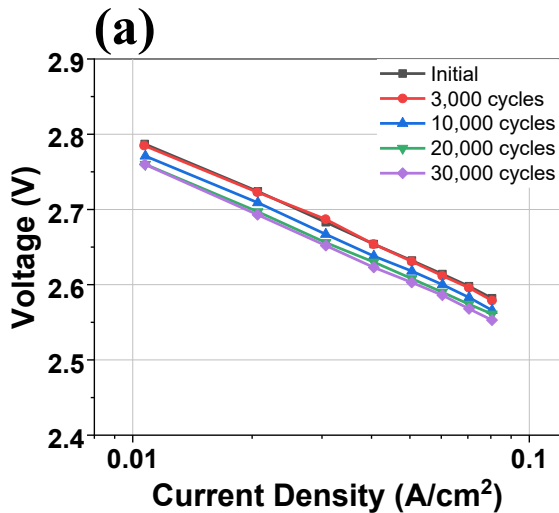
Protocol B exhibited a 36% higher degradation rate than Protocol A when the operating temperature was increased by 10 °C. This acceleration can be attributed, at least in part, to the thermally activated nature of Pt degradation processes. According to the Arrhenius relationship, an increase in temperature exponentially increases the reaction rate constant. Potential-induced degradation of Pt catalysts involves particle growth through processes such as Pt dissolution, migration, and redeposition. An increase in temperature can accelerate the rates of the associated oxidation and reduction reactions, thereby potentially promoting these processes and accelerating catalyst degradation.

Protocol C is a modified version of Protocol A using oxygen and an extended voltage range. The degradation rate increased by 85% when oxygen was used as the oxidant. Oxygen has approximately five times higher oxygen concentration than air. As mentioned previously, Pt catalyst degradation is associated with particle growth resulting from Pt dissolution and redeposition. Pt dissolution generally occurs at a high potential of approximately 1.188 V (Eq. 1). However, oxidized Pt can dissolve at a lower potential through a reaction with protons in the electrolyte (Eq. 3). Therefore, a higher degree of Pt oxidation can promote Pt dissolution. Pt oxidation can occur at potentials as low as 0.98 V, and the upper potential of 0.95 V applied in Protocol C is sufficiently high to promote Pt oxidation. The higher potential and increased oxygen concentration promote Pt oxidation, which in turn enhances Pt dissolution and accelerates the catalyst degradation rate. For this reason, operation with protocol C appears to have accelerated stack degradation by approximately 85%.



Protocol D is a modified version of Protocol A that combines a 10 °C higher operating temperature, the use of oxygen as the oxidant, and an extended voltage range. In Protocol D, the lower potential limit was further decreased to increase the amount of protons transported through the electrolyte membrane, thereby accelerating Pt dissolution. Under these conditions, the performance decreased by 40.9% more after only 15,000 cycles, indicating that the degradation was accelerated by more than a factor of two based on the number of cycles. This is likely attributable to the synergistic effects of several factors, including the increased rate of catalyst degradation reactions due to the elevated temperature, the enhanced rate of Pt oxidation resulting from the higher oxygen concentration and upper potential, and the increased Pt dissolution rate resulting from the lower lower-potential limit. These combined effects appear to have accelerated the degradation rate more significantly than those observed in Protocols B and C. Consequently, the catalyst degradation rate was accelerated by more than twofold through the combined effects of increased temperature and oxygen concentration and an extended voltage range.

3.1.2 Mass activity change during catalyst durability test



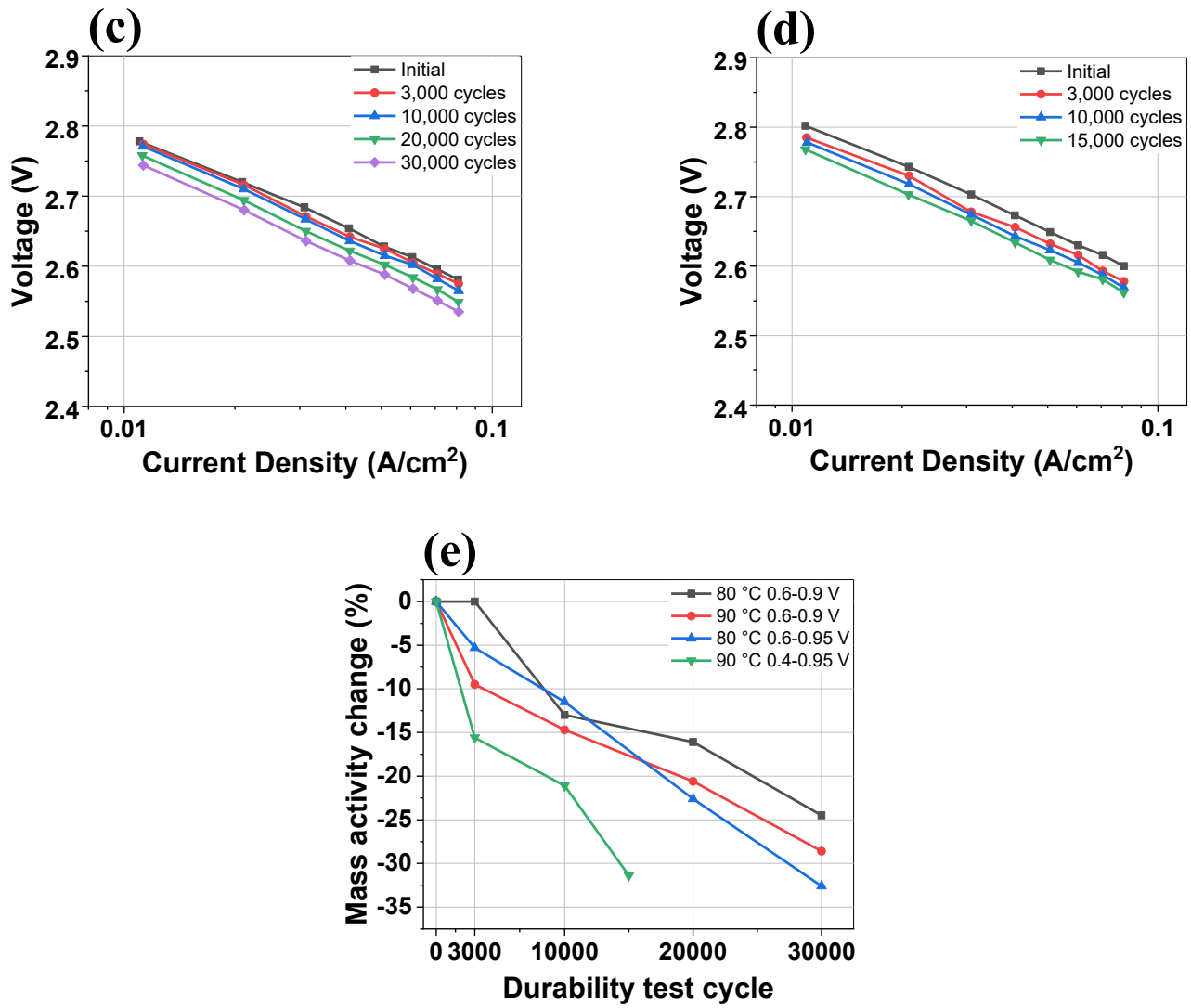


Figure 3. Tafel I–V curves during the AST for (a) Protocol A, (b) Protocol B, (c) Protocol C, and (d) Protocol D; (e) comparison of mass activity.

Degradation of the catalyst layer can be accompanied by increased reactant transport resistance. Because the observed loss in I–V performance reflects not only increased activation losses associated with catalyst degradation but also ohmic and mass-transport losses, the extent of performance degradation may be more pronounced. To isolate the effect of catalyst degradation on electrode performance, Tafel I–V curves were obtained in the low-current-density region, where mass-transport losses are minimized.

Figure 3 shows the Tafel I–V curves of the stack in the low current density region. The Tafel I–V curves were obtained by supplying hydrogen to the anode and oxygen to the cathode at a

pressure of 50 kPa to minimize the overpotential associated with mass transport. The Tafel I–V curves were then obtained after correcting for high frequency resistance (HFR) and hydrogen crossover current density (HCCD). The HFR was obtained from EIS measurements at each current density, while the HCCD was determined from LSV measurements. The mass activity was calculated from the current density at 2.7 V (0.9 V per cell) obtained from the Tafel I–V curves, normalized by the cathode Pt loading, as shown in Figure 3e.

The mass activity decreased to 25%, 27%, and 33% after 30,000 cycles, in Protocol A, B, and C, respectively. In protocol D, the mass activity decreased to 32% after 15,000 cycles. Relative to Protocol A, Protocol B, which elevated operating temperature, demonstrated a marginally accelerated degradation. Protocol C, characterized by the utilization of oxygen atmosphere and extended voltage cycling ranges, exhibited more substantial mass activity loss, with a degradation rate approximately 30% higher than protocol A. Significantly, Protocol D, which incorporated the combined effects of oxygen atmosphere, elevated temperature, and wide voltage ranges, induced a 32% reduction after merely 15,000 cycles. This corresponds to a degradation rate nearly twice that of other protocols, highlighting the synergistic detrimental effects of these combined stress factors on catalyst durability.

3.2 Postmortem of stack

After completion of the durability test, the stacks were disassembled, and the MEAs were assembled into single cells for electrochemical evaluation and post-mortem analysis.

3.2.1 ECSA change after AST

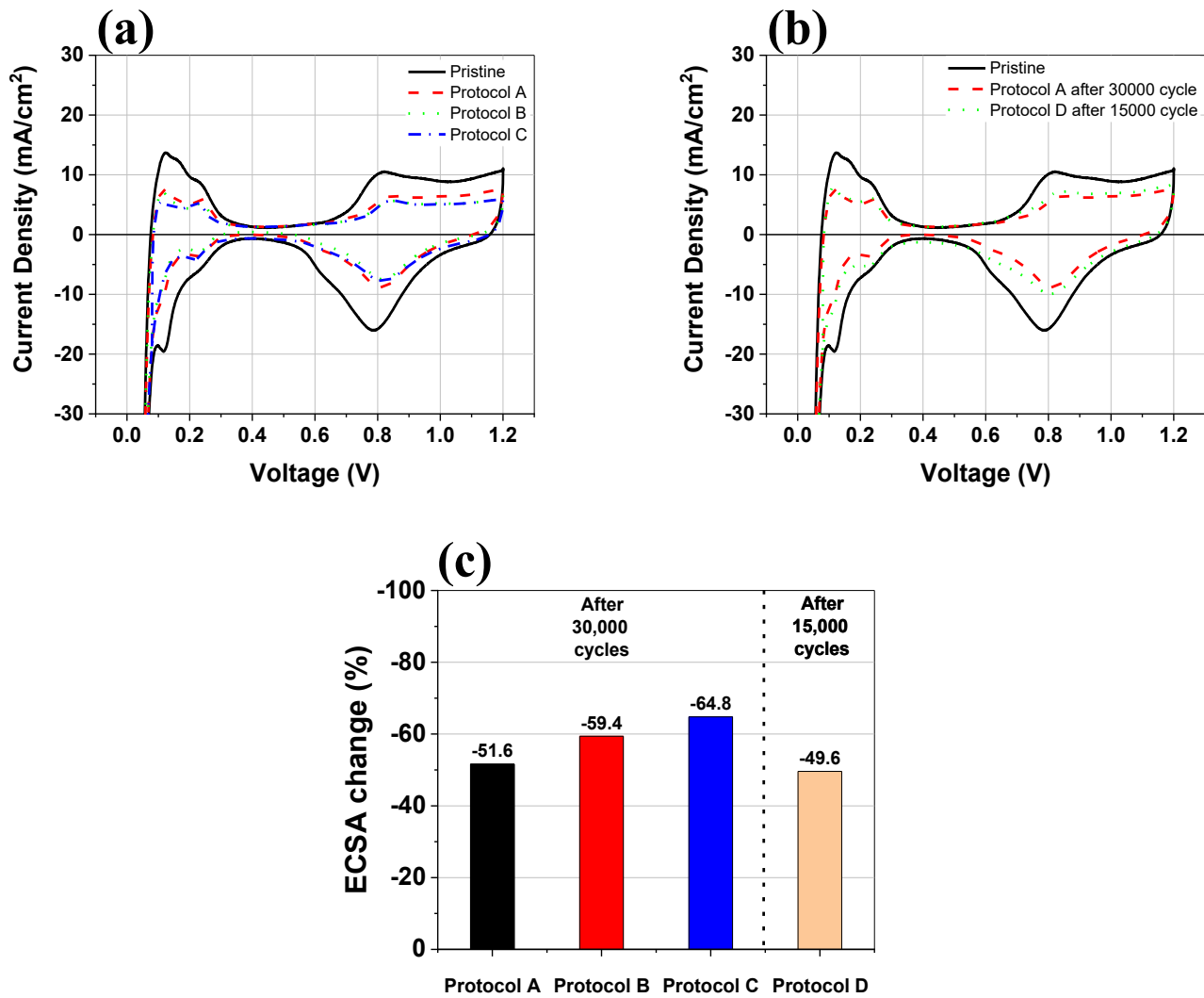


Figure 4. CV profiles after the durability test: (a) Protocols A, B, and C after 30,000 cycles; (b) Protocol D after 15,000 cycles compared with Protocol A; and (c) ECSA changes.

Figure 4 shows the CV results of the cell located in the center of the stack. After the durability test, all MEAs exhibited a decrease in ECSA due to catalyst degradation. After 30,000 cycles, the ECSA decreased by 51.6%, 59.4%, and 64.8% for Protocols A, B, and C, respectively. For Protocol D, the ECSA decreased by 49.6% after 15,000 cycles. When the degree of acceleration was compared based on the ECSA degradation rate, the 10 °C increase in operating temperature in Protocol B resulted in a 15% acceleration in the degradation rate. In Protocol C, the use of oxygen and an increased upper potential of 0.95 V, which promoted

catalyst oxidation, resulted in an approximately 25% acceleration in the degradation rate. The decrease in ECSA for Protocol D was comparable to that observed for Protocol A; however, it occurred over only 15,000 cycles, half the number of cycles used in Protocol A, indicating an approximately twofold increase in the ECSA degradation rate.

3.2.2 Changes in Catalyst Particle Size

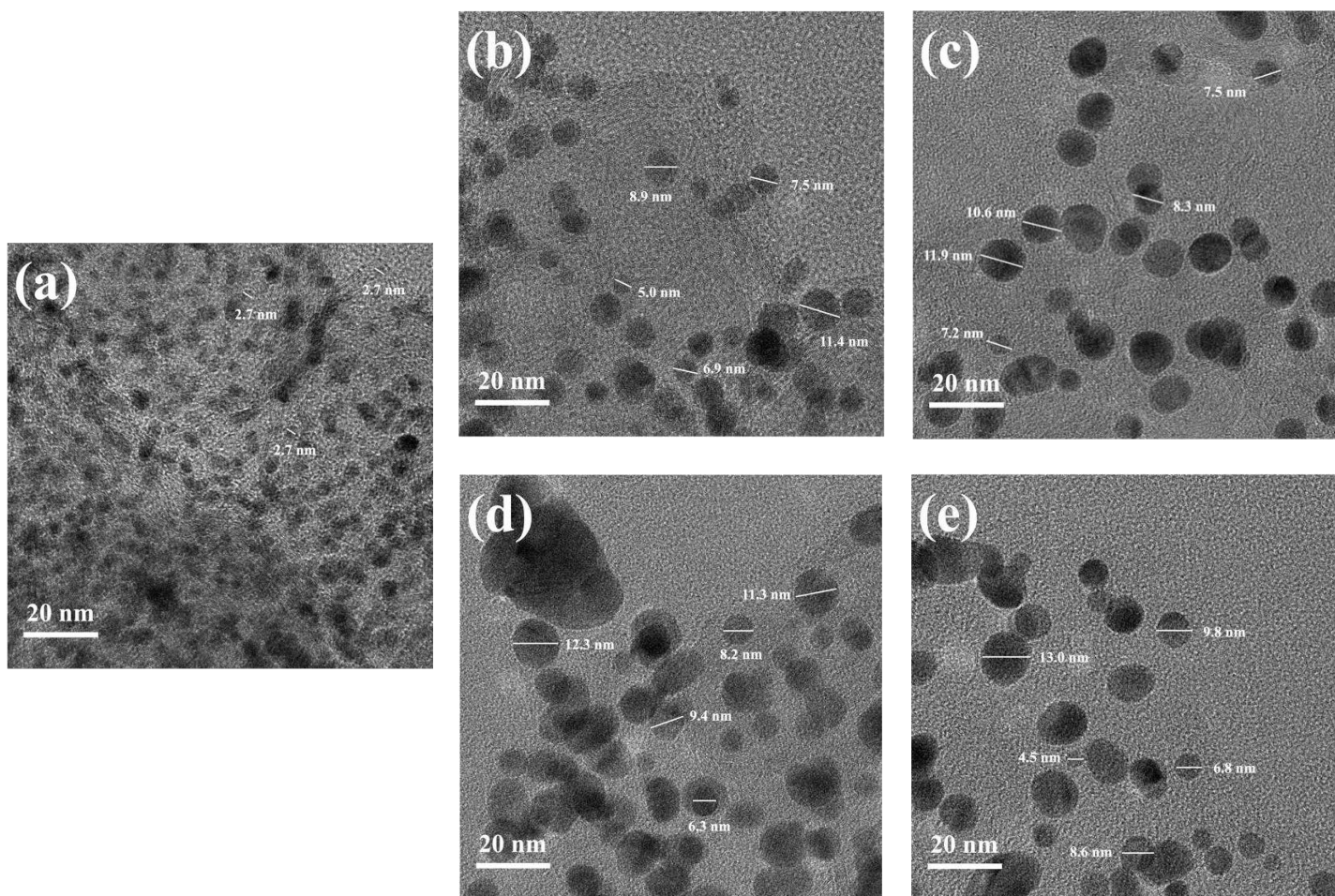


Figure 5. TEM particle size change after catalyst durability test. (a) Pristine (b) Protocol A (c) Protocol B (d) Protocol C (e) Protocol D.

Figure 5 shows TEM images of Pt particles in the cathode catalyst layers after each durability test protocol. The Pt particles in the pristine cathode catalyst layer had an average size of approximately 2.7 nm. After the catalyst ASTs, Pt particle growth was observed under all

protocols, resulting in larger particle sizes. In addition, the pristine cathode catalyst layer exhibited a dense distribution of small Pt particles. In contrast, after the ASTs, the Pt particles were more sparsely distributed, with noticeable distances between adjacent particles. There are two types of Pt particle growth mechanisms: 1) the coalescence growth mechanism in which agglomeration of Pt particles occurs due to random cluster-cluster collision on a corroded carbon support [24]; and 2) the Ostwald ripening mechanism, in which small Pt particles dissolve and precipitate on large particles to minimize the Gibbs free energy of the cluster [25, 26]. Therefore, these observations indicate that Pt particle growth and redistribution occurred during the durability tests through the dissolution and redeposition of Pt between adjacent particles during load cycling.

After degradation, Pt particles with a wide range of sizes, from approximately 3 to 13 nm, were observed in the catalyst layers. Protocol A exhibited a relatively smaller average particle size, while the particle size tended to increase from Protocol B to Protocol C. However, TEM images represent the morphology of localized regions within the catalyst layer. Therefore, to obtain a more objective comparison of Pt particle sizes, XRD analysis was performed to evaluate the Pt crystallite sizes.

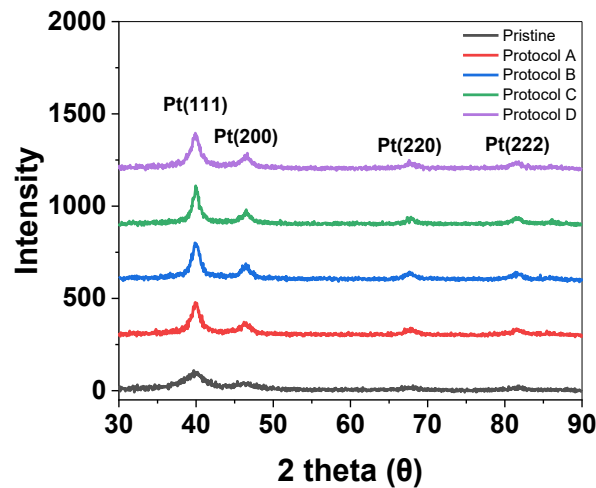


Figure 6. Changes in the XRD patterns of the cathode catalyst layer after degradation

Table 2. Pt particle size

Durability test protocol	Particle size
Protocol	nm
Pristine	2.7
Protocol A	5.1
Protocol B	5.8
Protocol C	6.8
Protocol D	5.6

Figure 6 shows the XRD analysis results of the cathode catalyst layers, and the Pt particle sizes calculated from the Pt (111) peak are summarized in Table 2. The initial Pt particle size was 2.7 nm. After 30,000 cycles, Pt particle growth was observed, with particle sizes increasing to 5.1, 5.8, and 6.8 nm for Protocols A, B, and C, respectively. For Protocol D, the Pt particle size increased to 5.6 nm after 15,000 cycles. The particle sizes observed by TEM are relatively larger than those estimated from the XRD results. This difference can be attributed to the different sampling characteristics of the two techniques. TEM provides localized information and may capture regions where significant particle growth has occurred, whereas XRD provides an average crystallite size representative of the catalyst layer as a whole.

The average Pt particle size after degradation followed the order of Protocol C > Protocol B > Protocol D > Protocol A. Notably, the greatest Pt particle growth was observed for Protocol C, which employed an oxygen-rich environment. In Protocol D, where both the operating temperature and upper and lower potential were increased, the Pt particles reached a size comparable to that observed for Protocol B after only 15,000 cycles.

These results suggest that the extent to which the catalyst is exposed to an environment favorable for Pt oxidation is a critical factor governing Pt particle growth. Pt particle growth is primarily associated with Pt dissolution and subsequent redeposition. Therefore, rather than simply increasing the rate of Pt dissolution by increasing temperature, it appears to be more important to facilitate Pt oxidation and establish an electrochemical potential range that promotes both Pt oxidation and dissolution. In particular, increasing the oxidant concentration and adjusting the upper and lower potential limits to conditions that favor Pt oxidation and dissolution appear to be effective approaches for accelerating Pt particle growth.

4. Conclusion

In this study, a stack-level catalyst AST was developed using an electric loader to evaluate catalyst degradation under dynamic load cycling conditions. A three-cell PEMFC stack was subjected to four durability protocols with different operating temperatures, oxidants, and voltage ranges, and the effects of these conditions on stack performance and cathode catalyst degradation were investigated. The stack performance degradation was accelerated by increasing the severity of the operating conditions. Increasing the operating temperature by 10 °C in Protocol B resulted in a 36% higher performance degradation rate than Protocol A. Protocol C, which employed oxygen and an extended upper potential, further accelerated the degradation rate by approximately 85%, demonstrating the significant influence of the oxidant and potential range on catalyst degradation. Protocol D, which combined elevated temperature, oxygen, and an extended voltage range, reached the end-of-life criterion after only 15,000 cycles and exhibited a degradation rate more than twice that of the less severe protocols based on cycle number. The ECSA decreased by 51.6%, 59.4%, and 64.8% after 30,000 cycles for Protocols A, B, and C, respectively, while Protocol D exhibited a 49.6% decrease after only 15,000 cycles. The mass activity also decreased under all protocols, reaching 25%, 27%, and

33% of the initial values for Protocols A, B, and C, respectively, and 32% after 15,000 cycles for Protocol D. TEM and XRD analyses further confirmed Pt particle growth and redistribution during load cycling. XRD analysis showed that the average Pt particle size increased from 2.7 nm in the pristine catalyst to 5.1, 5.8, 6.8, and 5.6 nm after Protocols A, B, C, and D, respectively. These results indicate that creating an environment favorable for catalyst oxidation is a key factor in accelerating catalyst degradation. In particular, increasing the oxidant concentration and applying an appropriate upper and lower potential range can promote Pt oxidation and dissolution, thereby accelerating catalyst degradation more effectively than increasing temperature alone. Overall, the proposed load-based AST enables catalyst durability to be evaluated directly at the PEMFC stack level without relying on potentiostat-controlled potential cycling. This approach provides a practical methodology for accelerating catalyst durability evaluation and may contribute to the development of reliable durability assessment protocols for PEMFC stacks.

Acknowledgment

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