



In situ proton exchange membrane fuel cell durability of poly(vinylidene fluoride)/polyelectrolyte blend Arkema M43 membrane

Tao Zhang^{a,*}, Wensheng He^a, James Goldbach^b, David Mountz^a, Jung Yi^c

^a Arkema Inc., 900 First Avenue, King of Prussia, PA 19406, United States

^b W.L. Gore & Associates Inc., 501 Vieve's Way, Elkton, MD 21921, United States

^c Samsung Electronics Co., Ltd., San 14, Nongseo-dong, Giheung-gu, Yongin-si, Gyeonggi-do 446-712, Republic of Korea

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ABSTRACT

A typical perfluorosulfonic acid (PFSA) polymer electrolyte membrane is composed of a single type of polymer in order to meet the strict requirements for a fuel cell membrane. The Arkema Inc. membrane technology provides a simple and lower cost route to the design of durable membrane materials. The membrane employs two intimately mixed polymers: Kynar[®] PVDF, which provides excellent mechanical characteristics, barrier properties and chemical stability, and a hydrocarbon polyelectrolyte for high proton conductivity and water transport. This work reports in-cell accelerated durability results of Arkema M43 membranes. Arkema M43 membranes demonstrated operation times that are 8–10 times longer than two other types of PFSA membranes under open-circuit voltage (OCV)-hold and voltage-cycle tests; these materials also exhibited significantly better durability than Nafion[®] NRE211 under relative humidity (RH)-cycle tests. Unlike PFSA, the membrane-electrode assemblies (MEAs) constructed using Arkema M43 membranes did not fail with catastrophic gas crossover in OCV-hold tests.

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1. Introduction

Polymer electrolyte membrane (also termed proton exchange membrane or PEM) fuel cells have received much attention in the past several decades as power sources for automobiles, portable devices, and power plants because of their high energy-conversion efficiency and low pollutant emissions [1–4]. As a proton conductor and fuel/oxidant separator, the polymer membrane is a key element in a PEM fuel cell. Membranes with high proton conductivity and mechanical and chemical stabilities are required for the high power densities and long lifetimes of the PEM fuel cells.

Current commercial PEMFC technologies typically utilize perfluorinated sulfonic acid (PFSA) materials as the polymer membrane [5]. These PFSA membranes face cost, performance, and durability limitations [6,7]. A typical PFSA polymer electrolyte membrane is composed of a single type of polymer that must meet all of the strict requirements for a fuel cell membrane. These criteria include proton conduction, electron and gas barrier characteristics, mechanical integrity, and chemical and electrochemical stability. Consequently, discovering and improving such a polymer that possesses all of the desired properties is complex and costly. Researchers at Arkema Inc. have taken an alternative approach to PEM design by addressing the challenges separately.

The Arkema M43 membrane employs two highly dissimilar polymers; poly(vinylidene fluoride) (PVDF), which provides excellent mechanical qualities (Table 1), barrier properties and chemical stability, is intimately blended with a polyelectrolyte for high proton conductivity and water transport [8]. The process is simple, provides a less expensive route to membrane material design, and allows for tailoring of membrane properties via adjustments in component ratios rather than synthesis of numerous new polymers. In addition, Arkema's blending technology has demonstrated that PVDF can be blended with a variety of highly protogenic polyelectrolytes [8,9]. Arkema's fuel cell membrane technology offers the unique combination of potential for low cost and high performance that is required to meet future market needs for membranes and membrane-electrode assemblies (MEAs).

In addition to cost, membrane durability is one of the primary limitations of PEM systems and continues to be an active area of research [7,10–13]. Although PFSA membranes are composed of relatively stable C–F bonds, their performance in PEM fuel cells decays over time due to chemical degradation and mechanical fatigue. The combination of chemical and mechanical degradation results in membrane thinning and pinhole formations, which leads to excessive reactant crossover, hot spots and, consequently, cell failure [7,14–17]. Various approaches have been taken to alleviate PFSA membrane durability issues, mostly by introducing mechanical reinforcement with porous supports or blends [18–22], adding radical scavenging agents [11,23–27], and reducing reactive sites [28]. Of note, chemical and mechanical degradation occurs

* Corresponding author. Tel.: +1 6108786296; fax: +1 6108786298.

E-mail address: zhangtao9703@yahoo.com (T. Zhang).

Table 1
Physical properties of the Arkema M43 and Ion-Power PFSA N111-IP membranes.

| Membrane | Equivalent weight (g mol ⁻¹) | Density (g cm ⁻³) | Water uptake (wt%) | X or Y swelling (%) | Thickness swelling (%) | Break tensile (MPa) | Elongation (%) | Tear strength (lb in ⁻¹) | Tear propagation (lb) |
|---------------------|--|-------------------------------|--------------------|---------------------|------------------------|---------------------|----------------|--------------------------------------|-----------------------|
| PFSA N111-IP | 1100 | 1.8 | 37 | 15 | 14 | 19 | 103 | 404 | 0.004 |
| Arkema M43 membrane | 800 | 1.5 | 60 | 20 | 10–15 | 27 | 95 | 934 | 0.018 |

simultaneously, and their effects on membrane properties and durability are coupled [29]. Membranes subjected to mechanical durability testing under nitrogen last several times longer than those tested under reactive gases where chemical degradation also occurs and expedites mechanical failure. In addition, a recent report [11] demonstrated that a PTFE-reinforced PFSA membrane showed superior chemical durability under OCV-hold testing conditions relative to non-reinforced PFSA membranes, suggesting that the improved membrane mechanical properties can extend its chemical durability. In addition to PFSA, various other membrane materials showed correlations between mechanical property degradation and load-cycle durability [30]. Thus, the above results stress that excellent bulk mechanical properties, chemically stable molecular structures, and the ability to suppress the formation of radical molecular species should be combined together in the design of novel durable polymer membranes.

Investigation of the in-cell durability of alternative low-cost membranes may provide guidance for future research and the development of new, high-performance materials beyond PFSA. This work reports the in-cell performance and accelerated durability results of Arkema's M43 membrane that incorporates a hydrocarbon polyelectrolyte in a PVDF blend. The membrane chemical durability was evaluated by OCV-hold tests with H₂/O₂ with non-saturated humidity at elevated temperatures. The mechanical durability was evaluated using RH-cycle tests under which the membrane was exposed to rapid humidity changes. Voltage-cycle durability tests were also conducted under low RH and elevated temperature conditions that impose both chemical and mechanical degradation factors on the membrane. The results were compared to non-reinforced PFSA membrane materials that were commercially available.

2. Experimental

2.1. Physical characterizations of membranes

The water uptake and swelling of membranes were measured with 4 cm × 4 cm membrane samples that were cut from a square die and dried overnight at 35 °C with 28 in Hg vacuum. They were promptly weighed using an analytical balance (resolution of 0.0001 g) after drying. The X/Y sample dimensions were measured with a ruler, and the thickness was determined using a screw-type micrometer (an average of five readings). The samples were then boiled in 18 MΩ deionized water for 1 h. After cooling, the samples were blotted dry, and their mass and dimensions were collected again. The water uptake and dimensional swelling was calculated from the difference in the measurements between the dried and hydrated samples; three samples were used for each material. The densities and equivalent weights were measured by weighing and titration, respectively, after the membrane was dried in the same procedure as above. Mechanical tests were conducted by the following procedures. All specimens were cut from a die and conditioned at room temperature and 50 ± 3% relative humidity for at least 24 h; five specimens were tested for each material. Tensile tests were carried out according to ASTM D882. Specimens have dimensions of 127 mm × 13 mm. The experiments were performed on a Zwick-Roell Z005 mechanical test instrument equipped with a

100 N load cell using a crosshead speed of 500 mm min⁻¹ and an initial grip separation of 35 mm. Tear initiation was tested according to ASTM D1004. The experiments were performed on a Zwick-Roell Z005 mechanical test instrument equipped with a 100 N load cell using a crosshead speed of 50 mm min⁻¹. Tear propagation was performed according to ASTM D1938. The experiments were performed on a Zwick-Roell Z005 mechanical test instrument equipped with a 100 N load cell using a crosshead speed of 250 mm min⁻¹.

2.2. Cell preparation and testing

Arkema's M43 membrane and two types of commercially available PFSA membranes (Ion-Power N111-IP and Dupont Nafion® NRE211) were studied in this work. All three membranes have original thickness of 25 μm. The proprietary hydrogen PEM fuel cell gas-diffusion electrodes employed in this work feature a Toray TGP-H-060 carbon paper backing and a catalyst layer that contains a PFSA ionomer and 0.4 mg cm⁻²/side Pt loading. Membrane electrode assemblies were bound by hot-pressing one piece of gas-diffusion electrode (GDE) on each side of the membrane. The MEA was subsequently assembled in a 25 cm² single cell hardware (Fuel Cell Technologies, Inc., 3-serpentine flow field). Fuel cell tests were performed on Teledyne Medusa® RD test stands equipped with Scribner 890CL load boxes. A BekkTech BT-522 test stand was used to carry out relative humidity-cycle durability test. Nitrogen purging procedures were always followed prior to start up and shut down of a cell. All cells were conditioned under fully humidified H₂/air at 80 °C by cycling between 0.75 V and 0.6 V until a steady performance was established.

2.3. Hydrogen crossover measurement

Hydrogen crossover intensity was measured electrochemically periodically throughout the OCV hold and voltage-cycle durability tests to monitor the membrane status. Hydrogen and nitrogen gases were flowed to the anode and cathode side of the cell, respectively. After the cell voltage dropped to 0.1 V, a steady-state voltage step scan (30 s per 10 mV step) was performed on the cathode from 0.1 to 0.5 V with the anode as the reference electrode using a Solartron® 1287 potentiostat. The limiting anodic current, where the linear fit intercepts at zero on the current axis, corresponds to the hydrogen crossover rate to the cathode where the hydrogen is electrochemically oxidized, while the slope is the reciprocal of the electrical shorting resistance.

2.4. Effluent water tests

Effluent water samples from the cell were periodically collected and analyzed by ion chromatography for fluorine and sulfur emission rates. The Metrohm® IC instrument uses a conductivity detector and Metrosep A Supp5 column for anion analysis. The limit of detection is approximately 0.05 ppm (by weight). A three to five point calibration curve, covering the range of the samples run, is prepared each time samples are analyzed.

Table 2
Accelerated durability protocols for Arkema M43 and PFSA membranes.

| Durability test | Operation | Temp (°C) | RH (%) | Pressure (psig) | Gas flow (slpm) | Failure criteria |
|-----------------|-------------------------------|-----------|-----------|-----------------|--|--|
| OCV-hold | Hold at OCV | 90 | 30 | 0 | H ₂ /O ₂ : 1/1 | H ₂ crossover > 10 mA cm ⁻² or OCV < 0.6 V |
| Voltage-cycle | OCV (1 min) and 0.4 V (1 min) | 90 | 50 | 0 | H ₂ /O ₂ : 0.5/1 | H ₂ crossover > 10 mA cm ⁻² or OCV < 0.6 V |
| RH-cycle | 150% (2 min) and 0% (2 min) | 80 | 150 and 0 | 0 | Air/air: 1/1 | Air hydraulic crossover > 5 sccm at 20 kPa ΔP |

2.5. Accelerated durability test

Three test protocols were followed (Table 2) to evaluate the in-cell durability of the membranes under accelerated conditions. An elevated cell temperature, reduced humidity, pure oxygen gas, rapid humidity changes, and rapid load changes were used to accelerate membrane degradation. Periodic diagnoses, including hydrogen gas or air hydraulic crossover, electrical shorting, electrochemical area, were carried out to probe the cell status. A pristine MEA was always used for each durability test.

3. Results and discussion

3.1. Polarization

The hydrogen/air polarization curves and current interrupt Ohmic resistances of the Arkema M43 and Nafion® NRE211 membrane under different humidity conditions are illustrated in Fig. 1. Arkema's M43 membrane has lower proton conductivity and produces slightly lower performance under 100/70% relative humidity than the Nafion® NRE211 at reduced relative humidity. The difference in performance is more drastic at 50/50% RH.

3.2. OCV-hold (chemical durability)

As shown in Fig. 2, MEAs produced with the Arkema M43 membrane maintained high OCV values for significantly longer times in the OCV-hold durability test than those composed of PFSA membranes. The curves of OCV value versus time for all three samples experienced a three-stage behavior: an initial rapid drop, a middle slower drop, and a final catastrophic drop. OCV spikes were observed after hydrogen crossover diagnosis when nitrogen gas flowed through the cathode. As shown in Fig. 2b, the Arkema M43 membrane exhibits a lower initial hydrogen crossover rate than the PFSA. This important parameter reduces formation of chemically detrimental peroxide radicals and local hot spots; thinner membranes could be used while maintaining a minimum value of crossover, as defined as that of the 25 μm PFSA membranes. Interestingly, the Arkema M43 membrane did not fail with catastrophic crossover increase as PFSA do under OCV-hold test, as confirmed by subsequent nitrogen hydraulic crossover testing of the end-of-life (EOL) MEA. Similar observations have been previously reported [10,11], wherein hydrocarbon materials investigated did not show crossover failure under OCV-hold test. These results show the strength of Arkema's M43 membrane over the PFSA studied, which fail locally with high crossover rate under OCV-hold durability; these data also suggest that OCV-hold tests may not be appropriate or harsh enough for Arkema M43 membranes and certain hydrocarbon materials.

The effluent water samples were analyzed with ion chromatography to characterize fluoride and sulfate emission. The fluoride and sulfate emission rates of PFSA controls and the Arkema M43 membrane are plotted in Fig. 3. Both the fluoride and sulfate emission results from PFSA controls are similar to those previously reported [10,31]. In contrast to the PFSA materials, Arkema M43 membranes showed essentially no fluoride emission until the very end of test due to the extraordinary chemical stability of the PVDF matrix. The sulfate emission rates from Arkema M43 and PFSA

membranes tested were similar. Gel permeation chromatography was also used to analyze the effluent water samples from Arkema M43 membranes, and no oligomers were detected. Elemental analysis on the sulfur element content of the end-of-life membrane showed no major sulfur loss beyond that detected as sulfate by ion chromatography during OCV-hold durability tests of Arkema M43 membranes. Encouragingly, the Arkema M43 membrane, featuring a non-fluorinated hydrocarbon polyelectrolyte, exhibits a similar sulfate emission rate to the PFSA studied in OCV-hold durability tests. This result, together with significantly longer lifetimes and no catastrophic crossover failure for Arkema M43 membranes, supports the notion that failure of PFSA occurs more locally and earlier than Arkema M43 membrane; these observations demon-

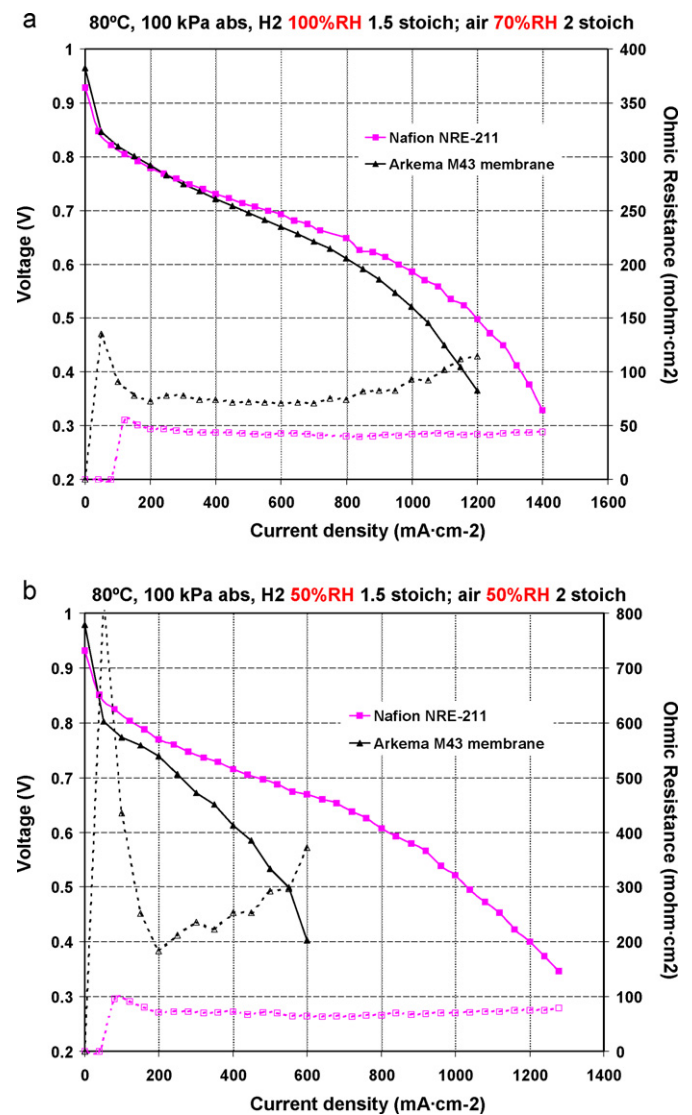


Fig. 1. (a) H₂/air polarization curves of Arkema M43 membrane and Nafion® NRE211 membrane under 80 °C and 100/70% relative humidity. (b) H₂/air polarizations curves of Arkema M43 membrane and Nafion® NRE211 membrane under 80 °C and 50/50% relative humidity.

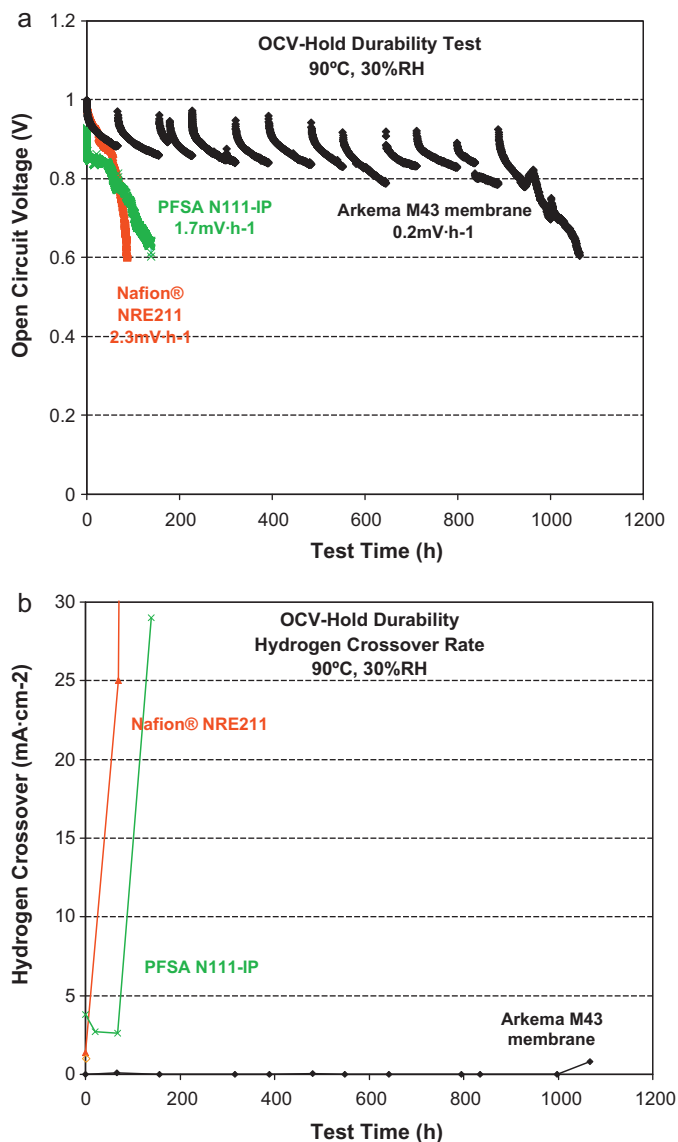


Fig. 2. (a) Open circuit potential versus test time during OCV-hold durability test on Arkema M43 membrane and PFSA membranes. (b) Hydrogen crossover versus test time during OCV-hold durability test on Arkema M43 membrane and PFSA membranes.

strate a distinct difference in durability between PFSA and some hydrocarbon membranes. As a reminder, the chemical durability of PFSA membranes has been significantly improved by introducing radical scavenger additives [11,23–27]. The effects of these stabilizing agents on Arkema M43 membranes are out of the scope of this paper, and, therefore, comparisons were made to plain PFSA.

After MEA failure was observed in the OCV-hold durability, the Arkema M43 membrane MEA was reconditioned until a steady performance was achieved before EOL performance was evaluated. As illustrated in Fig. 4, both air and oxygen Tafel plots were lower at the EOL than at the beginning-of-life (BOL). Additionally, the oxygen gain was larger at the EOL than at the BOL. Finally in Fig. 4(b), a cyclic voltammogram of the cathode of a post-mortem MEA after OCV-hold durability tests showed less electrochemically active area when compared to a fresh MEA. All of these results strongly suggest that degradation is occurring in the electrodes, which is not surprising under these harsh conditions; contrastingly, the Arkema M43 membrane MEA generated long-lasting and high OCV.

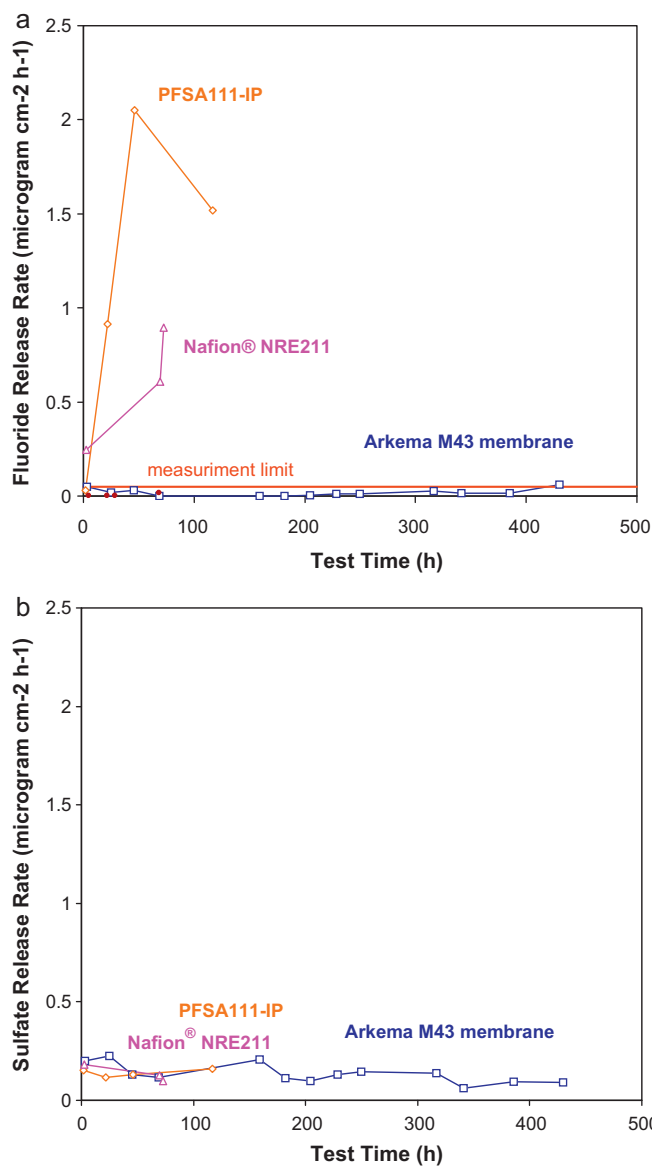


Fig. 3. (a) Fluoride emission rate during OCV-hold durability testing. (b) Sulfate emission rate during OCV-hold durability testing.

3.3. RH-cycle (mechanical durability)

The mechanical durability of Arkema M43 and PFSA membranes was tested by exposing the MEAs to rapid changes in relative humidity (150 ↔ 0% RH every 2 min) of air at 80°C. The MEA was periodically diagnosed by an air hydraulic crossover rate at 80°C under a 20 kPa pressure gradient. The hydraulic air crossover rate is plotted in Fig. 5, where the Arkema M43 and Ion-Power PFSA111IP membranes reached the target without failure until 20,000 cycles (1333 h), when the tests were voluntarily stopped. However, the Dupont Nafion® NRE211 showed inferior durability and failed around 6000 cycles in this specific test. The PFSA mechanical durability results here are similar to those in previous studies [7,29,32]. The long testing time and lack of failure with Arkema M43 membranes and Ion-Power PFSA111IP suggests that both membranes are sufficiently mechanically robust to withstand the stresses experienced in the current protocol developed for automotive applications.

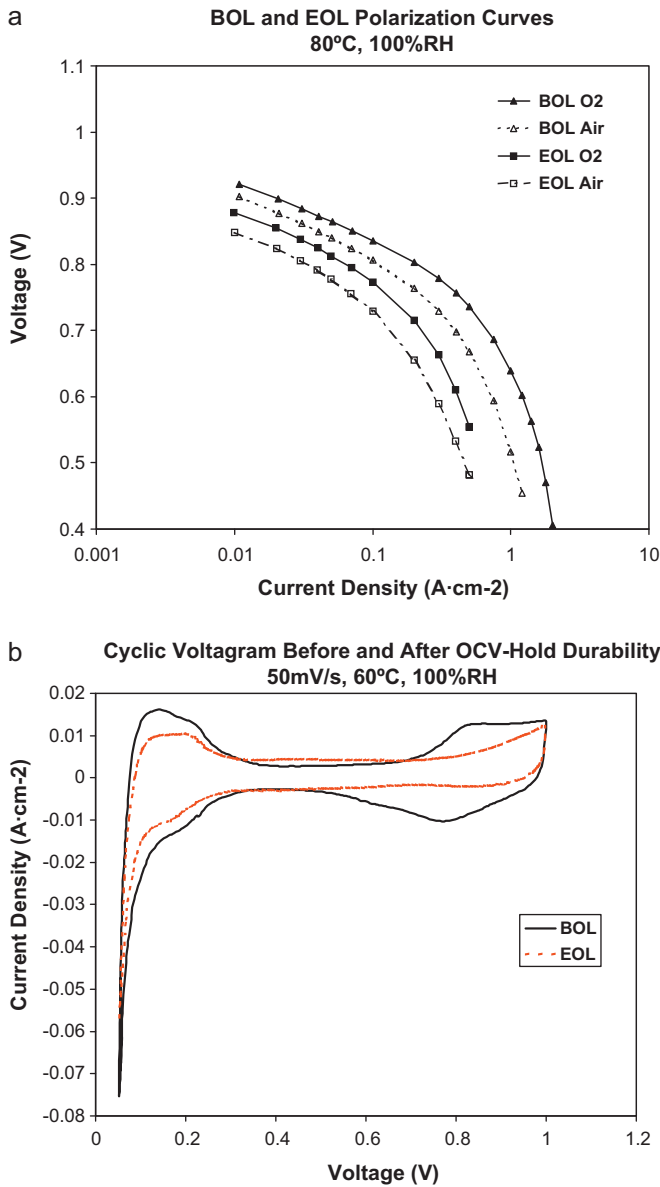


Fig. 4. (a) Tafel plots of an Arkema M43 membrane MEA before and after OCV-hold durability test. (b) Cyclic voltammogram of an EOL Arkema M43 membrane MEA after OCV-hold durability test versus BOL MEA.

3.4. Voltage-cycle (chemical and mechanical durability)

Voltage-cycle durability tests were also carried out under low RH conditions to impose an environment where both accelerated chemical and mechanical degradations occur. Such a combination of chemically and mechanically degradative circumstances can be encountered in automotive applications where frequent changes of load are expected. Here OCV, rather than a higher constant potential, was used to reduce the electrode degradation. As shown in Fig. 6, Arkema M43 membranes lasted 8–10 times longer than PFSA membranes in voltage-cycle durability testing before the hydrogen crossover rate reached the designated failure point (10 mA cm^{-2}). In contrast to OCV-hold and RH-cycle tests, Arkema M43 membranes showed gas-crossover failure at its EOL in this voltage-cycle durability test. Similar observations have been reported by Sethuraman et al. [10], who described that the investigated biphenyl sulfone hydrocarbon membrane showed hydrogen crossover failure under voltage-cycle tests, but not under OCV-

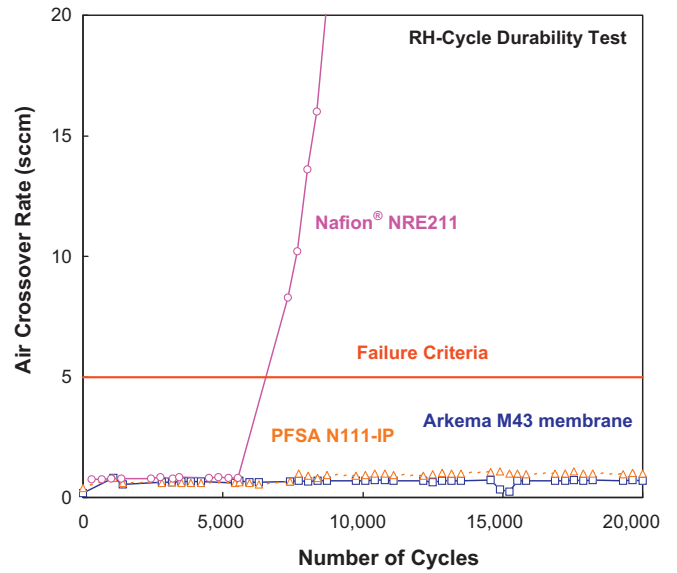


Fig. 5. Air hydraulic crossover rate during relative humidity-cycle durability test.

hold durability tests. These results suggest that voltage-cycle tests could be a more effective protocol for certain membrane materials as a test of both chemical and mechanical durability than individual chemical or mechanical testing. Fig. 7 illustrates the OCV and current density data from Arkema M43 and PFSA membranes in voltage-cycle tests. Clearly, the Arkema M43 membrane generated a significantly higher OCV value throughout the test than those composed of PFSA due to the superior gas barrier properties of Arkema M43 membranes. However, Arkema M43 membranes produced lower current density under 0.4 V, compared to PFSA membranes due to lower proton conductivity at reduced RH. A separate voltage-cycle test was performed using a MEA constructed with an Ion-Power PFSA111 membrane by cycling between OCV and 0.1 A cm^{-2} , closely mimicking the current generated by Arkema M43 membranes at 0.4 V, to investigate the effects of drawn current density on the durability. As shown, the MEA constructed with the Ion-Power PFSA111 lasted for a significantly shorter time than the MEA constructed with an Arkema M43 membrane. In summary,

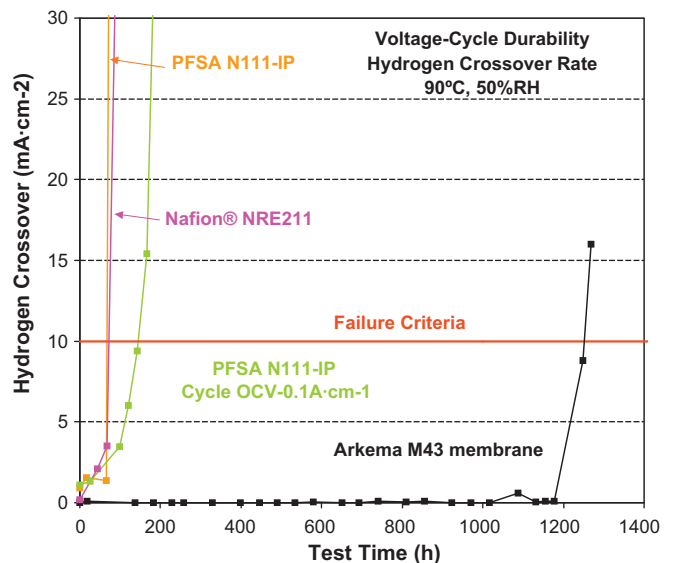


Fig. 6. Hydrogen crossover rate of Arkema M43 membrane and PFSA MEAs during voltage-cycle durability test.

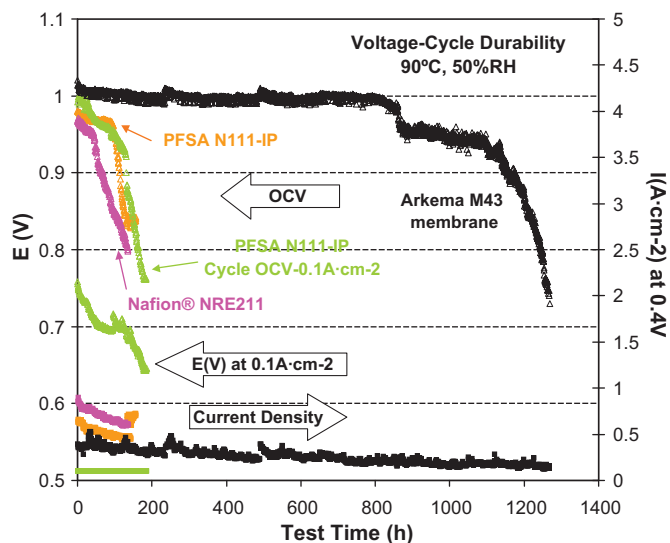


Fig. 7. Open circuit potential and current density at 0.4 V of Arkema M43 membrane and PFSA MEAs during voltage-cycle durability test.

Arkema M43 membranes demonstrated voltage-cycle operations that were 8–10 times superior to the PFSA membranes studied.

The EOL performance of Arkema M43 membranes was obtained after re-conditioning the cell. The data are plotted with the BOL performance in Fig. 8. Due to the high crossover and shorting after failure, the Arkema M43 membranes' EOL polarization produced a low OCV. Also, the EOL iR-free performance is significantly lower than that at the BOL, showing a significant loss of electrode performance. The voltage was only cycled to OCV, instead of a higher potential in the voltage-cycle test. Nevertheless, the electrode performance degradation with Arkema M43 membranes was not surprising when considering that voltage-cycles promote platinum dissolution-precipitation [33,34], significantly higher OCV, and longer operation Arkema M43 membrane produced than PFSA did.

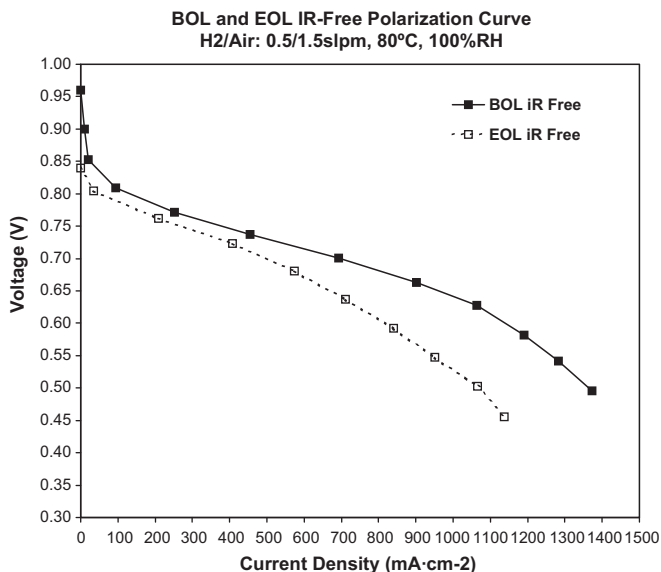


Fig. 8. IR-free polarization curve of an Arkema M43 membrane MEA before and after voltage-cycle durability test.

4. Conclusion

The Arkema membrane technology provides a simple and less expensive route to membrane materials. The membrane employs two intimately mixed polymers: Kynar® PVDF (excellent mechanical characteristics, barrier properties and chemical stability) and a hydrocarbon polyelectrolyte (high proton conductivity and water transport). To develop a durable membrane, Arkema membrane technology combines characteristics including excellent bulk mechanical properties, a chemically stable molecular structure, and the suppressed formation of chemically reactive species due to reduced gas permeability. In this work, Arkema M43 membranes demonstrated operation times that were 8–10 times longer than two types of PFSA membranes under OCV-hold and voltage-cycle tests; the Arkema M43 membranes also exhibited significantly better durability than Nafion® NRE211 under RH-cycle tests. Unlike PFSA, the MEAs constructed using Arkema M43 membranes did not fail with catastrophic gas crossover in OCV-hold test. This result, together with sulfur emission rates similar to unmodified PFSA and the significantly superior durability of Arkema M43 membranes, strongly suggests the presence of different degradation mechanisms of Arkema M43 membranes relative to the studied PFSA, which fail locally with high crossover rates under OCV-hold durability. OCV-hold tests may not be appropriate or harsh enough for Arkema M43 membranes and certain hydrocarbon materials. Under voltage-cycle testing, both Arkema M43 and PFSA membranes failed with high gas crossover, which is believed to be related to both mechanical and chemical stresses exerted on the membranes in this protocol.

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