Quarterly Progress Report

Project Title: System contaminant testing and analysis for proton exchange membrane fuel cells

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Status:

Life tests with and without siloxane contamination were repeated using Gore MEAs to alleviate the effect of previously observed edge failures with GM MEAs. Both life tests were completed (>500 h in each case) and results are summarily presented. As a reminder, specific diagnostic tests performed during the MEA Chemical Stability included:

- Cell open circuit voltage at the end of the 12 hr hold (figure 1)
- Cell voltage at 200 mA/cm² (figure 1)
- Cell high frequency resistance at 200 mA/cm² (figure 1)
- Cell electronic (shorting) resistance (figure 1)
- Cell hydrogen crossover (figure 1)
- Effluent water analyses
  - Fluoride (figure 2)
  - Sulfate (figure 3)
- Note: The above diagnostics (HFR, H2X, Shorting) were performed at the OCV hold conditions, i.e. 90C, 30% RH, 150 kPa.

Diagnostics performed at the beginning of test (BOT) and end of test (EOT) included:

- Polarization curves under wet and dry conditions (figure 4)
  - Wet: (80C, 100%RH, 182 kPa)
  - Dry: (80C, 35% RH, 150 kPa)
- Anode (figure 5) and cathode (figure 6) catalyst active areas
- Hydrogen crossover at 28C, 0 kPa (figure 7)
- Electronic cell resistance at 28C, 0 kPa (figure 8)

Some of these data are still being processed and therefore some figures are partially incomplete (figures 2 and 3 Loctite 5039 results).
The most significant result relates to figure 1 summarizing measurements performed during the life test itself. It is observed that the hydrogen crossover current significantly increases for the baseline test (by approximately an order of magnitude) whereas the test with siloxane injection led to a constant hydrogen crossover current. Therefore, under the original assumption that the introduction of siloxane compounds would accelerate the degradation, the baseline results may be jeopardizing comparisons with the siloxane based life test. For instance, the larger crossover could explain the decrease in the OCV value (mixed potential).

Other important results from figure 1 include a significant increase in cell resistance in the presence of siloxane. This is not unexpected as siloxane was previously observed to penetrate the ionomer and affect its elastic modulus. It is not believed that the hydrogen crossover would affect this conclusion because the cell resistance remains constant during the baseline test. A similar conclusion is reached in relation to the cell performance. The performance decrease is significantly larger for the test that includes siloxane injection. Other results provide additional clues to determine the contamination mechanism.

For instance, oxygen polarization curves (figure 4) indicate that the catalyst, the cell resistance and the limiting current (the cell could not be operated at the larger current densities) are affected by the presence of the contaminant (effects are larger than for the baseline test which include a larger hydrogen crossover current). The negative kinetic effect is further supported by the catalyst active areas (figures 5 and 6).

MEAs were sent to GM for additional ex situ tests.

In summary, the presence of the siloxane appears to affect all types of performance losses. However, additional diagnostics will be required to better identify the source of the increased mass transport loss (change in surface hydrophobicity affecting liquid water removal, catalyst layer pore blockage, change in carbon surface groups, etc). From that standpoint GM representatives should consider adding specific ex situ tests. Additionally, the test strategy may need to be reconsidered because the presence of a larger hydrogen crossover current for the baseline test affects data interpretation. However, from the opposite perspective, the mitigation effect of siloxane on the hydrogen crossover does require further analysis and will benefit from the IC results of the effluent water currently underway, and from the post-mortem results from GM. Although siloxane may have induced embrittlement within the MEA, the chemical stability protocol does not contain a mechanical cycle, such as rH cycling, to induce brittle failure modes within the MEA. For future testing, incorporating a mechanical membrane degradation test should be considered. Also more specific accelerated tests for contamination could also be considered.
Figure 1. Overview of chemical stability diagnostic results (left: baseline, right: Loctite 5039).

Figure 2. Effluent water fluoride analysis results (left: baseline, right: Loctite 5039).
Figure 3. Effluent water sulfate analysis results (left: baseline, right: Loctite 5039).

Figure 4. Beginning and end of test polarization curves (left: baseline, right: Loctite 5039).
Figure 5. Beginning and end of test anode CVs (left: baseline, right: Loctite 5039).

Figure 6. Beginning and end of test cathode CVs (left: baseline, right: Loctite 5039).
Figure 7. Beginning and end of test hydrogen crossover results (left: baseline, right: Loctite 5039).

Figure 8. Beginning and end of test shorting resistance results (left: baseline, right: Loctite 5039).

Plans for Next Quarter and Key Issues:

- Completion of the Gore MEA siloxane tests
- Decision to proceed with repeat tests or to modify the test protocol to avoid confounding results
- Development and construction of a liquid injection system to support long term tests with leached contaminants from system materials

Patents: No patents were generated during this reporting period.

Publications / Presentations: No publications or presentations were generated during this reporting period.