Extended Durability Testing of an External Fuel Processor for a Solid Oxide Fuel Cell (SOFC)

Final Technical Report

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1 Executive Summary

Durability testing was performed on an external fuel processor (EFP) for a solid oxide fuel cell (SOFC) power plant. The EFP enables the SOFC to reach high system efficiency (electrical efficiency up to 60%) using pipeline natural gas and eliminates the need for large quantities of bottled gases. LG Fuel Cell Systems Inc. (formerly known as Rolls-Royce Fuel Cell Systems (US) Inc.) (LGFCS) is developing natural gas-fired SOFC power plants for stationary power applications. These power plants will greatly benefit the public by reducing the cost of electricity while reducing the amount of gaseous emissions of carbon dioxide, sulfur oxides, and nitrogen oxides compared to conventional power plants.

The EFP uses pipeline natural gas and air to provide all the gas streams required by the SOFC power plant; specifically those needed for start-up, normal operation, and shutdown. It includes a natural gas desulfurizer, a synthesis-gas generator and a start-gas generator. The research in this project demonstrated that the EFP could meet its performance and durability targets. The data generated helped assess the impact of long-term operation on system performance and system hardware. The research also showed the negative impact of ambient weather (both hot and cold conditions) on system operation and performance.

1.1 Introduction

LG Fuel Cell Systems, Inc. is developing MWe-scale, solid oxide fuel cell power plants for stationary power application. An important part of the SOFC power plant is the external fuel processor. The EFP uses pipeline natural gas and air to generate all the gas streams required by the SOFC power plant and eliminates the need for on-site storage of high-pressure, bottled gases such as nitrogen and hydrogen. It generates non-flammable reducing gas (for start-up/shutdown), synthesis gas (for low-load operation) and desulfurized natural gas (for normal operation). These gas streams will enable the SOFC power plant to operate at high system efficiency (up to 60% fuel in to electricity out). The high efficiency of the SOFC power plant will give it much lower gaseous emissions of carbon dioxide, sulfur oxides, and nitrogen oxides compared to conventional power plants. This high efficiency when coupled with high availability and low maintenance costs will result in a low cost of electricity for the SOFC power plant.

1.2 Objectives

The main goal of this project was to perform extended durability testing of the three EFP Subsystems. The specific objectives were to:

- Conduct long-term tests in relevant environments for the EFP Subsystems needed to support operation of a SOFC power plant. The Subsystems included:
  1) Desulfurizer Subsystem
  2) Start-gas Subsystem
  3) Synthesis-gas Subsystem

- Determine any impact of ambient temperatures (hot and cold environments) on performance and component reliability

- Determine system responses times for startup and transient operation
1.3 Technical Barriers

This project addressed the following technical barriers as they relate to the three external fuel processor Subsystems:

(A) Durability
(B) Performance
(C) Start-up Time and Transient Operation

2 Approach

The approach for this project included conducting durability tests in relevant environments using full-scale or nearly full-scale EFP components for a MWe-scale SOFC power plant. The components were designed and built as part of previous projects and were made available to this project for durability testing. An Outdoor Test Facility was constructed as part of a separate project funded by the Ohio Third Frontier Program and enabled the EFP to be tested under extreme weather conditions. These conditions would be representative of those expected for a SOFC power plant operating in northeast Ohio. Figure 1 shows a photograph of the EFP Subsystems installed in the Outdoor Test Facility in North Canton, Ohio.

The planned testing included the following:

- Operate the Desulfurizer Subsystem for at least 8,000 hours in an outdoor (hot and cold) environment using pipeline natural gas. This Subsystem generates desulfurized natural gas and operates whenever the SOFC is generating power. Therefore it is expected to operate for a much longer period compared to the other Subsystems. The planned test period represents the time between planned one-year maintenance intervals for the Desulfurizer in the SOFC power plant.

- Operate the Start-gas Subsystem for multiple start-ups and up to 200 hours in an outdoor environment using only natural gas and air to generate product gas with low flammables content. This Subsystem is expected to operate for less than 50 hours per year. The planned testing simulates up to a five-year service life for this Subsystem.

- Operate the Synthesis-gas Subsystem for multiple start-ups and for at least 1,200 hours in a heated enclosure using only natural gas and air to generate product gas with a hydrogen concentration less than 30% by volume. This Subsystem is used only during low-load operation of the SOFC to balance the thermal input and is required to operate for only a few hundreds of hours per year. The planned testing simulates up to a five-year service life for this Subsystem.

Post-test analyses of hardware were to be performed after completing the durability tests. Subsystem components (catalysts, sorbents, piping, reactors, insulation, valves, heaters, heat exchangers, nitrogen membrane, etc.) were to be inspected for deposits, signs of wear, damage, corrosion, and erosion. Physical and chemical analyses were to be performed on components as warranted.

Successful completion of the durability tests would demonstrate that the EFP Subsystems are ready for demonstration with a large-scale SOFC power plant.
3 Comparison of Accomplishments with Objectives

The project was initiated in August 2008 and ended in June of 2012. The following planned tasks were completed on this project:

- Hydrogen Safety Plan was issued and approved by DOE’s Hydrogen Safety Panel as planned
- Installed Desulfurizer and Start-gas Subsystems in the Outdoor Test Facility as planned
- Installed Synthesis-gas Subsystems in an indoor (laboratory) test facility as planned
- Commissioned control software, mechanical hardware and electrical hardware for Desulfurizer Subsystem, Synthesis-gas Subsystem and Start-gas Subsystem as planned
- Completed 10,062 hours of testing on the Desulfurizer Subsystem. This was more than the 8,000 hours in the original plan.
- Completed 122 hours of testing on the Start-gas Subsystem. This was less than the 200 hours in the original plan due to problems experienced with the original catalyst. A second catalyst was installed and additional tests performed. A total of 17 start-ups were performed with the second catalyst.
- Completed 1,230 hours of testing on Synthesis-gas Subsystem. The longest continuous operating period was about 1000 hours. Total testing exceeded the planned 1,200 hours of operation. A total of 23 start-ups were successfully performed.
- Samples from post-test inspections of the Desulfurizer and Synthesis-gas Subsystems were analyzed.
- Samples were not taken during post-test inspections of the Start-gas Subsystem due to the short duration of the testing. Only visual inspections were performed on this Subsystem.

4 Results and Discussion

4.1 Desulfurizer Subsystem

The Desulfurizer Subsystem generates pressurized, desulfurized natural gas (DNG) from high-pressure pipeline natural gas. The sulfur specification for pipeline natural gas is a maximum of 10 parts per million (PPM) on a volume basis. The target maximum allowable sulfur level in the DNG for use in the LGFCS SOFC power plant is 0.1 PPM or 100 parts per billion (PPB).

The Desulfurizer Subsystem was evaluated from July 2010 though June 2012. The software packages for the Subsystem’s control and safety systems were commissioned in July 2010. They were verified and validated for unattended operation. This enabled cost-effective long-term operation of the Desulfurizer Subsystem in the Outdoor Test Facility to support durability testing. The outlet of the Subsystem was connected to a ground flare for safe disposal of the desulfurized natural gas since a MWe-scale SOFC was not available to consume the gas. The Desulfurizer Subsystem was tested to determine the impact of ambient temperature on start-up time and system operation. The testing also determined the transient response to load changes and the Subsystem’s durability.
The Desulfurizer Subsystem used a natural gas burner to provide warm gas to heat-up its sorbent bed. The bed is comprised of an alkali oxide-based sorbent. The heat-up from an ambient temperature of 25°C required about 12 hours. This time increased to 27 hours when the heat-up was performed at an ambient temperature of -23°C, the coldest temperature recorded at the Outdoor Test Facility. Figure 2 shows the Desulfurizer Subsystem operating on February 22, 2011 with one foot of snow and an ambient temperature of -23°C. The heat-up time at the cold condition was longer than desired. Future system designs may include additional trace heating or weather protection to reduce the heat-up time during cold conditions. Two additional issues were observed during operation at low ambient temperature. During the first winter season the HVAC in the instrumentation and controls cabinet could not maintain the temperature above the low limit (2°C). This issue was corrected by placing a small supplemental heater in the cabinet. The second issue was related to maintaining constant reactant supply pressures. The supply lines for air and natural gas were not heat traced at the inlet pressure regulators. These pressure regulators failed to adequately control the supply pressures due to freezing and buildup of condensed liquids. Once these regulators were heat traced they properly controlled the supply pressures.

After the sorbent was preheated, it was maintained in a standby state using electric heaters. When in the standby state, the flow of reactants (natural gas and air) was initiated for start-up of the catalytic reactor in the Desulfurizer Subsystem. The reactors converts all the fuel sulfur species to sulfur oxides for removal by the sorbent. Start-ups from the standby state were performed at ambient temperatures ranging from -23 to 35°C. The start-up time required for producing DNG ranged from 56 minutes (at 35°C) to 74 minutes (at -23°C). The start-up time includes the time needed to heat the catalytic reactor, to initiate the oxidation reactions and to allow the system to reach thermal equilibrium. The start-up time was considered acceptable for a stationary power application.

The transient response for the Desulfurizer Subsystem was tested over a load range from 10% to 40% of the design DNG flow rate. The range used for the load change was limited by the natural gas compressor and not the Desulfurizer Subsystem. The time required for the load change was 25 seconds. This gave a load rate of change of 72% per minute which was slightly less than the target set by the U.S. Department of Energy (80% per minute). However this result exceeded the rate (40% per minute) deemed acceptable by LGFCS for stationary power application. Process variables showed a well-controlled response during the load change. The plot in Figure 3 shows the small impact of the load change on the operating pressure and temperatures for the Desulfurizer Subsystem.

The Desulfurizer Subsystem produced desulfurized natural gas from September 2010 though June 2012. It logged a total of 10,062 hours on stream. The Subsystem maintained the sulfur level in the DNG to below 50 ppb (the lower detection limit of the on-line sulfur analyzer) for the vast majority of the durability test period. This level was well below the target sulfur level (100 ppb). Figure 4 shows the total sulfur level measured in the desulfurized natural gas as a function of time on stream. The only operational issue observed during Desulfurizer Subsystem testing was spikes in the DNG sulfur content above 200 ppb during several low-load operating points. Additional tests were performed to address this issue. Results showed that accurate control of air flow was critical during low-load operation to insure proper oxidation of the fuel sulfur for its removal by the sorbent. Also shown in Figure 4 is the level of total hydrocarbons measured in the DNG. It ranged from 90 to 95% and was typically a few percentage points lower than the total hydrocarbons measured in the raw natural gas. The reduction was caused by the conversion of a small amount of hydrocarbons in the raw natural gas to carbon dioxides during the oxidation process that converts all the fuel sulfur to sulfur oxides for removal.

The Desulfurizer Subsystem successfully endured trips due to ancillary equipment failures (natural gas compressor trips, ground flare flameouts, and site electric power outages). There were also planned shutdowns for several holidays and to remove test-coupon samples. A total of 30 start-up cycles were
The plot in Figure 5 shows the percent of target load for the Desulfurizer versus time along with a number of the start-up cycles.

The Desulfurizer Subsystem worked very well at ambient temperatures up to 35°C, the highest temperature tested. The only problem observed at high ambient temperature was an issue with the HVAC control for the temperature in the instrumentation and controls cabinet. The HVAC set up did not keep the instrumentation and controls cabinet below the high limit temperature (47°C). Once the HVAC set up was corrected there were no other issues caused by high ambient temperature.

Inspections of the Desulfurizer Subsystem hardware found only one significant issue. A stainless steel flex line on the start-up burner had corroded and started to leak. The corrosion was due to sulfuric acid attack. Apparently moisture with sulfur oxides had condensed in the low point of line and caused the corrosion. The flex line was replaced with schedule 80 pipe. The system design will be change to eliminate low points for moisture to collect.

Coupon samples of the Desulfurizer Subsystem’s vessel material (316 stainless steel) and two alternate materials (Nitronic®30-AL201 and Nitronic®50-S20910) were exposed to system conditions for over 8,000 hours. The alternate materials were selected based on their lower costs compared to 316 stainless steel and their reported higher corrosion resistance. Both alternate materials did show lower corrosion rates (0.026 and 0.036 millimeters/year) compared to SS316 (0.084 millimeters/year) and are being considered for the vessel design for the commercial product.

### 4.2 Start-gas Subsystem

The Start-gas Subsystem uses a small amount of DNG with compressed air to generate the gas required for SOFC start up or shutdown. The gas is mostly nitrogen with a small amount of hydrogen and carbon oxides. Its concentration ranges from non-flammable (Hydrogen ~ 3%) to weakly flammable (Hydrogen ~ 12%). The Start-gas Subsystem was installed on the Outdoor Test Facility and connected to a ground flare for safe disposal of the Start gas since a MWe-scale SOFC was not available to consume the gas. Cold weather conditions made calibration of the Subsystem’s sensors for measuring hydrogen and oxygen concentrations nearly impossible. The Subsystem was enclosed to protect it from the weather (see Figure 6) which allowed calibrations to be completed and testing to proceed.

An important aspect of the Start-gas Subsystem operation is rapid catalyst light-off for start up. Several trials were conducted for “cold light-off” and “hot relight” conditions to establish the best procedures for reliable startup. The goal was to initiate reactions without a transient condition that could adversely impact catalyst durability due to overheating. During SOFC power plant operation, hot relight will likely be conducted more often than a cold light-off. Of the two, the hot relight, which is conduction from the standby condition, is the more difficult to execute without overheating the catalyst. A total of 17 light-off/relight tests were performed. In each case, a minimum of approximately 1.5 minutes was required from initiation of the start sequence to the measurement of the target hydrogen content in the start gas. Flow conditions at start-up that matched the steady state process condition were acceptable with respect to a temporarily higher catalyst outlet temperature and hydrogen concentration during light-off.

The hot relight procedure was more difficult to execute due to the potential for a temporary drop in reactants temperature caused by heat sinks in the Subsystem. However, in spite of these heat sinks, successful hot relights were demonstrated without failures. A closer coupling between the pre-heater and catalyst reactor should be employed in future Start-gas Subsystem designs to minimize the drop in temperature due to the heat sinks.
The Start-gas Subsystem was tested over a range of operating conditions to generate Start gas with the required amount of flammables. The low end of the range is referred to as the lean condition while the high end is referred to as the rich condition. All the tests were conducted using an online hydrogen sensor to measure hydrogen concentration in the product gas. Gas samples were also extracted and analyzed off-line using a gas chromatograph (GC) to determine major constituents in the product gas (carbon dioxide, carbon monoxide, nitrogen, oxygen and methane). The primary benefit of the GC was to determine methane in the product gas. The methane represented the slip of reactants which became significant at the rich condition.

The first catalyst installed in the Start-gas Subsystem overheated during a control system malfunction during commissioning tests. This overheating likely damaged the catalyst as indicated by higher than expected methane concentrations in the product gas and unstable product gas composition particularly at the rich condition. As load (total flow) was lowered, the catalyst temperature and the measured product gas composition stabilized, but methane slip was still elevated indicating inadequate conversion. Visual inspections of the first catalyst showed some physical evidence of damage likely due to the overheating. The catalyst was replaced with a new catalyst of an identical size and composition. This unplanned event reduced the time available for testing the Start-gas Subsystem.

Testing of the Start-gas Subsystem with the replacement catalyst totaled 122 hours. The Subsystem produced gas with a wide range of flammables content. The plot in Figure 7 shows how the flammables content was quickly changed as a function of operating conditions. Unfortunately these conditions also gave higher than expected methane concentrations (methane slip). It was theorized that the high level of methane slip observed was due to air and fuel bypassing the catalyst. This was suspected due to difficulties encountered during the installation and sealing of the replacement catalyst in the test reactor. Test results support this theory by showing stable centerline temperatures that approximate thermodynamic equilibrium. Process simulation calculations, using a mechanism whereby some air and fuel bypass the reactor and then oxidize hydrogen down stream of the reactor, corroborated the measured dry product gas compositions. From test results at the rich operating condition, it was also theorized that poor methane conversion along the centerline of the catalyst may be contributing to elevated methane slip. These results indicate that further catalyst testing, including alternate catalyst formulations, is needed. The results also highlight the need for proper catalyst installation with a good seal to minimize bypass and to ensure reliable gas production.

Although a stable rich condition was eventually achieved, significant methane slip was observed. In addition, the methane slip increased at the rich operating condition. Significant methane slip must be avoided as a constituent of the Start gas since at intermediate to high temperatures in the SOFC the methane may be a precursor to carbon formation at the anode. It was not possible to decipher to what degree the methane slip was attributed to air and fuel bypass versus insufficient inherent catalyst activity.

Due to the limited duration (122 hours) of the Start-gas testing, only visual post-test inspections were performed on this Subsystem. No signs of significant wear or corrosion were observed. Since additional tests are planned for this Subsystem in a follow-on program, no Subsystem materials were removed for physical or chemical analyses.

### 4.3 Synthesis-gas Subsystem

The Synthesis-gas Subsystem was installed inside a test enclosure (see Figure 8) to simulate its operating environment which was planned to be inside the SOFC power plant enclosure. The Synthesis-gas Subsystem uses a catalytic reactor to generate hydrogen from room temperature natural gas and air. The synthesis gas is fed to the spent fuel burner to help maintain SOFC temperature during low-load operation. The burner is designed to operate on spent fuel from the SOFC (dilute hydrogen).
and not natural gas, hence the need for synthesis gas. The Synthesis gas subsystem was tested to determine its start-up characteristics and the impact of operating time on its performance.

The catalytic reactor in the Synthesis-gas Subsystem was heated for start-up using air preheated by an electric heater. The heat-up time was 62 minutes. Once the catalyst was at temperature, the natural gas and air flows were initiated. Within 40 seconds the reactor began producing hydrogen. After about 120 seconds the hydrogen concentration was near the target level. The hydrogen production during start-up closely followed the catalyst outlet temperature. A plot showing hydrogen concentration and catalyst temperatures during start-up is shown in Figure 9. The time for start-up from the hot standby condition was 2 minutes. This result was considered acceptable for the SOFC stationary power application.

The Synthesis-gas Subsystem was tested for a total of 1,230 hours. After the first 230 hours of testing a control system software error caused overheating of the catalyst that resulted in an unacceptable reduction in performance. The control system error was corrected and the catalyst was replaced. After these changes the Subsystem was successfully operated for 1,000 hours. The hydrogen content in the product gas declined slowly with overtime. At the completion of the testing, the hydrogen concentration had dropped to about 85% of the initial target value (equivalent to 15% decline per 1,000 hours). The plot in Figure 10 shows the decline is hydrogen content. The decline was consistent with a loss in catalyst activity. The decline in hydrogen concentration was within an acceptable range for the SOFC stationary power application.

The transient response for the Synthesis-gas Subsystem was evaluated during a load change from 17% to 50% load. The load change was performed over a 55-second period. This gave a load rate of change of about 36% per minute which was less than the target set by the U.S. Department of Energy (80% per minute). However this result was close enough to the rate (40% per minute) deemed acceptable by LGFCS for stationary power application. A faster rate of change is likely possible with additional tuning of the natural gas and air flow control valves. The hydrogen concentration in the product gas remained within 95% of the target value during the transient while the outlet temperature showed a slight increase (40°C). The plot in Figure 11 shows the impact of the load change on the operating temperatures and hydrogen concentration for the Synthesis-gas Subsystem.

The Synthesis-gas Subsystem was disassembled after all testing had been completed. Visual inspections were performed. The heaters, inlet piping and control valves showed no signs of degradation or deposits. A light coating of carbon was observed at the outlet of the catalyst section and in the outlet piping. The carbon deposition on the catalyst may have contributed to the observed reduction in hydrogen yield over time. The reactor and its internal components (all alloy 625- Inconel) showed some signs of degradation. Photos of these components are shown in Figure 12. Metallurgical analyses of the components were performed. No sulfidation corrosion was detected in any of the components. Oxidation corrosion was active in all the components with exception of the support piece. It contained no active discernable corrosion mechanism or carburization. Various other surfaces were found to be carburized. The locations containing the deepest oxidation were congruent with the locations of maximum carburization. This is intuitive considering that carbide formation depletes chromium leaving metal more susceptible to oxidation. The sleeve (foil liner) was penetrated by oxide. Carbides were found throughout its entire wall. The reactor outlet (tube) suffered oxidation. Carbides were found throughout its entire wall. The Thermowell contained oxide penetration at all examined locations. Oxide penetration increased in severity towards the hot end (tip). The tip was carburized from the outer diameter side penetrating most of the wall thickness. The results for the Thermowell were the most disconcerting. A protective coating on the Thermowell or an alternative material will be considered in the design for the commercial product.
5 Conclusions

The desulfurizer Subsystem durability testing logged over 10,000 hours. The subsystem worked well and met its performance requirements. Its results included:

- Start-up times from the standby condition ranged from 56 minutes to 74 minutes depending on ambient temperature
- Transient response (load rate of change) was 72% per minute
- Sulfur level in desulfurized natural gas was less than 50 ppb for most of the testing
- Some sulfur breakthrough observed at the low-load condition

The start-gas Subsystem durability testing logged a total of test 122 hours. The Subsystem met most of its performance requirements. Its results included:

- Start-up time from the standby condition was 1.5 minutes
- Hydrogen concentrations achieved the target values (3 to 12%)
- Significant methane slip was observed in the product gas from the Start-gas Subsystem. This could impact SOFC operation during start up. Further catalyst testing, including alternate formulations, is needed to address the methane slip.

The Synthesis-gas Subsystem durability testing logged a total of test 1,337 hours. The Subsystem worked well and met its performance requirements. Its results included:

- Start-up time from the standby condition was 2 minutes
- Transient response (load change rate) was 36% per minute
- Hydrogen concentration achieved the target value
- Hydrogen concentration declined at a rate of 16% per 1,000 hours
- Signs of oxidation and carburization observed for the Synthesis-gas reactor internal components. These should be addressed in the design for the commercial product.

6 Publications/Presentations

Papers:

Presentations:
- “Extended Durability Testing of External Fuel Processor for 1MWe SOFC” presented by Anant Upadhyayula at 2012 U.S. DOE HYDROGEN PROGRAM and VEHICLE TECHNOLOGIES PROGRAM ANNUAL MERIT REVIEW and PEER EVALUATION MEETING on May 15, 2012
- “Extended Durability Testing of External Fuel Processor for 1MWe SOFC” presented by Mark Perna and Anant Upadhyayula at 2011 U.S. DOE HYDROGEN PROGRAM and VEHICLE TECHNOLOGIES PROGRAM ANNUAL MERIT REVIEW and PEER EVALUATION MEETING on May 10, 2011
- “Extended Durability Testing of External Fuel Processor for 1MWe SOFC” presented by Mark Perna at the 2010 U.S. DOE HYDROGEN PROGRAM and VEHICLE TECHNOLOGIES PROGRAM ANNUAL MERIT REVIEW and PEER EVALUATION MEETING on June 8, 2010

“Extended Durability Testing of External Fuel Processor for 1MWe SOFC” presented by Mark Perna at 2009 U.S. DOE HYDROGEN PROGRAM and VEHICLE TECHNOLOGIES PROGRAM ANNUAL MERIT REVIEW and PEER EVALUATION MEETING on May 18, 2009

“Extended Durability Testing of External Fuel Processor for 1MWe SOFC” presented by Mark Perna to the DOE Project Officer at the PROJECT KICK-OFF MEETING on March 18, 2009

**Acronyms**

SOFC - solid oxide fuel cell
MWe - megawatt, electrical
EFP - external fuel processor
DNG - desulfurized natural gas
Ppm – parts per million
Ppb – parts per billion
Figure 1 - EFP Subsystems in the Outdoor Test Facility

Figure 2 - Desulfurizer Subsystem Being Tested in the Outdoor Test Facility
Figure 3 – Transient Response of Desulfurizer Subsystem
Figure 4 – Total Sulfur Level and Hydrocarbons in Desulfurized Natural Gas

Figure 5 – Operating Load for Desulfurizer Subsystem
Figure 6 - Start-gas Subsystem (with and without enclosure)

Figure 7 - Start-gas concentrations
Figure 8 - Synthesis-gas Subsystem in Test Enclosure

Figure 9 – Hydrogen Concentration in Synthesis Gas and Catalyst Temperatures During Start up
Figure 10- Decline of Synthesis gas Hydrogen concentration with time

Figure 11 – Transient response for 17% to 50% load on the Synthesis-gas Subsystem
Synthesis-gas reactor parts after 1000hrs operation

Figure 12 – Synthesis-gas catalyst and the reactor components